

ROMP-based Recyclable Magnetic Acylation Reagentsⁱ

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Supporting Information

1. TEM pictures	S2
2. ¹H-NMR assay	S3
3. Filtration experiments	S3
4. NMR spectra	S4

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1. TEM pictures

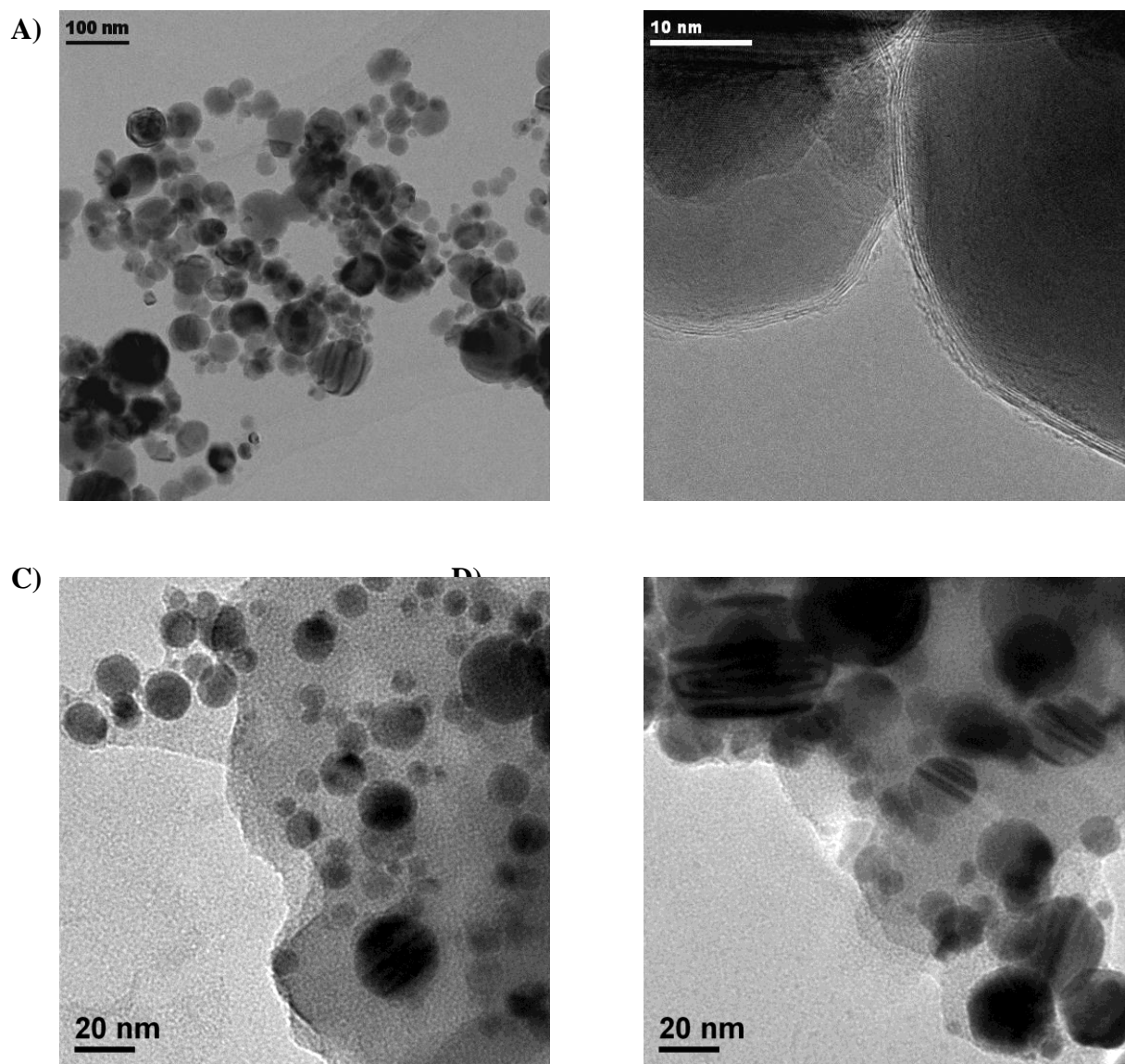


Figure S1. TEM pictures of pristine Co/C nanobeads **1** (A, B), as-prepared magnetic Co/C ROMPgel **5** (C) and **5** after five cycles of acylation of amines and subsequent recycling (D). No substantial changes between C and D are noticeable.

2. ^1H -NMR assay

^1H -NMR (300 MHz, CDCl_3 , 10% MeOH)

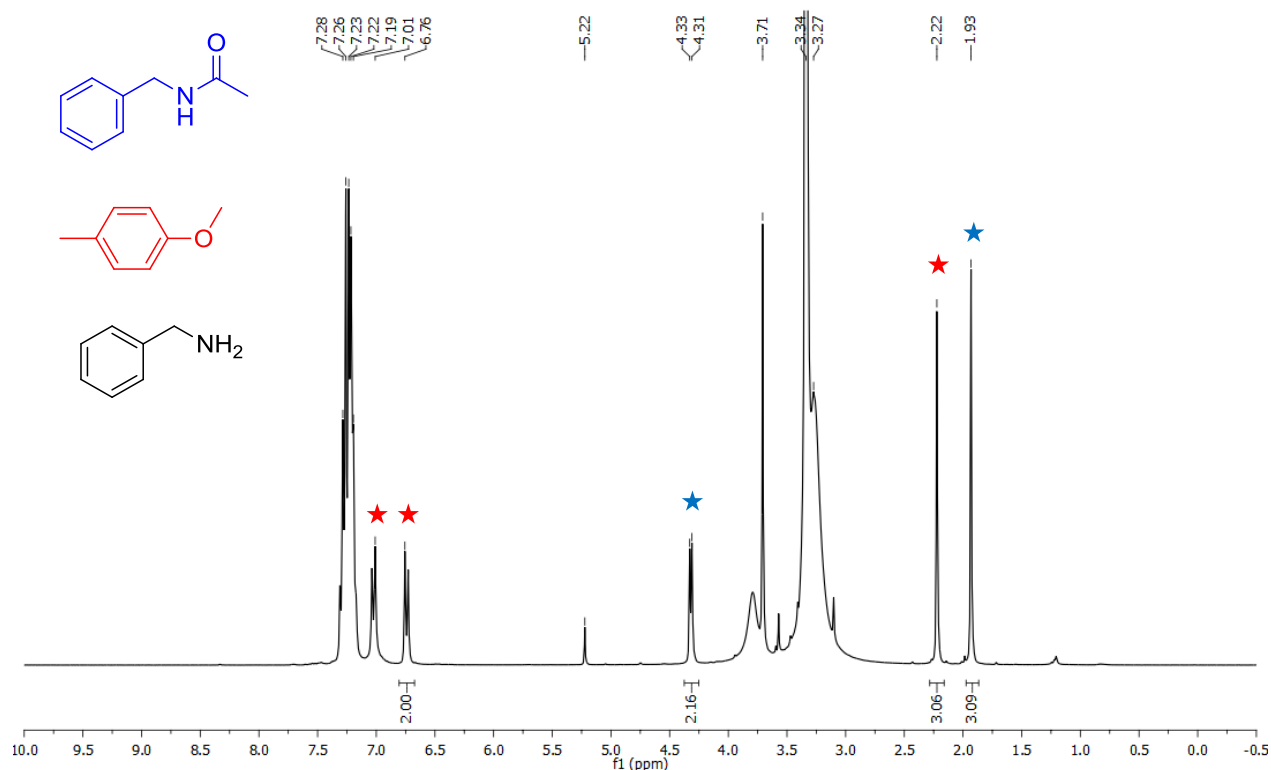


Figure S2. ^1H -NMR picture as example for the analysis of the loading of ROMPgel **5** by NMR-assay is depicted. The integration of the benzylic CH_2 doublet (4.32 ppm) of the N -benzylacetamide (**8a**) was compared with that of the phenylic CH groups (6.75 ppm) of the 4-methylanisole (internal standard). With the product/standard ratio (1.08), the amount of standard in solution (120 μmol), and the amount of nanoparticles used (50 mg) a loading of 2.59 mmol/g was calculated.

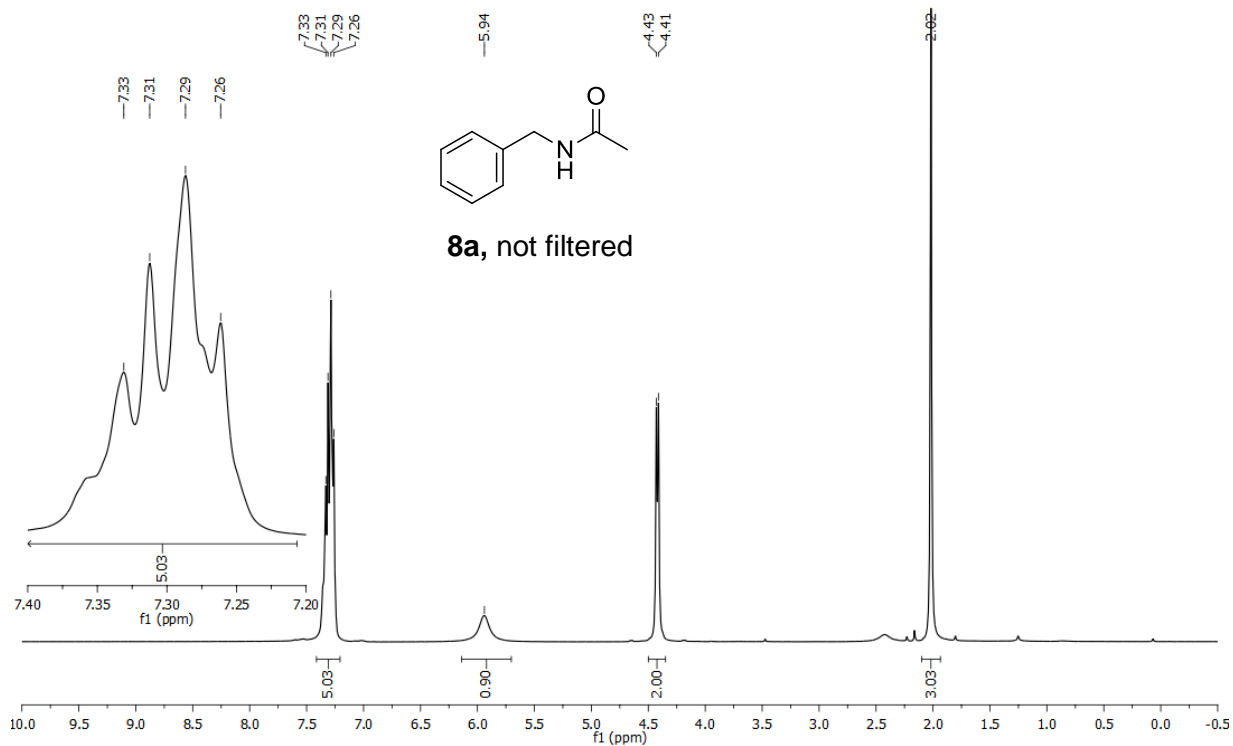
3. Filtration experiments

To quantify the removal of resin and/or polymer traces from the product solution by filtration the following experiment was performed:

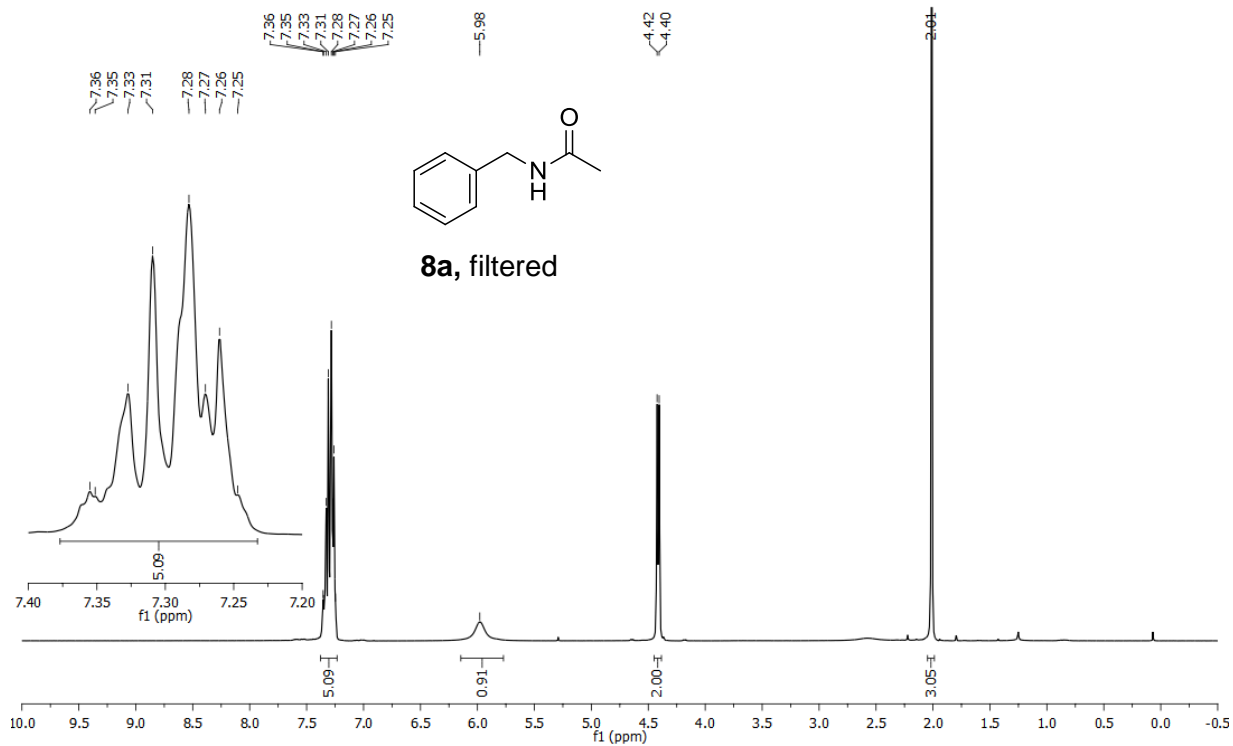
Following to the general procedure Fe/C ROMPgel **5** (400 mg, 0.90 mmol, 2.26 mmol/g) loaded with acetyl groups was stirred with benzylamine (76 μL , 0.7 mmol) in 6 mL of a $\text{CHCl}_3/\text{MeOH}$ (9:1) mixture for 5 h. The resin was removed by magnetic decantation and washed twice with 5 mL $\text{CHCl}_3/\text{MeOH}$ (9:1). Evaporation of the solvents resulted in 89.2 mg (0.6 mmol, 86%) of crude N -benzylacetamide **8a** as a pale brown solid. The product was dissolved in CH_2Cl_2 and filtered over cotton. After evaporation 88.0 mg (0.59 mmol, 84%) of the product was obtained. The filter was dried under vacuum for several hours. The weight gain of the filter was determined as 1.0 mg, which corresponds to 0.25 wt% of the resin used.

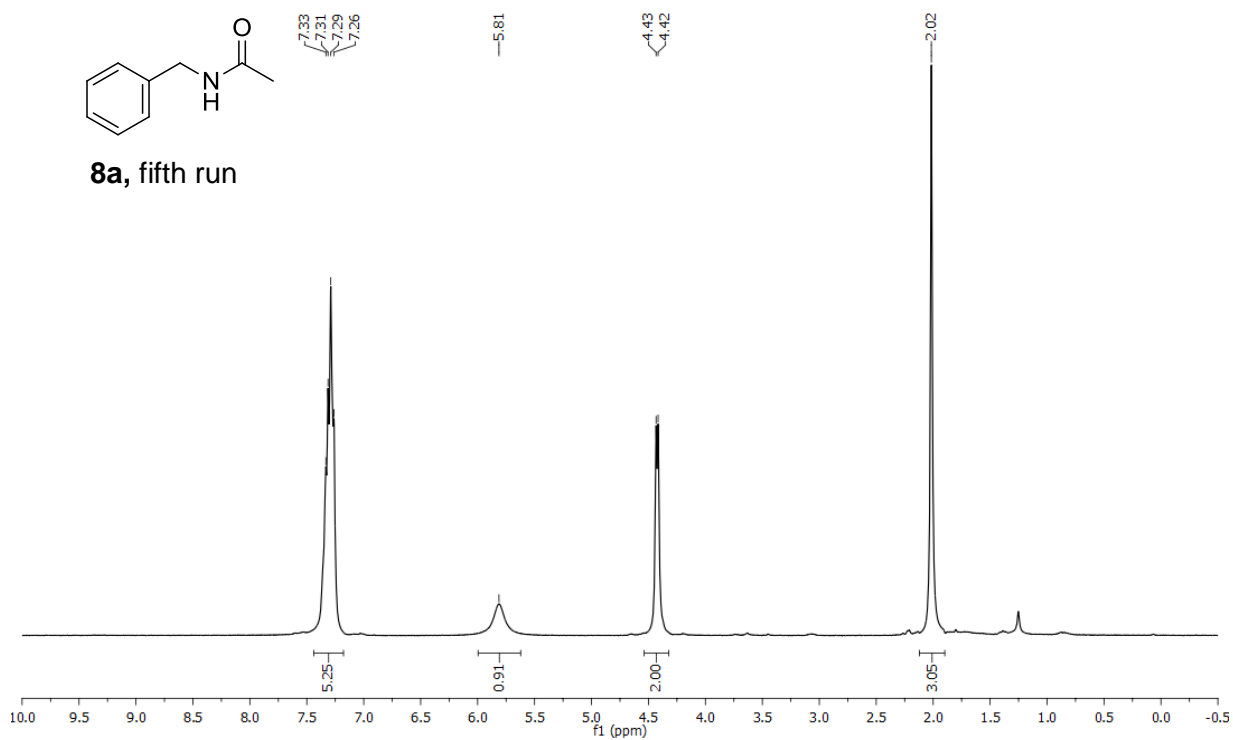
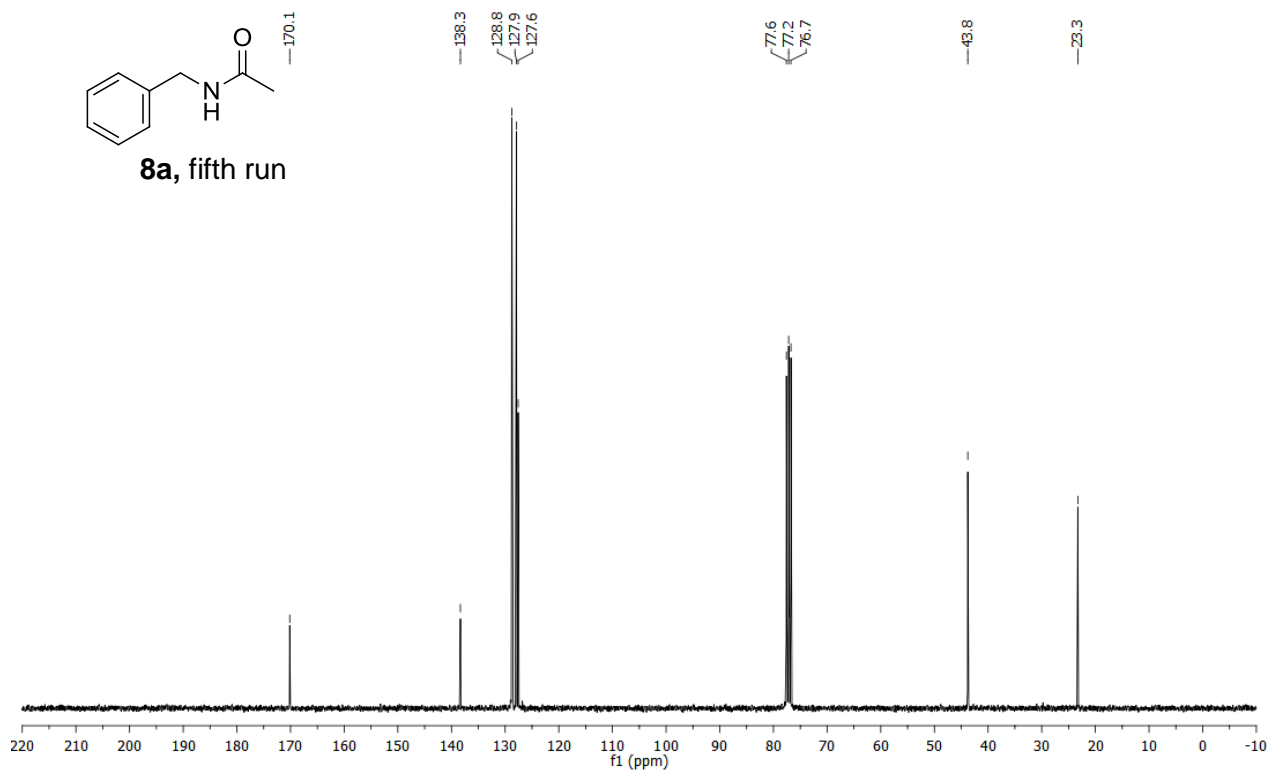
4. NMR spectra

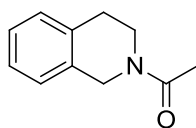
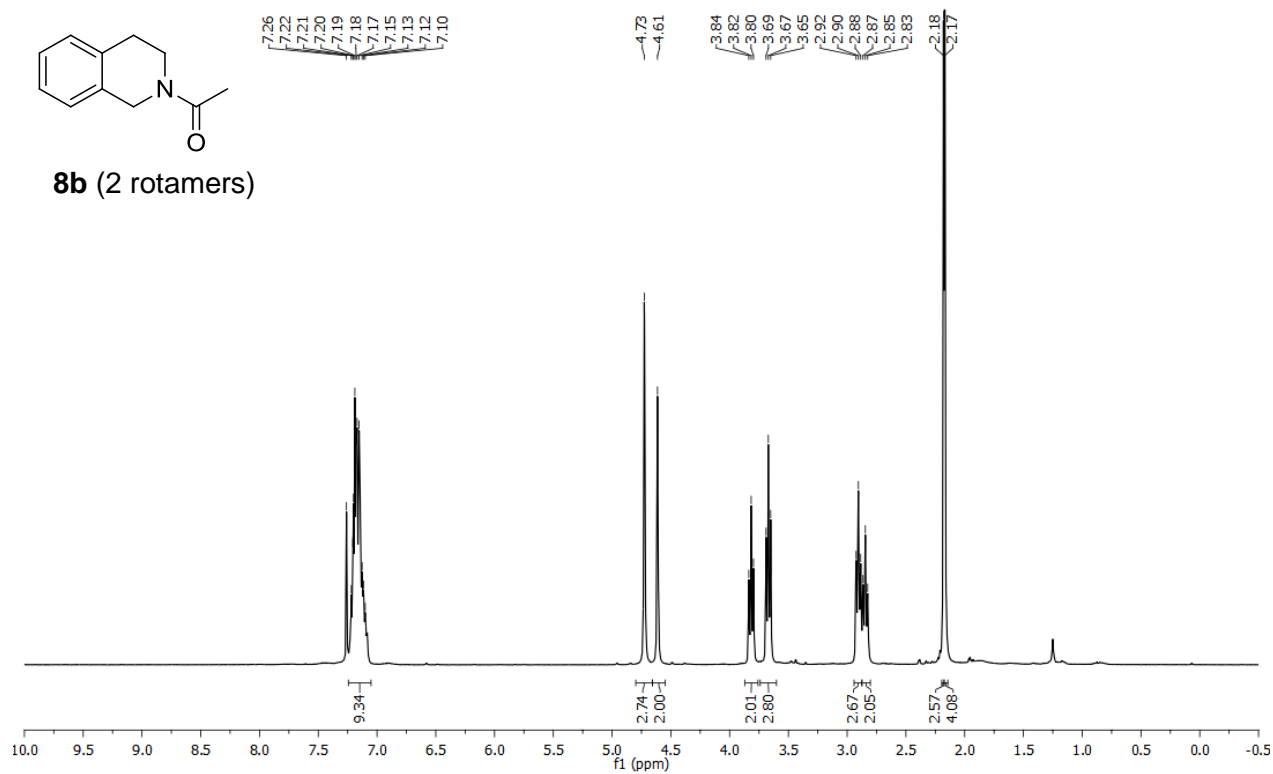
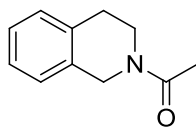
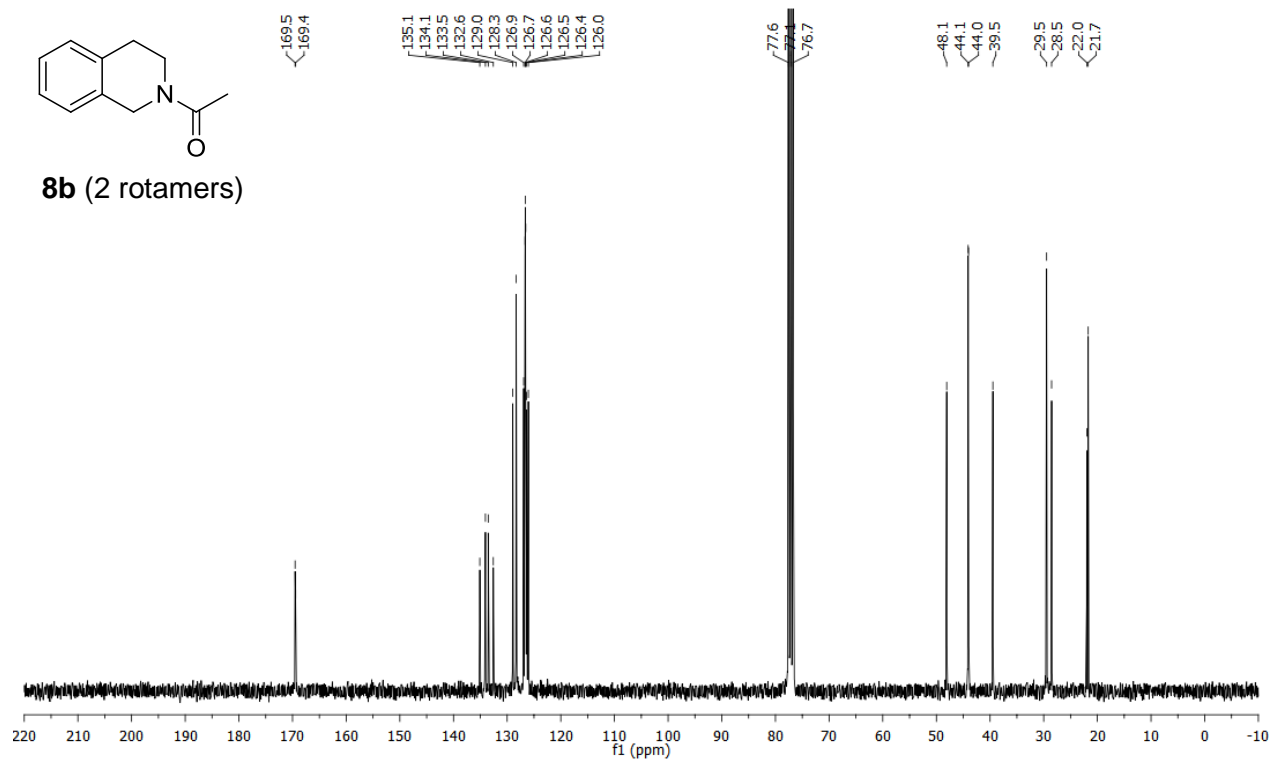
^1H -NMR (300 MHz, CDCl_3)

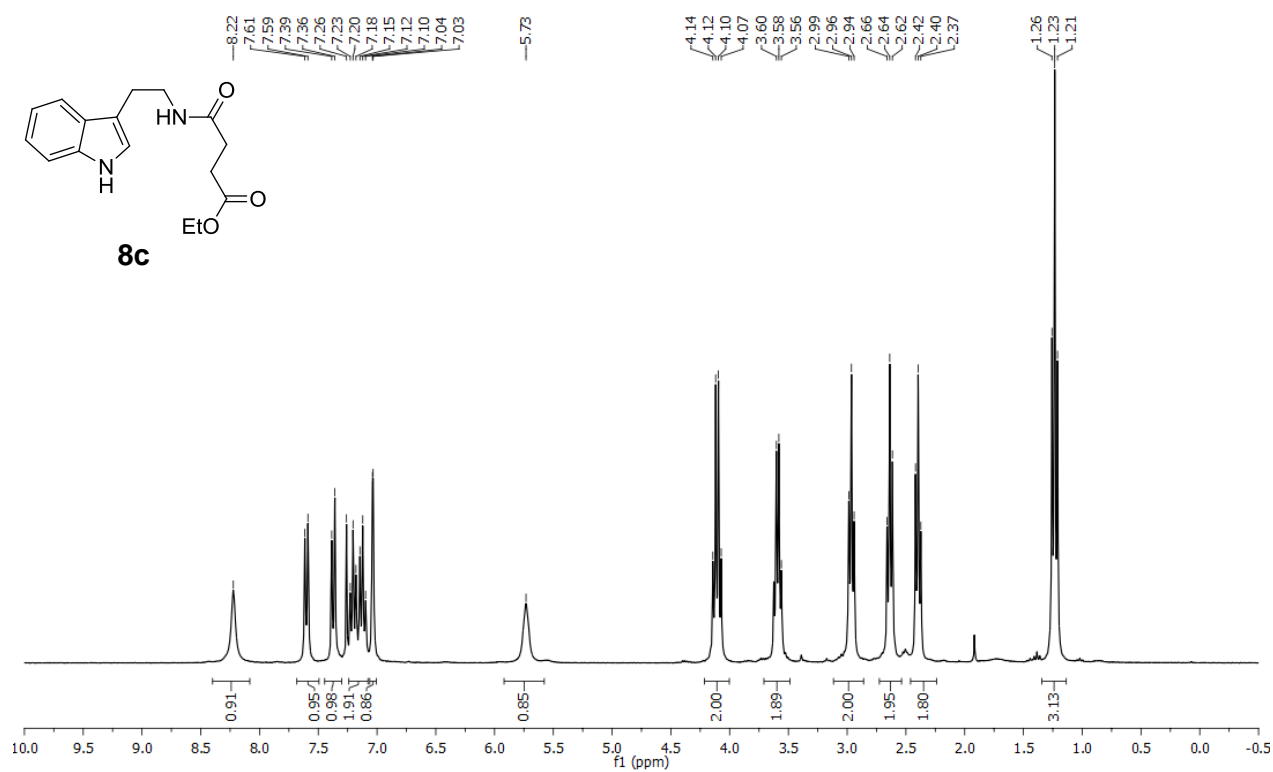
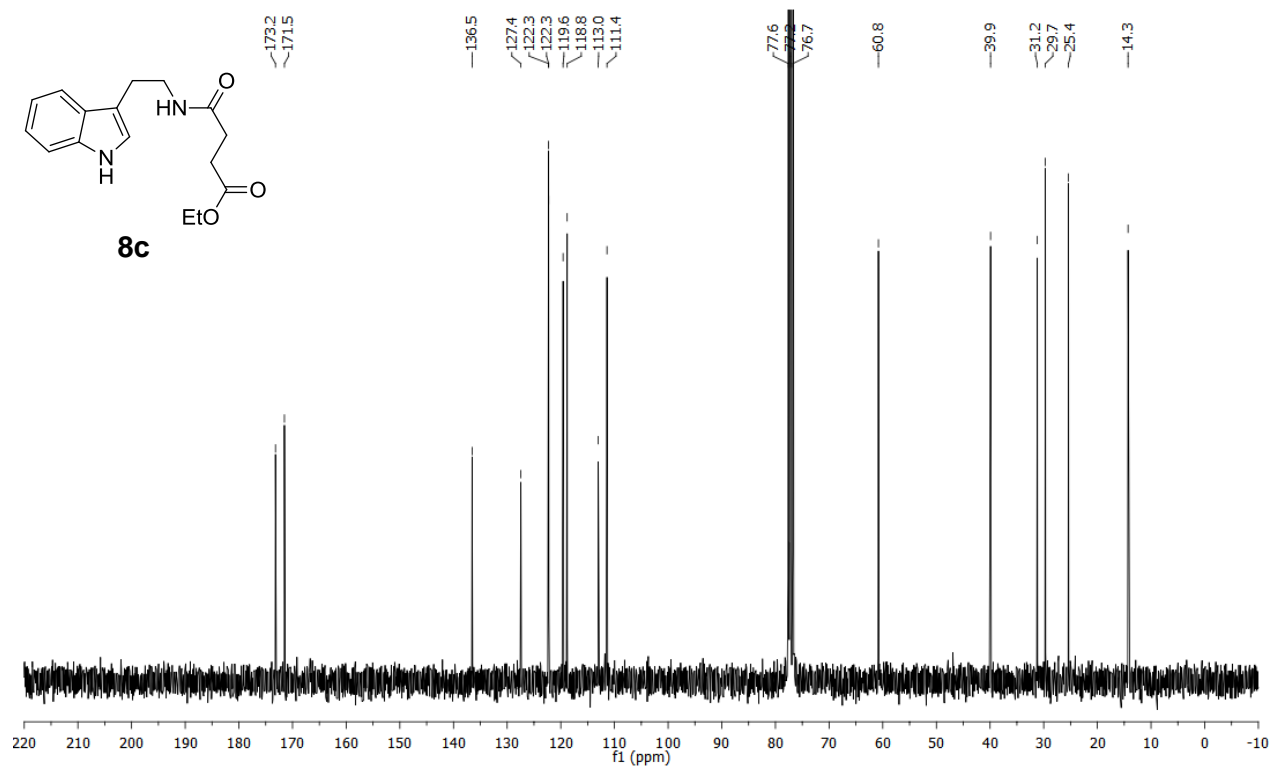


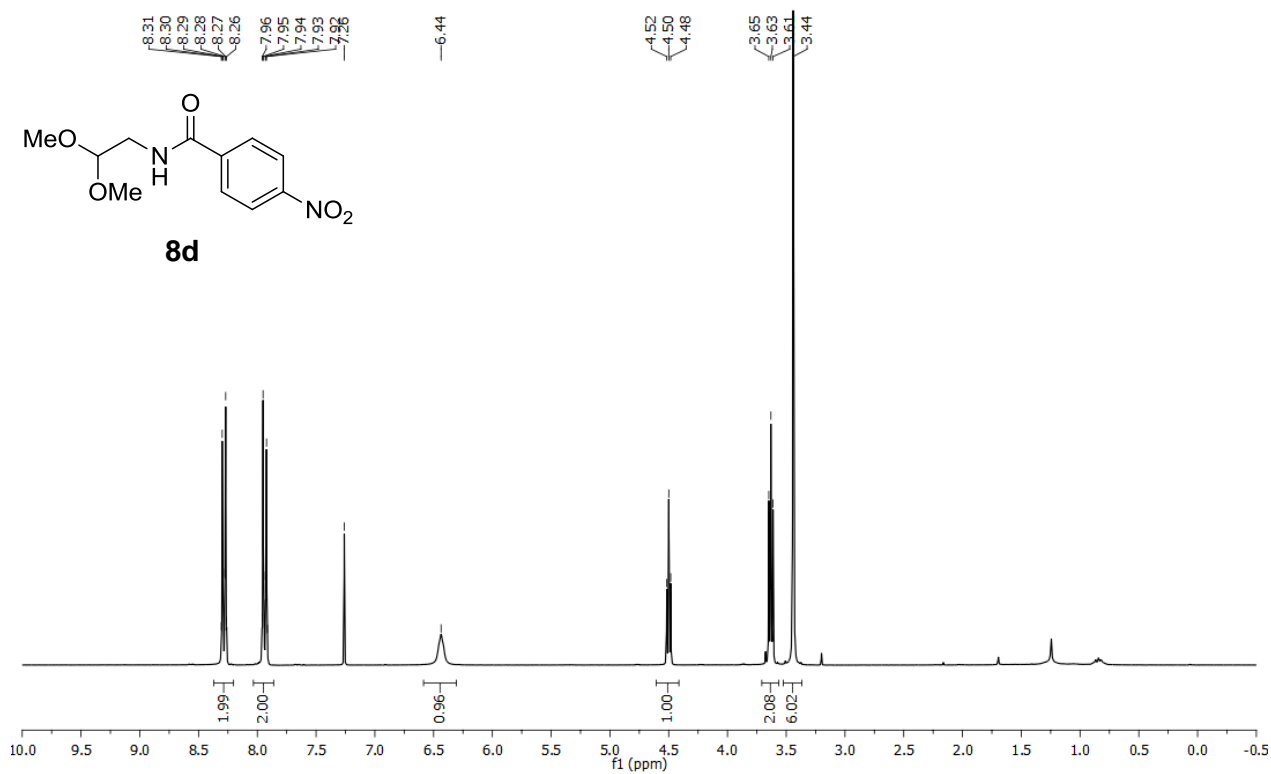
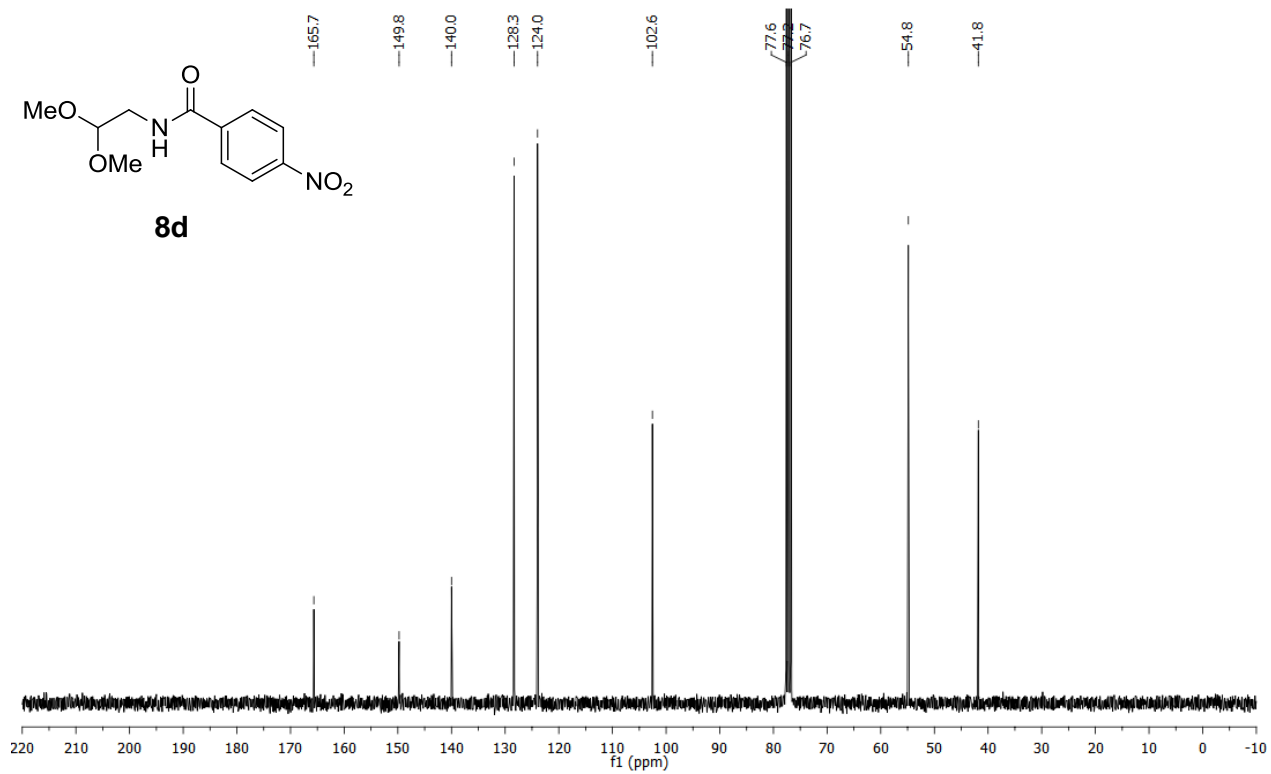
^1H -NMR (300 MHz, CDCl_3)

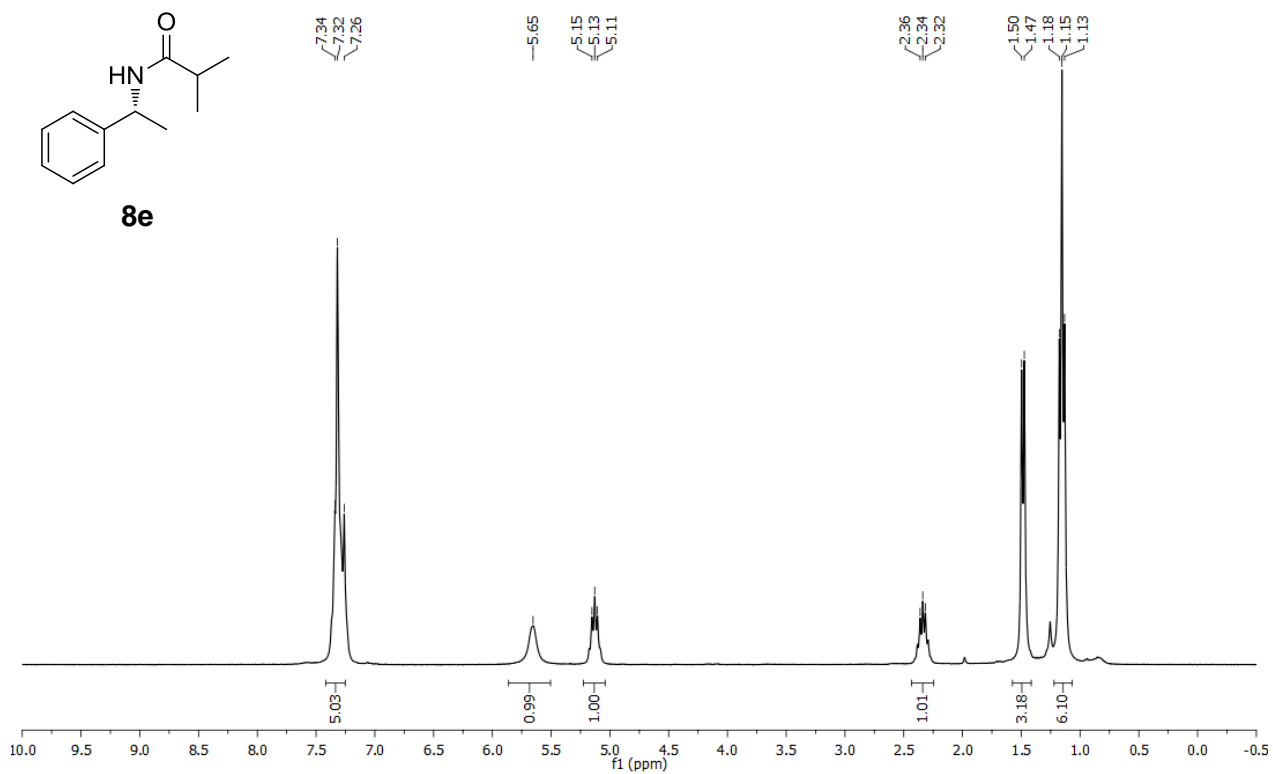
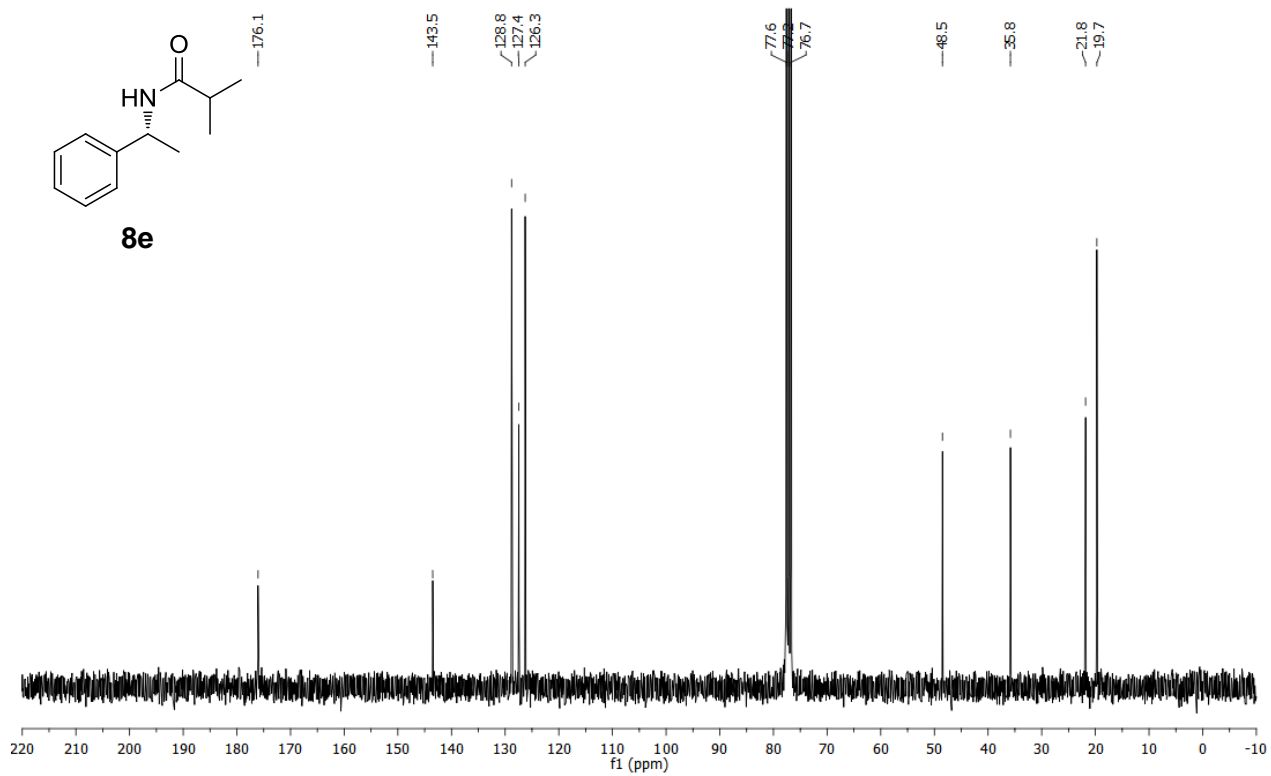


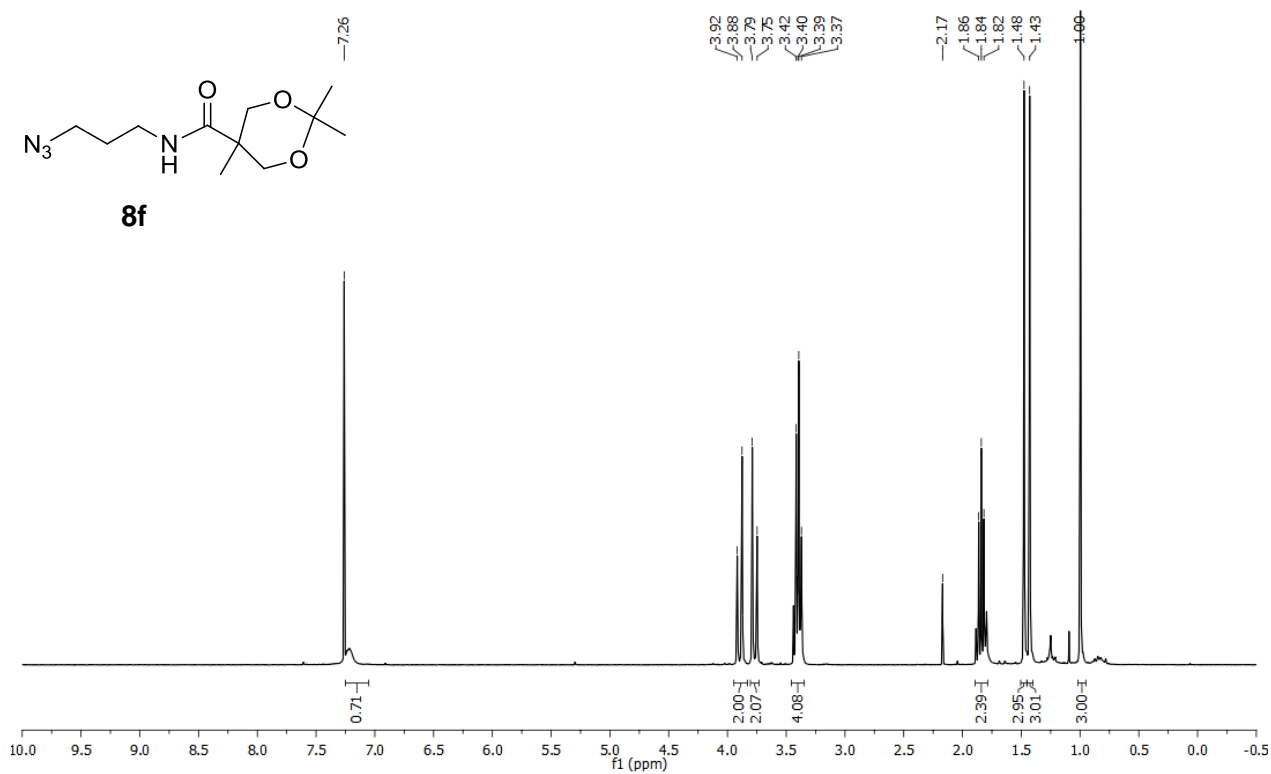
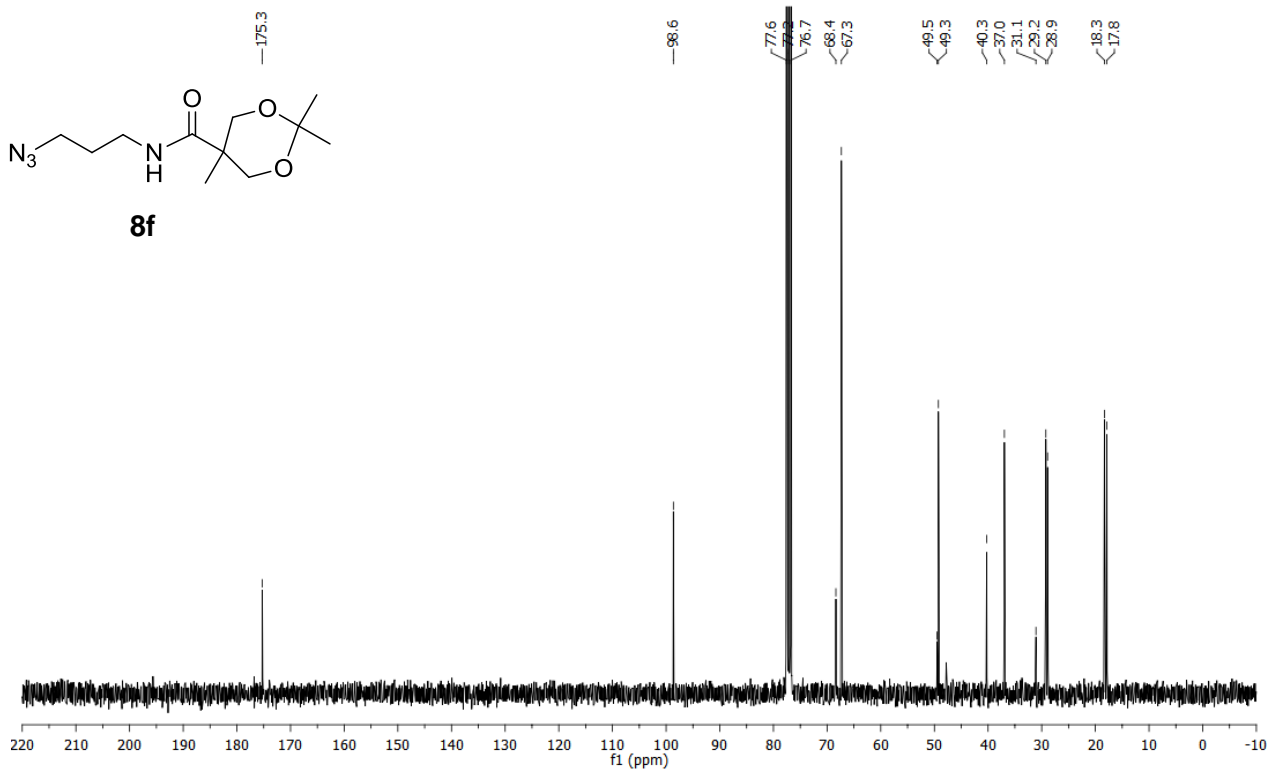
¹H-NMR (300 MHz, CDCl₃)¹³C-NMR (75.5 MHz, CDCl₃)

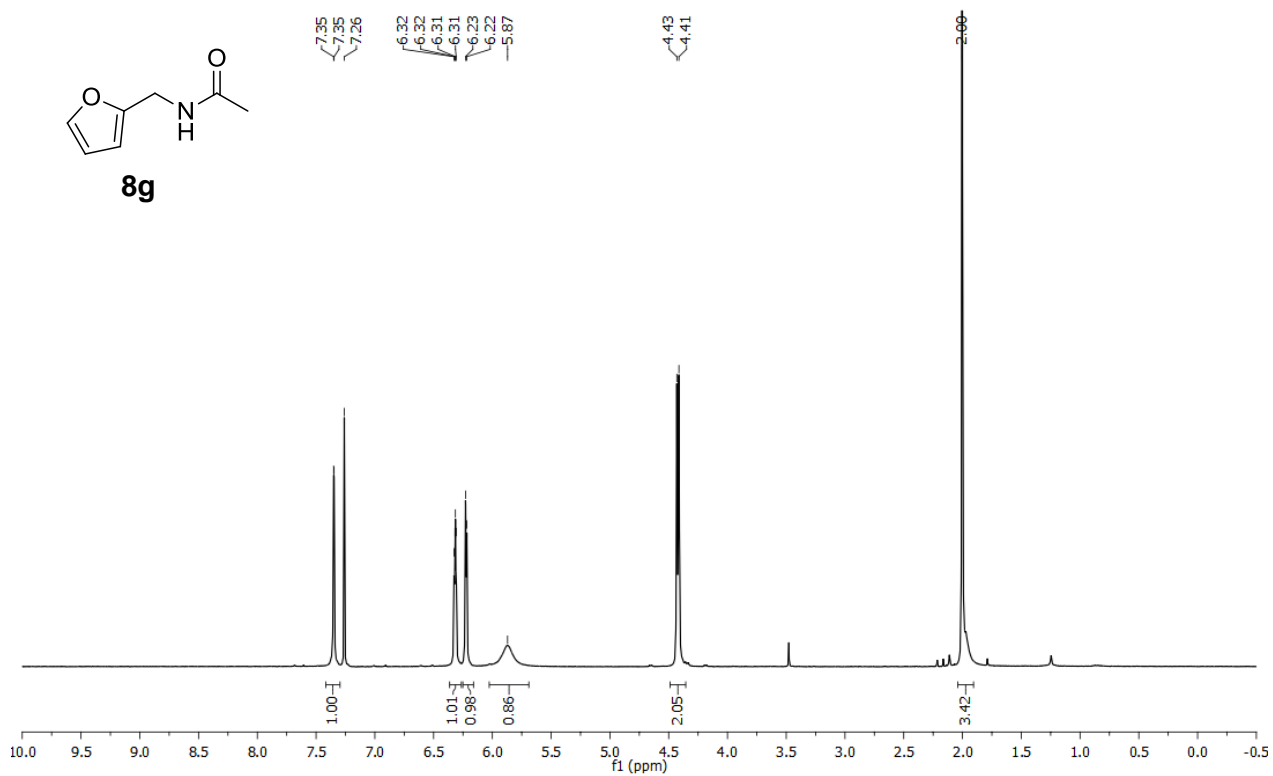
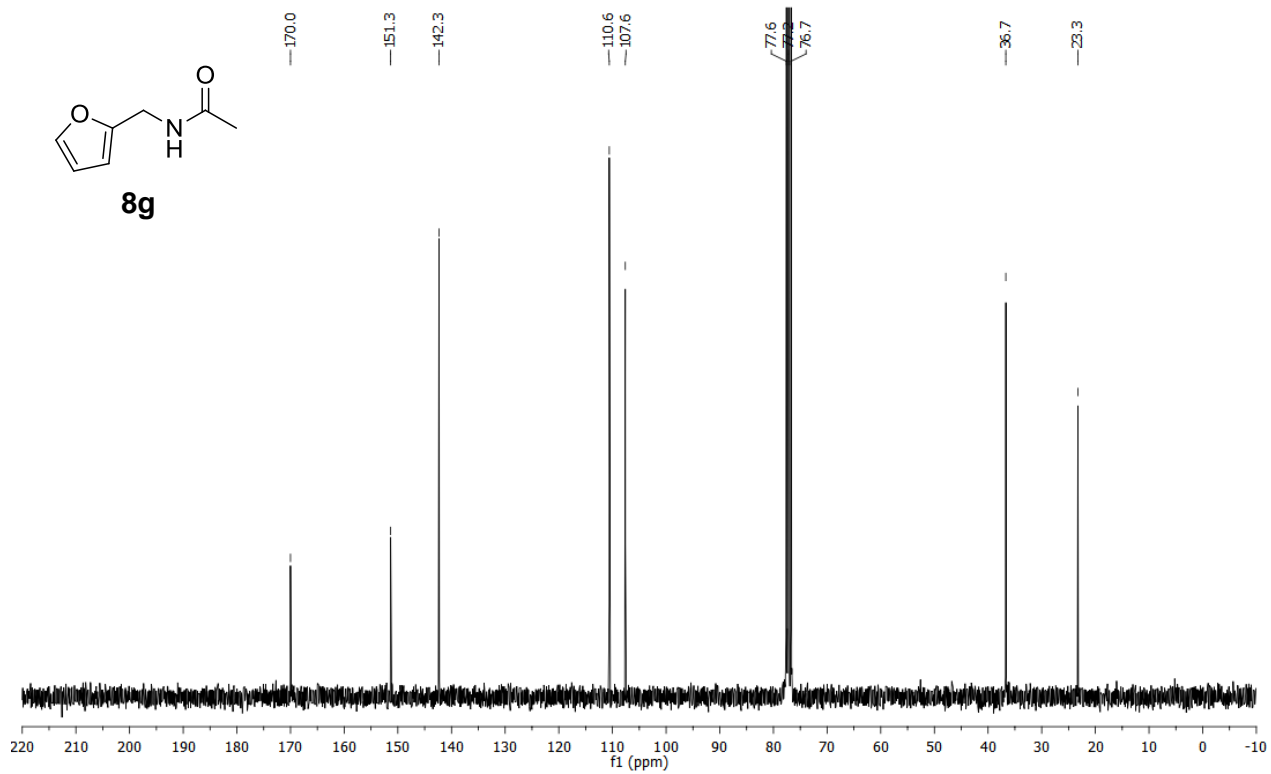
¹H-NMR (300 MHz, CDCl₃)**8b** (2 rotamers)¹³C-NMR (75.5 MHz, CDCl₃)**8b** (2 rotamers)

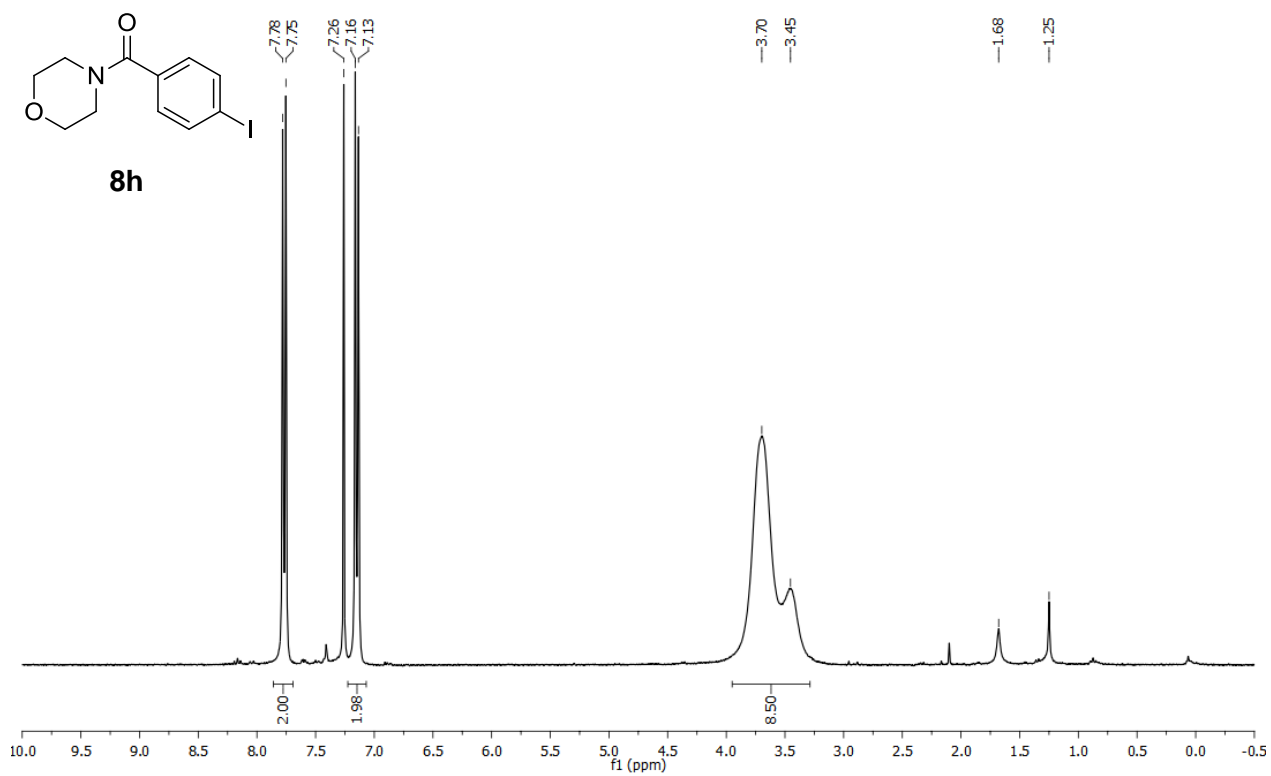
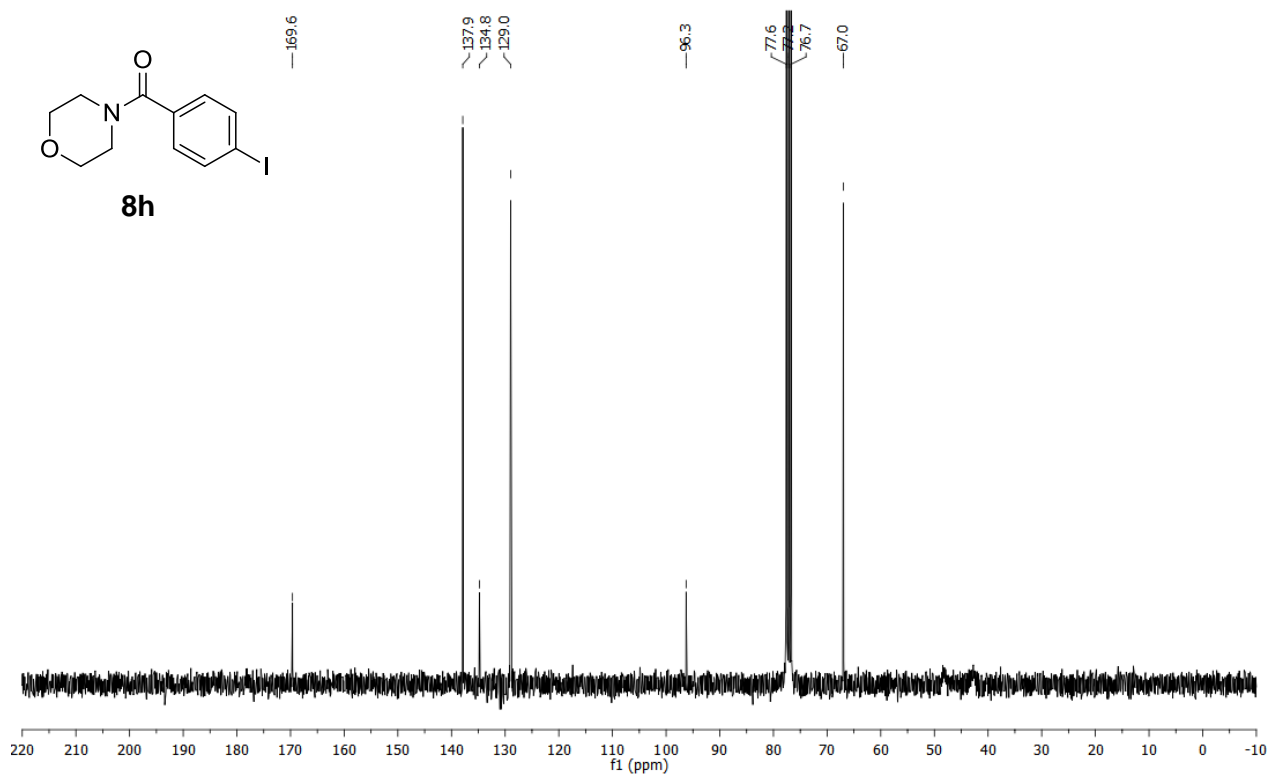
¹H-NMR (300 MHz, CDCl₃)¹³C-NMR (75.5 MHz, CDCl₃)

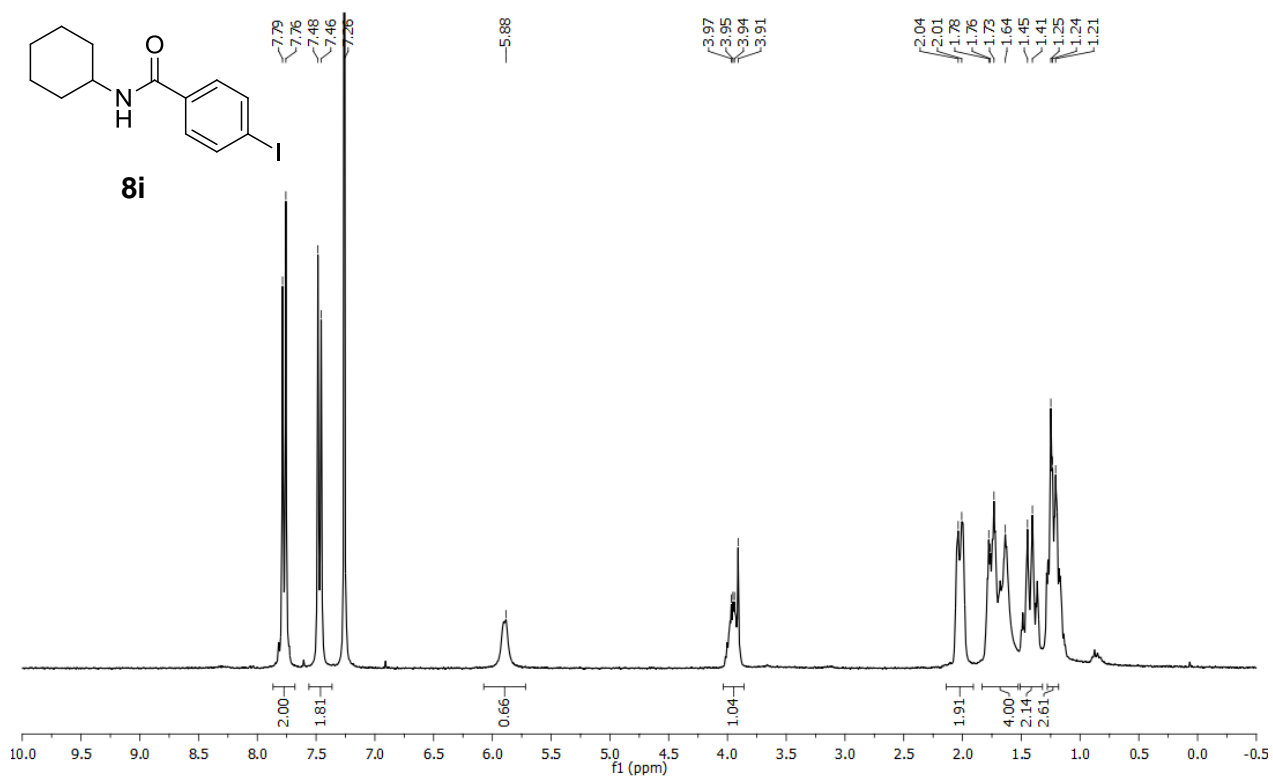
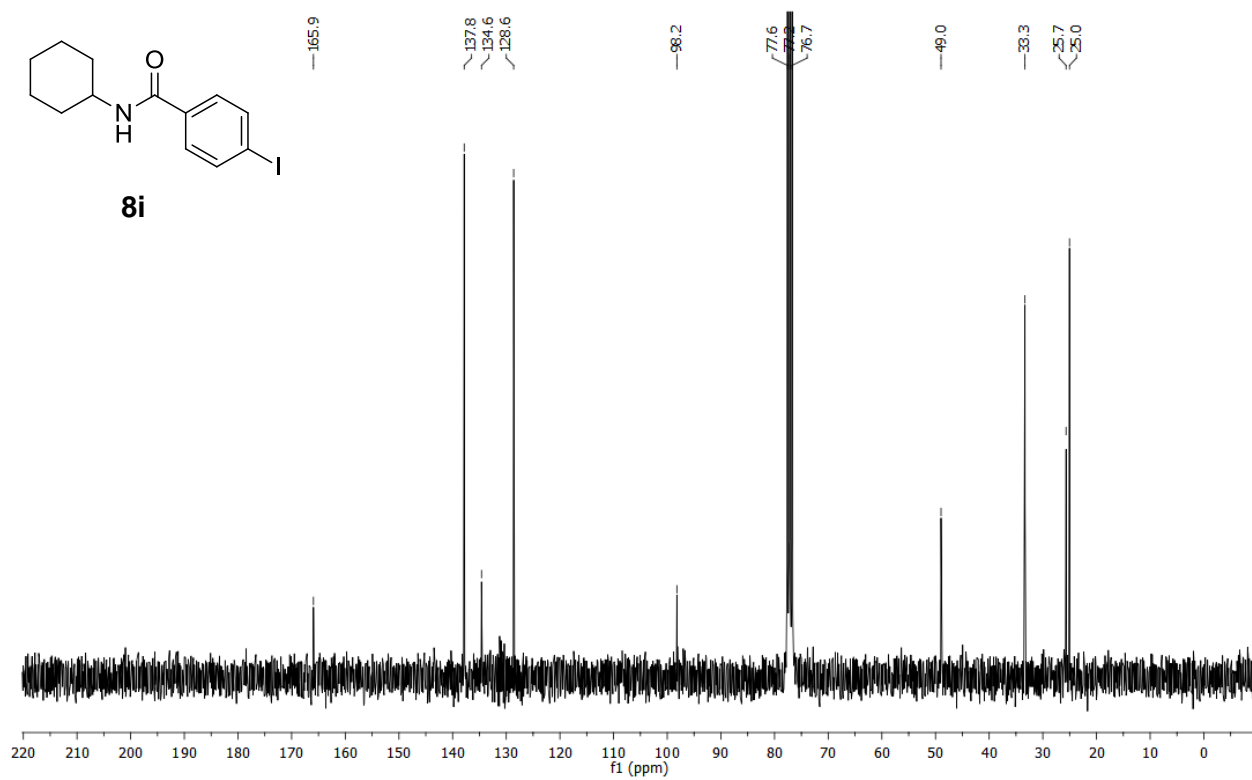
¹H-NMR (300 MHz, CDCl₃)¹³C-NMR (75.5 MHz, CDCl₃)

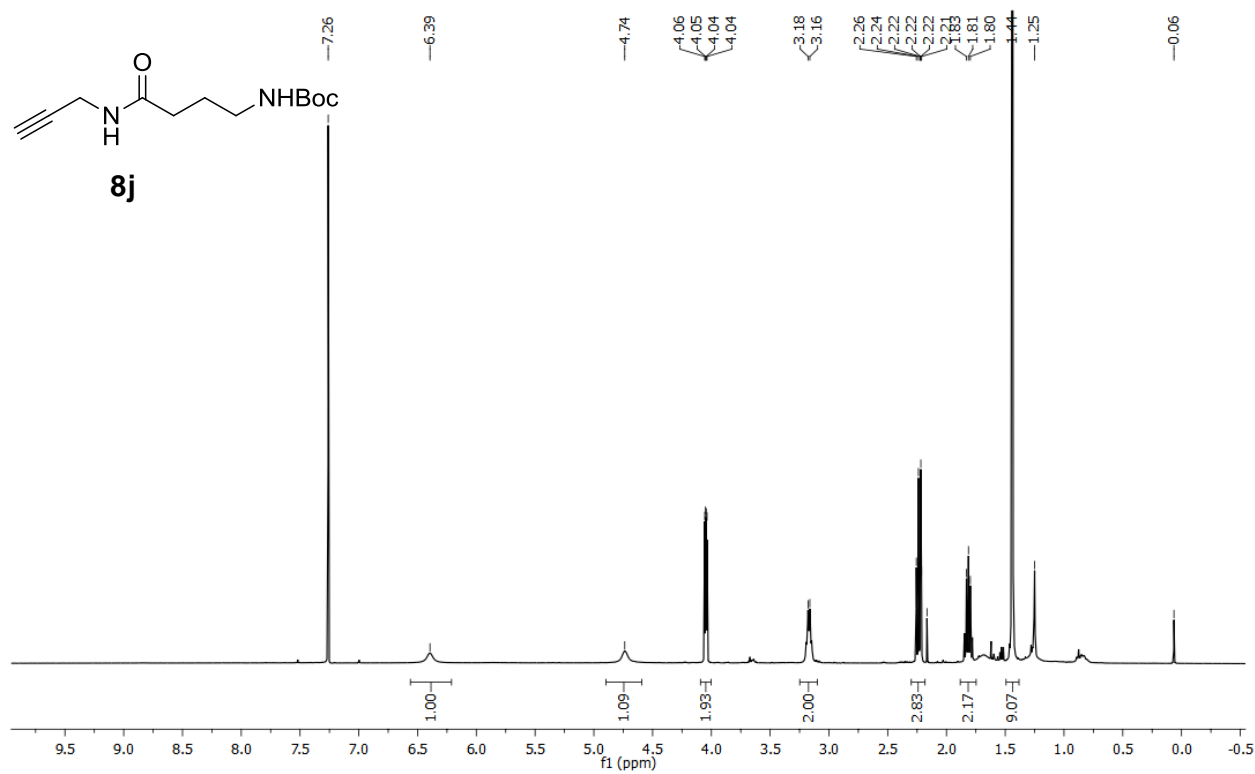
¹H-NMR (300 MHz, CDCl₃)¹³C-NMR (75.5 MHz, CDCl₃)

$^1\text{H-NMR}$ (300 MHz, CDCl_3) $^{13}\text{C-NMR}$ (75.5 MHz, CDCl_3)

$^1\text{H-NMR}$ (300 MHz, CDCl_3) $^{13}\text{C-NMR}$ (75.5 MHz, CDCl_3)

¹H-NMR (300 MHz, CDCl₃)¹³C-NMR (75.5 MHz, CDCl₃)

¹H-NMR (300 MHz, CDCl₃)¹³C-NMR (75.5 MHz, CDCl₃)

¹H-NMR (400 MHz, CDCl₃)¹³C-NMR (100.6 MHz, CDCl₃)