Supplementary Information (SI) for Organic & Biomolecular Chemistry. This journal is © The Royal Society of Chemistry 2024

# Conversion of aromatic methyl ketones to esters and carboxylic acids using o-pthalaldehyde as oxidant

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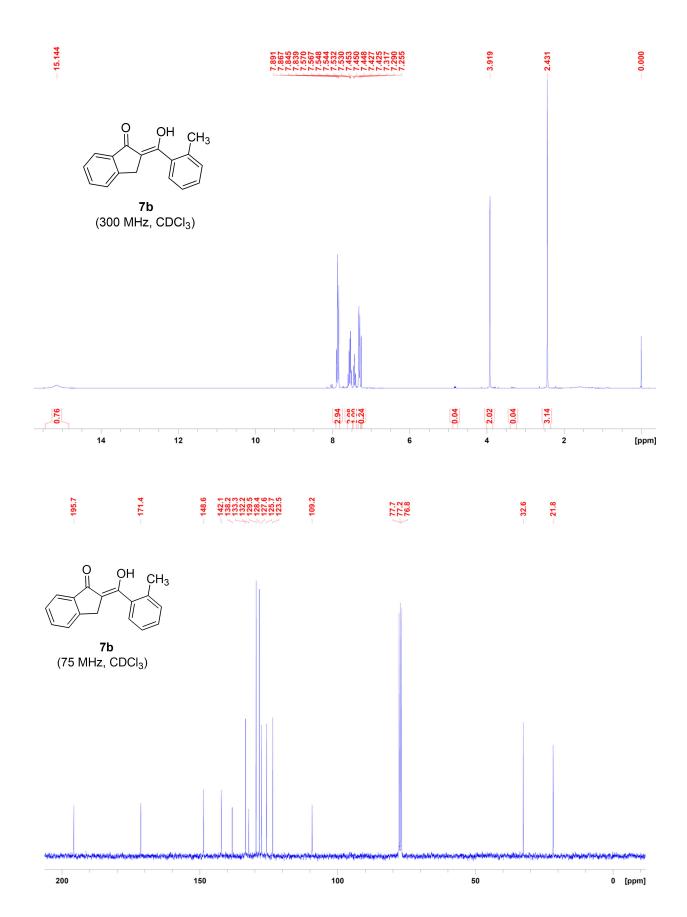
## **Experimental Section:**

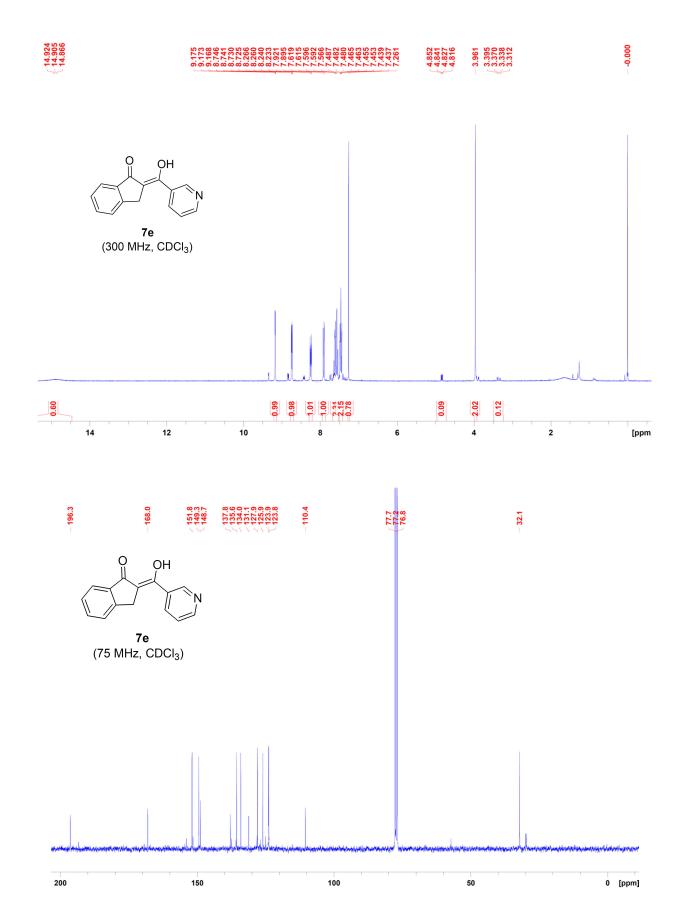
### Gram scale synthesis of ester 8ha:

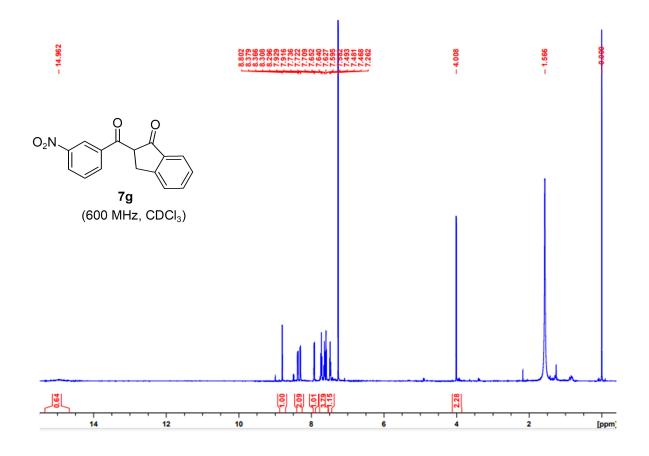
In a round bottle flask sodium hydroxide (1.45 g, 36.25 mmol) was dissolved in ethanol (150 ml) at room temperature. The reaction flask was cooled to 0  $^{\circ}$ C and to that o-pthalaldehyde (5.80 g, 43.17 mmol) and 2-acetyl naphthalene **6h** (5.00 g. 33.30 mmol) were added respectively. The reaction was stirred at 0  $^{\circ}$ C for 1 hour and then slowly warmed to room temperature over 1 hour and monitored by TLC. The reaction mixture was then neutralized with 10% HCl (20.0 ml). The alcohol from the reaction was evaporated and the solid residue was extracted with ethyl acetate (2x100 ml). The combined organic layer was washed with water (50 mL) and dried over anhydrous sodium sulfate before evaporated to get 10.65 gm crude 1,3 -diketone **7h** which was used as such for the next reaction. The crude diketone **7h** was placed in a 250 ml round bottom flask and to that n-butanol (100 mL) and  $\ln(\text{OTf})_3$  (850 mg, 1.51 mmol) were added. The flask was then sealed with rubber septa and heated for 8 hours at 110  $^{\circ}$ C upon which almost all starting diketone **7d** was completely consumed as observed by TLC. Then the alcohol from the reaction mixture was evaporated and the crude residue was loaded in silica gel column. Elution with 5 % EtOAc in Hexane gave pure ester **7d** (5.70 g, 27.37 mmol) in 82% overall yield.

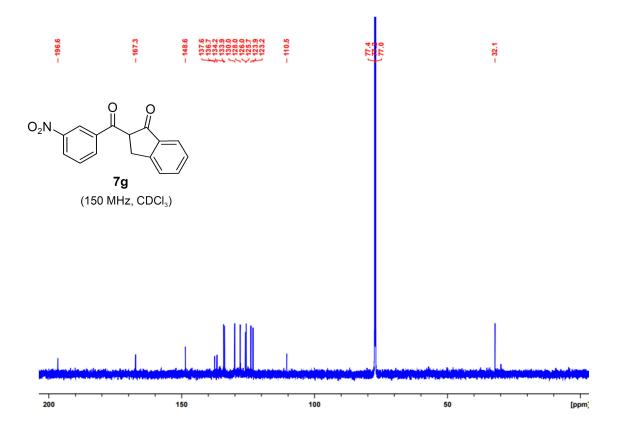
#### One pot conversion of methyl ketone 6c to ester 8c:

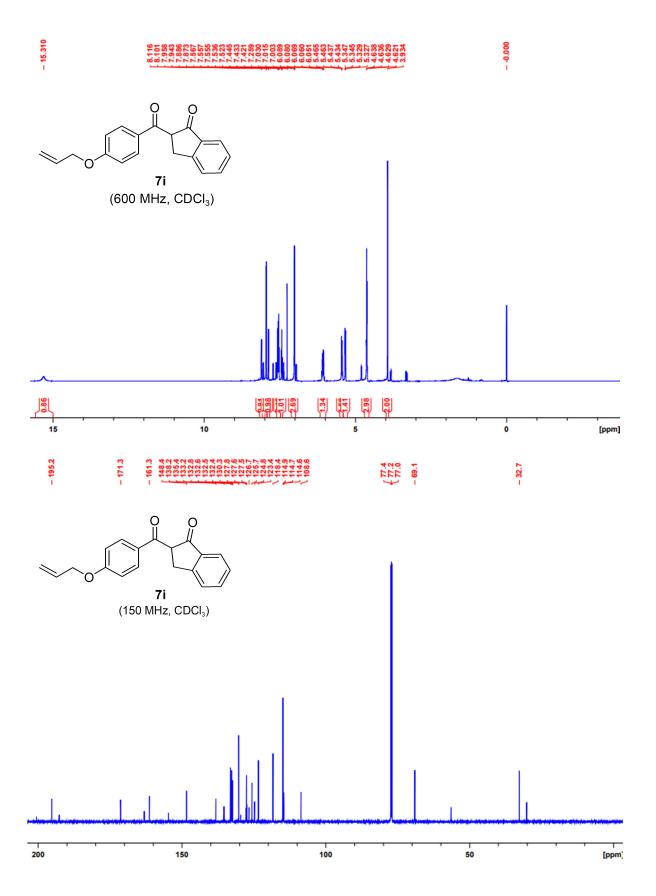
In a round bottle flask sodium hydroxide (90 mg, 2.25 mmol) was dissolved in n-butanol (10 ml) in a round bottom flask at room temperature. The reaction flask was cooled to 0 °C and to that o-pthalaldehyde (350 mg, 2.61 mmol) and aryl methyl ketone **6c** (300 mg, 2.00 mmol) were added respectively. The reaction was stirred at 0 °C for 1 hour and then slowly warmed to room temperature over 2 hour and monitored by TLC. The reaction mixture was then neutralized with drop-wise addition of 20% HCl. After that  $In(OTf)_3$  (60 mg, 0.11 mmol) was added to the reaction and the reaction mixture was refluxed for 8h. Then the alcohol from the reaction mixture was then evaporated and the crude residue was loaded in silica gel column. Elution with 10% EtOAc in Hexane gave pure ester **8** (325 mg, 1.56 mmol) in 78% yield.

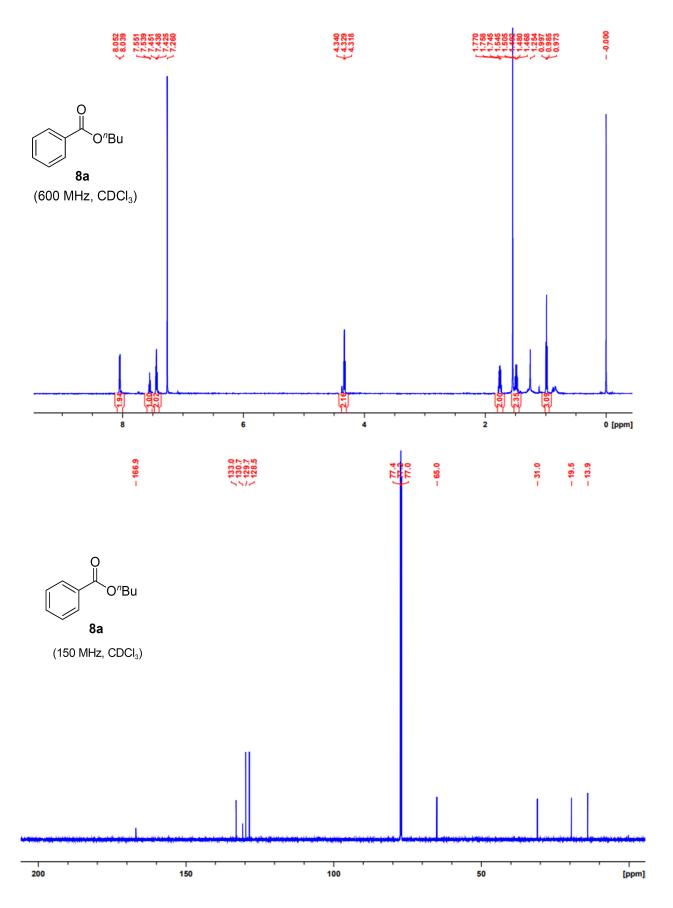


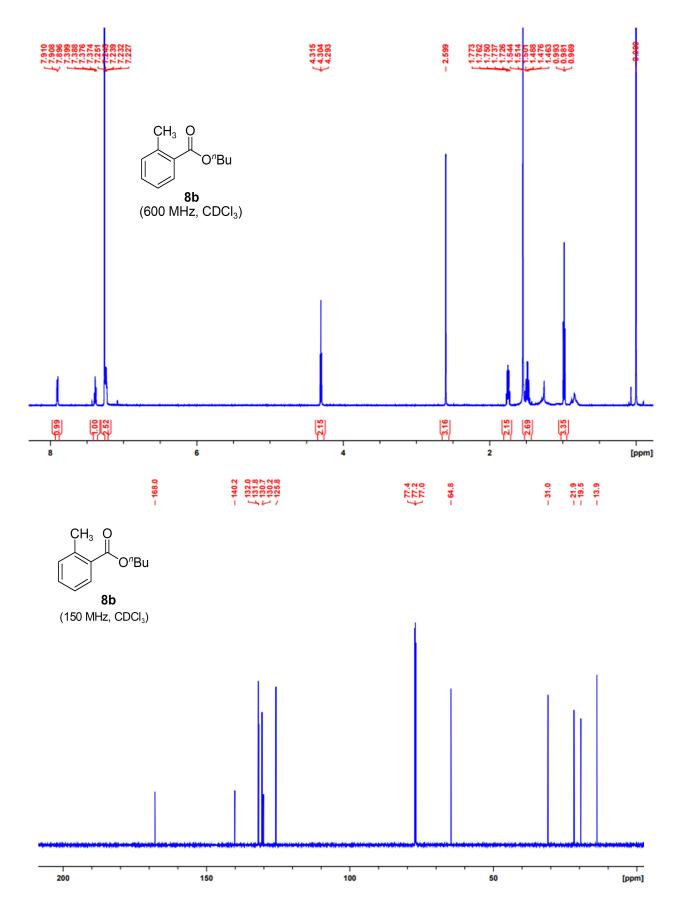


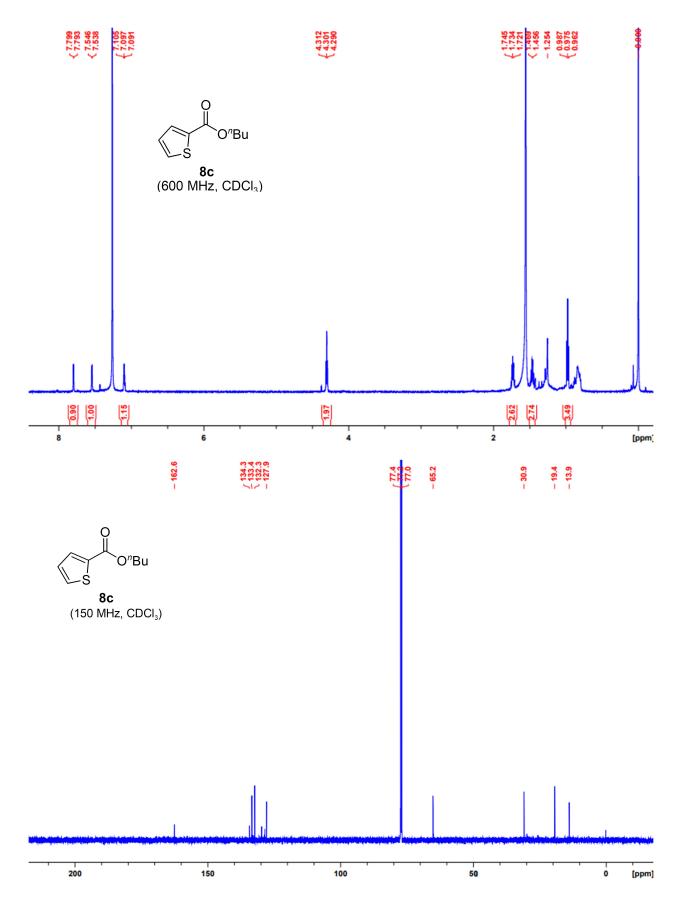


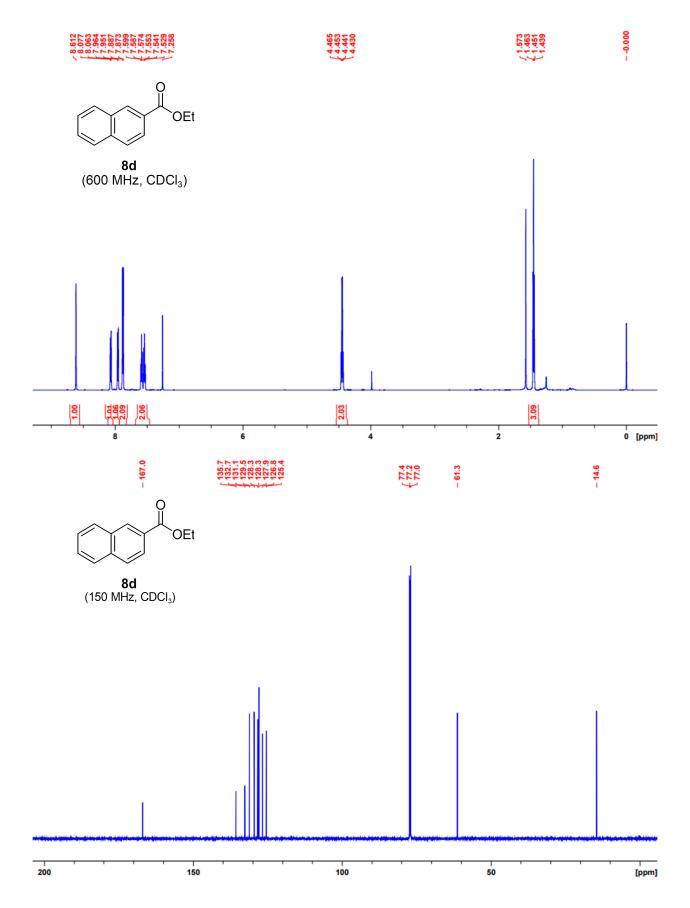


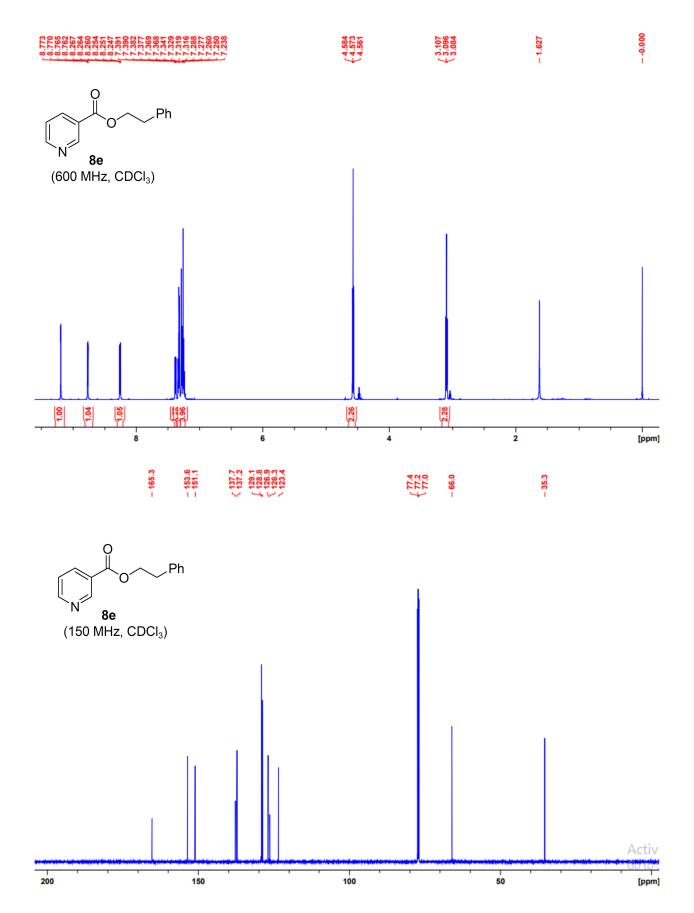


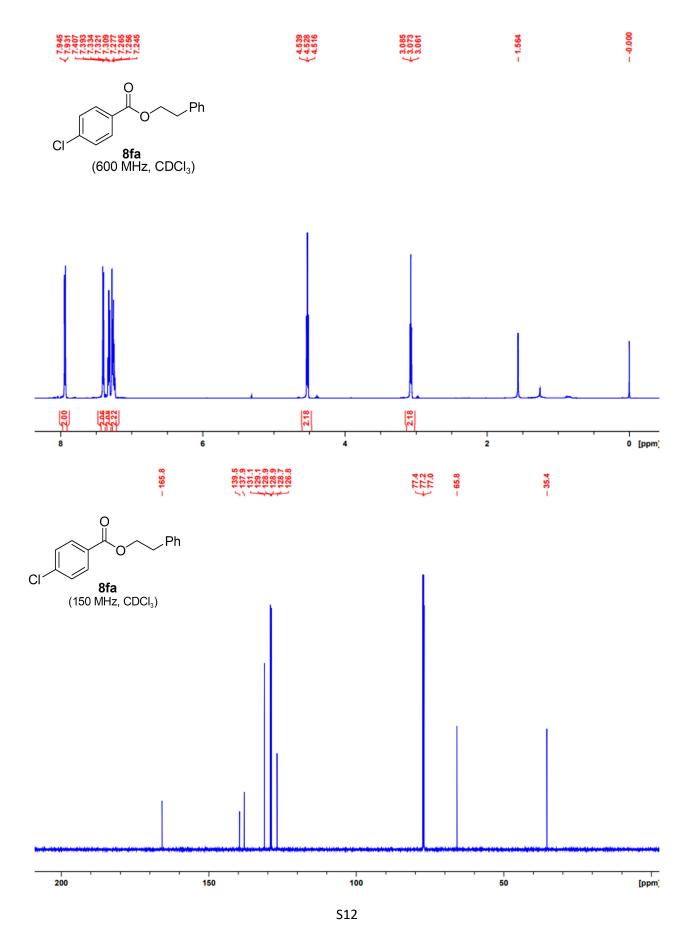


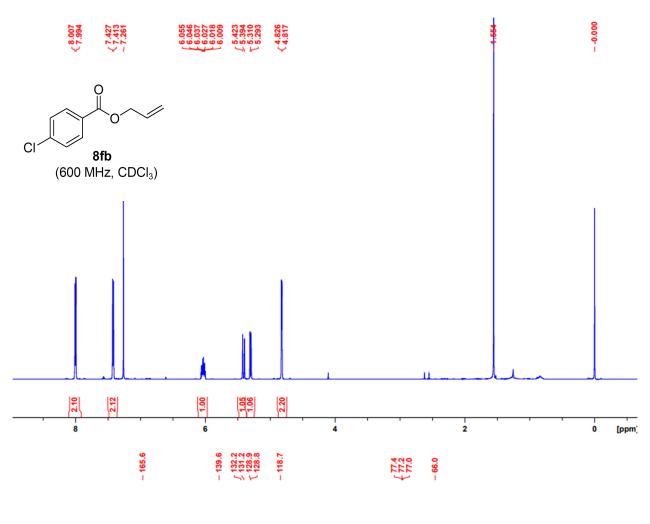


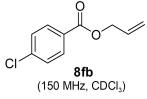


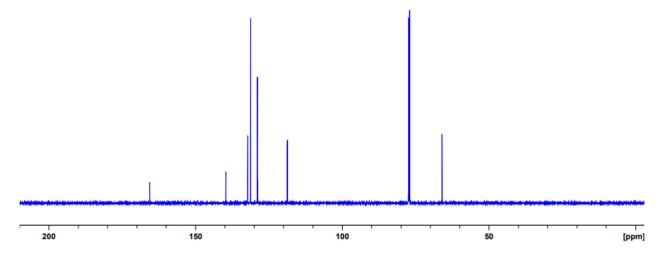


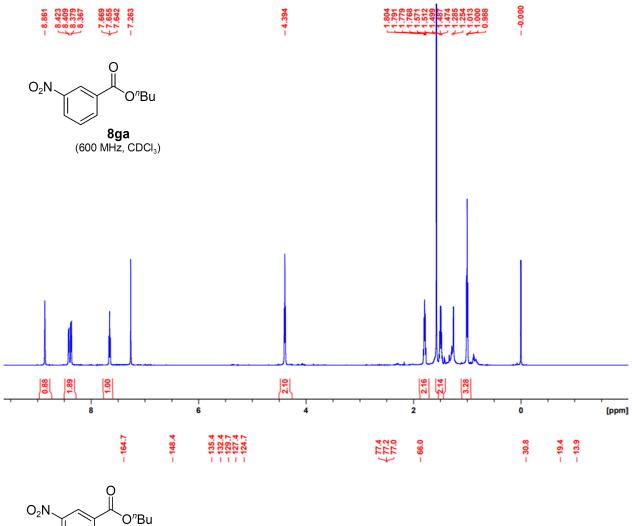




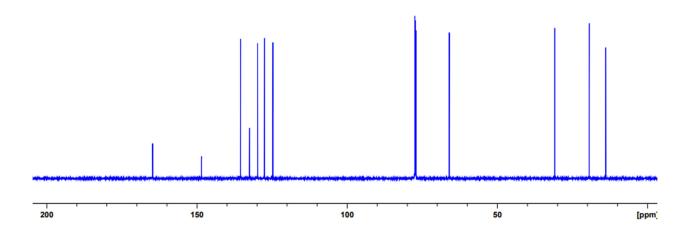


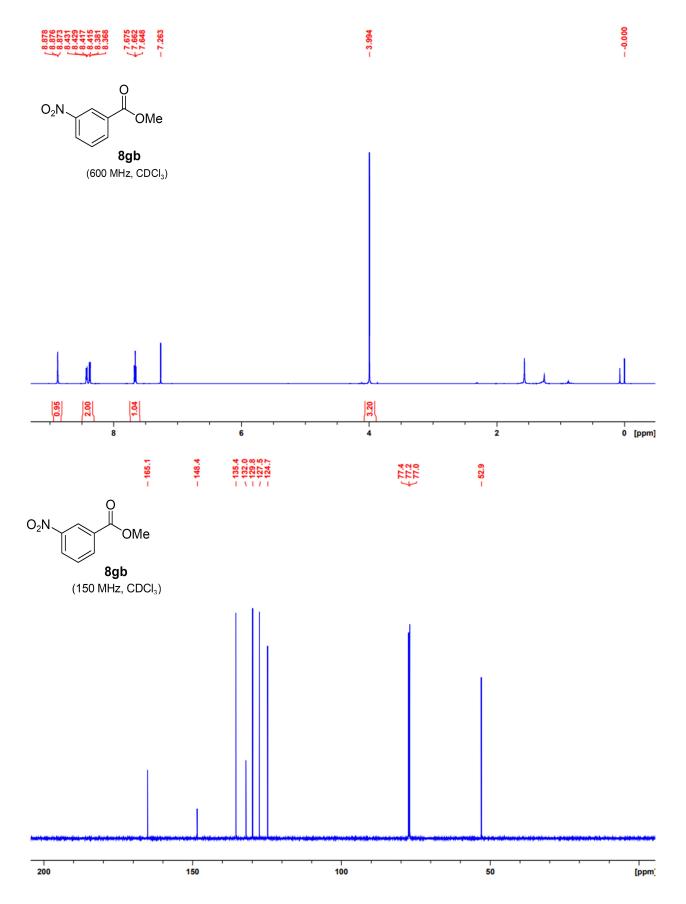


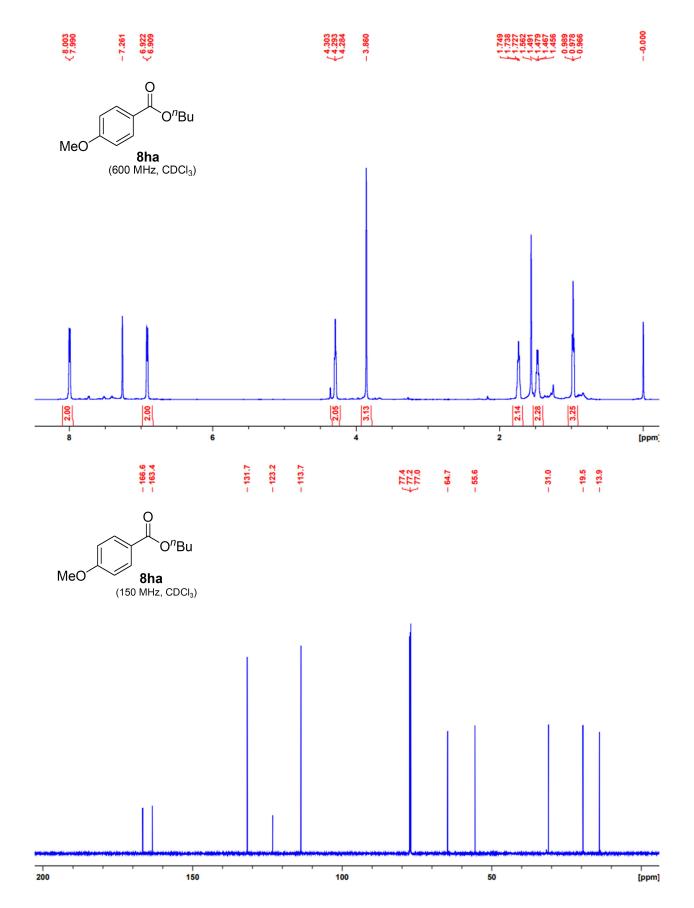


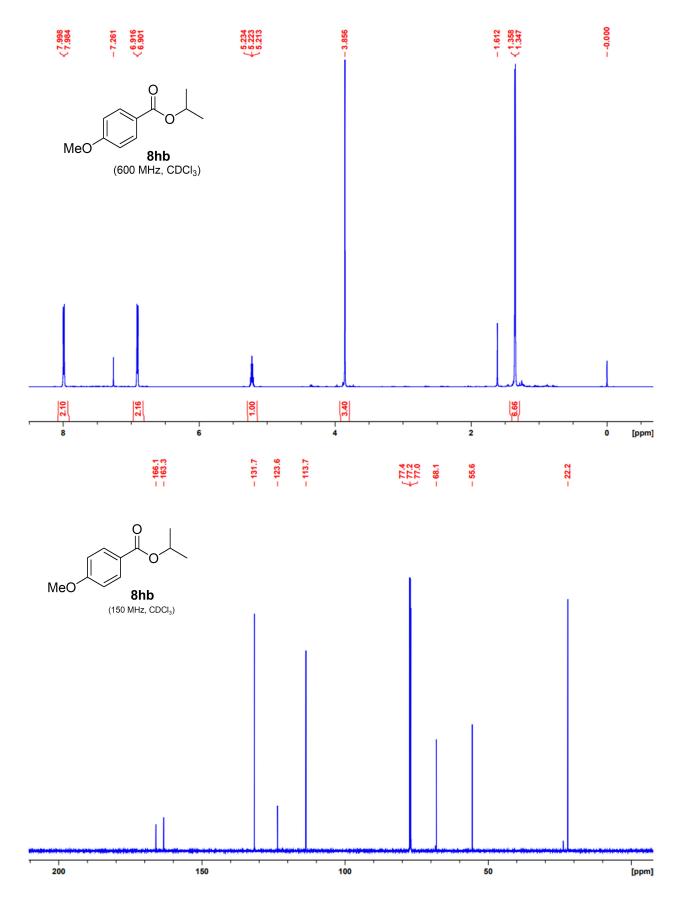


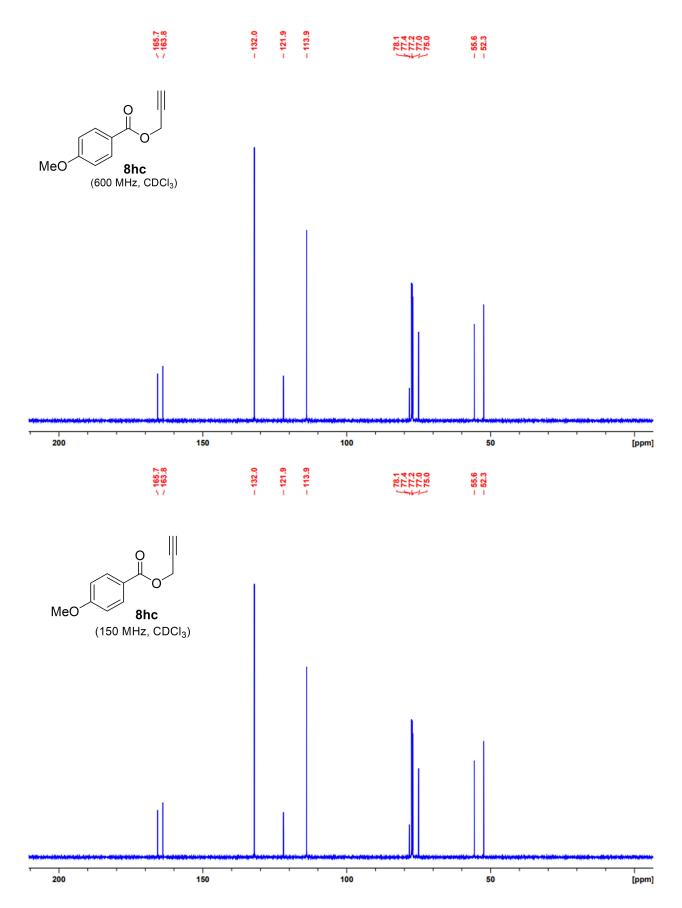


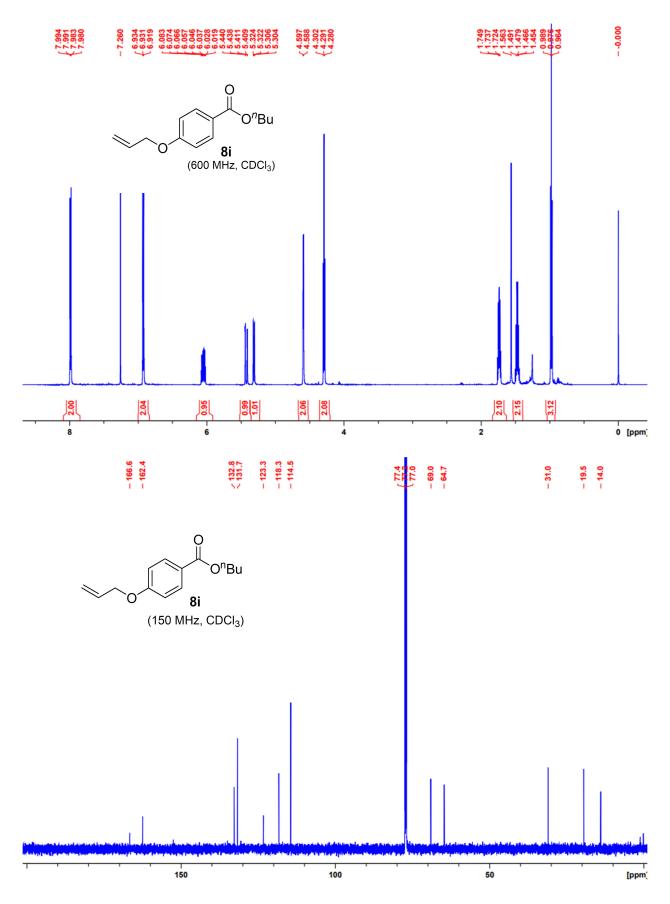


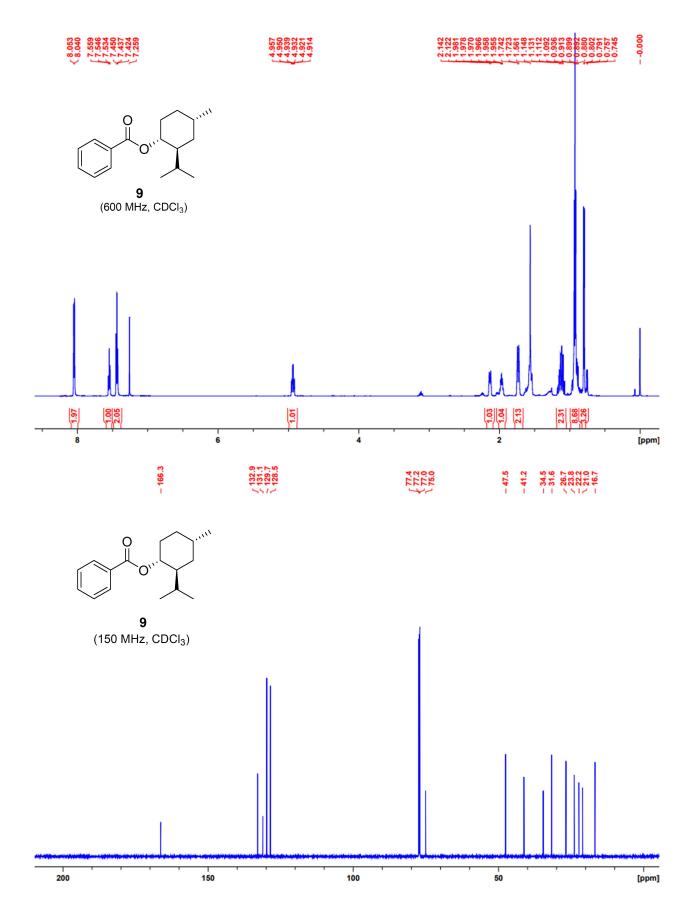


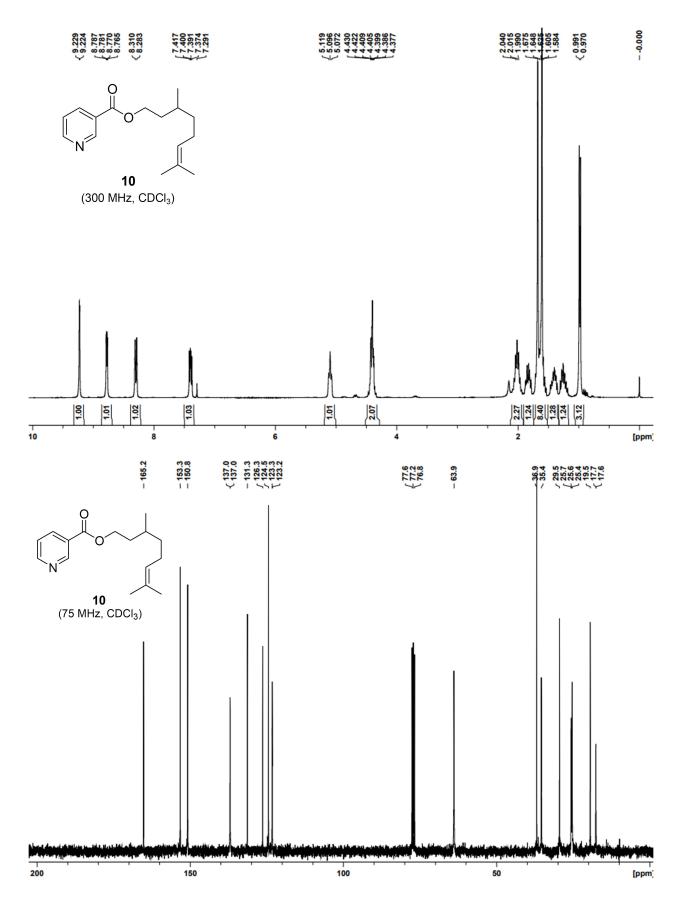






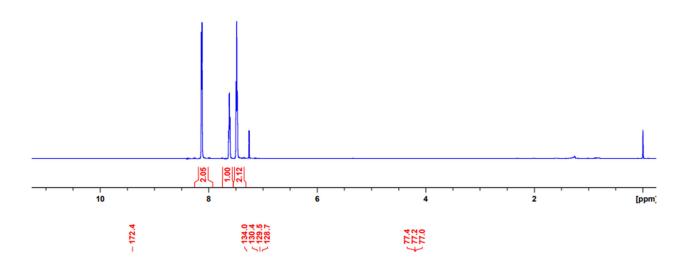




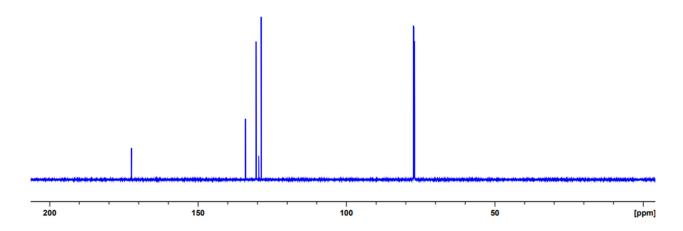


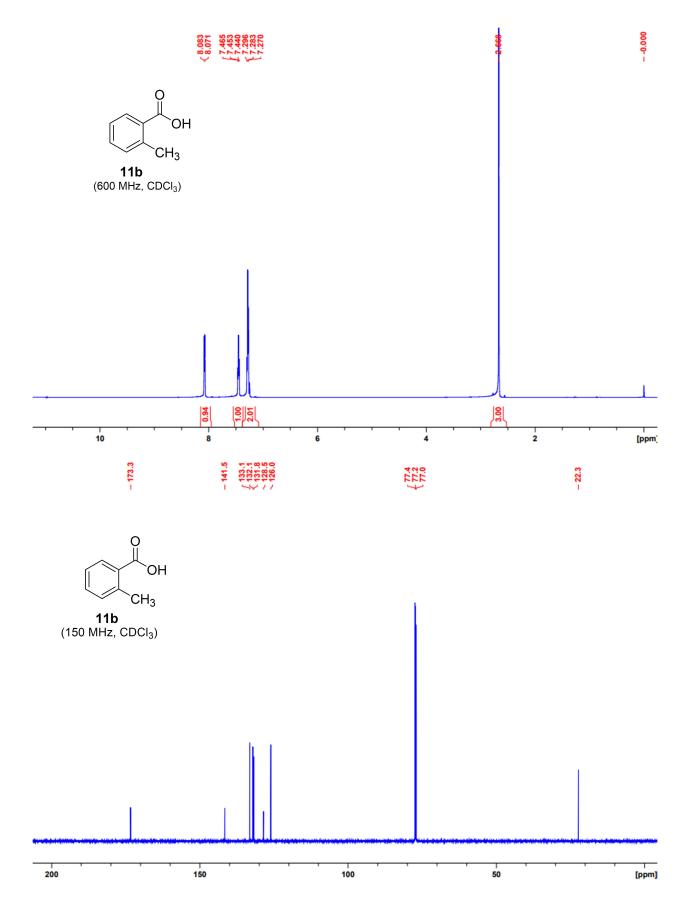


**11a** (600 MHz, CDCl<sub>3</sub>)



**11a** (150 MHz, CDCl<sub>3</sub>)

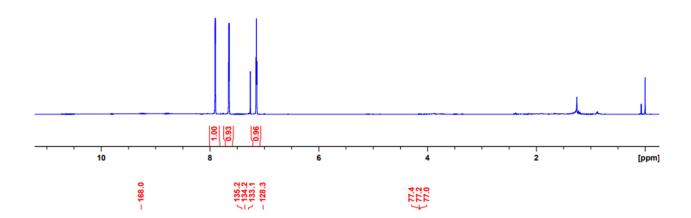




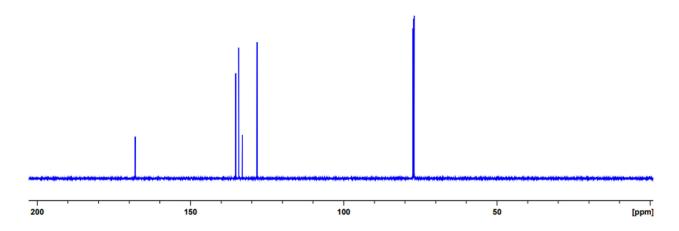


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**11c** (600 MHz, CDCl<sub>3</sub>)



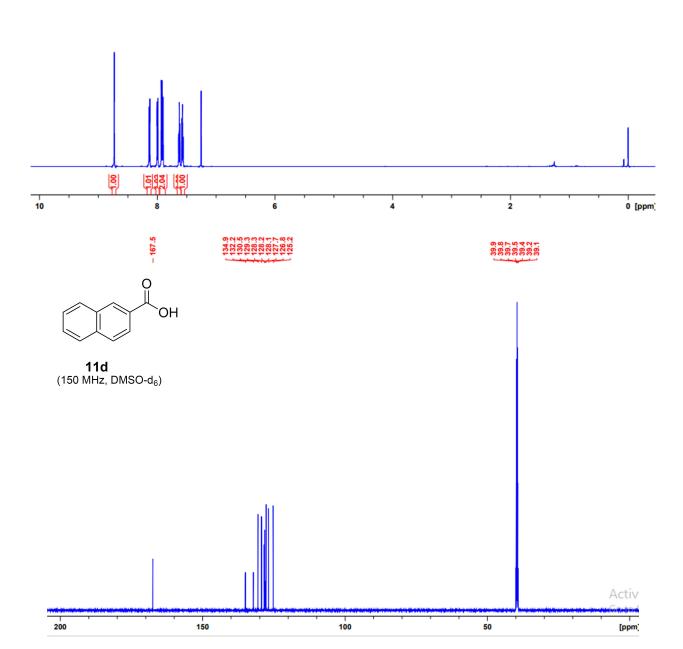
**11c** (150 MHz, CDCl<sub>3</sub>)

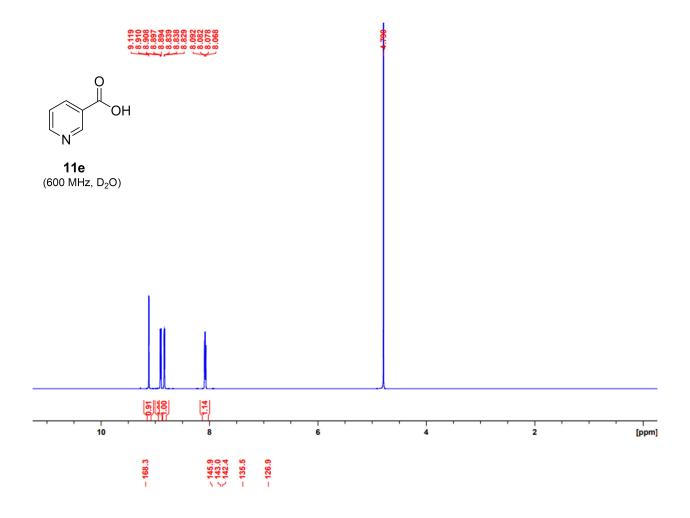




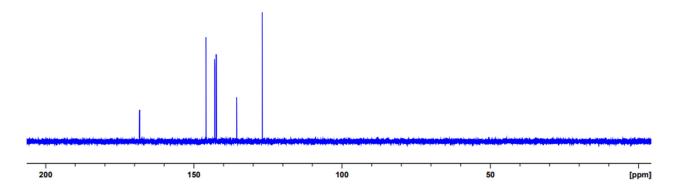


**11d** (600 MHz, CDCl<sub>3</sub>)





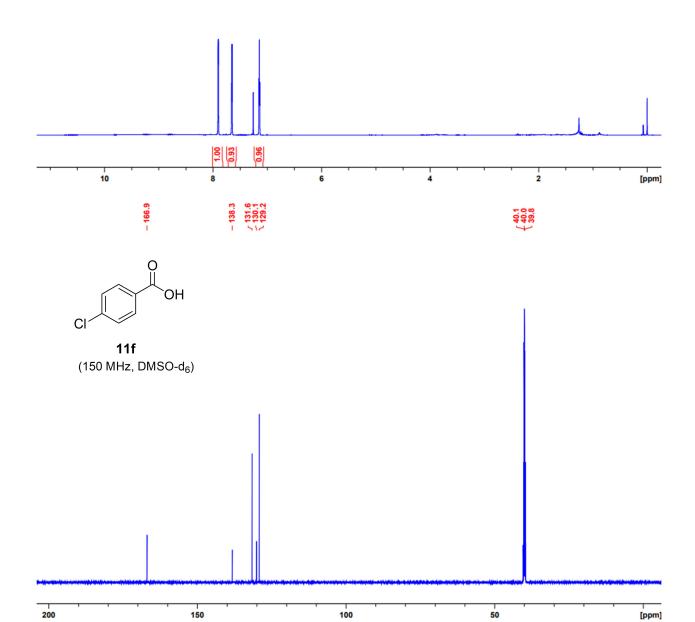
**11e** (150 MHz, D<sub>2</sub>O)







**11f** (600 MHz, CDCl<sub>3</sub>)





--0.000

