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

Phenyliodonium Diacetate Mediated Carbotrifluoromethylation of *N*-Acylhydrazones

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Supporting Information

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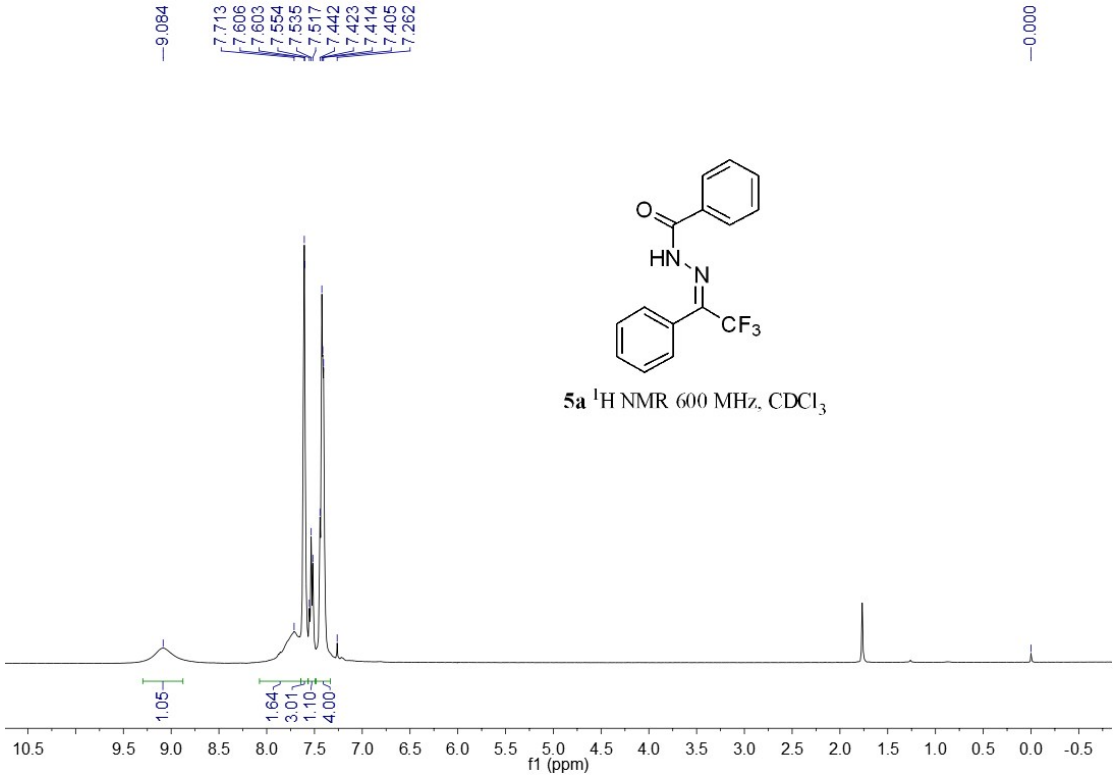
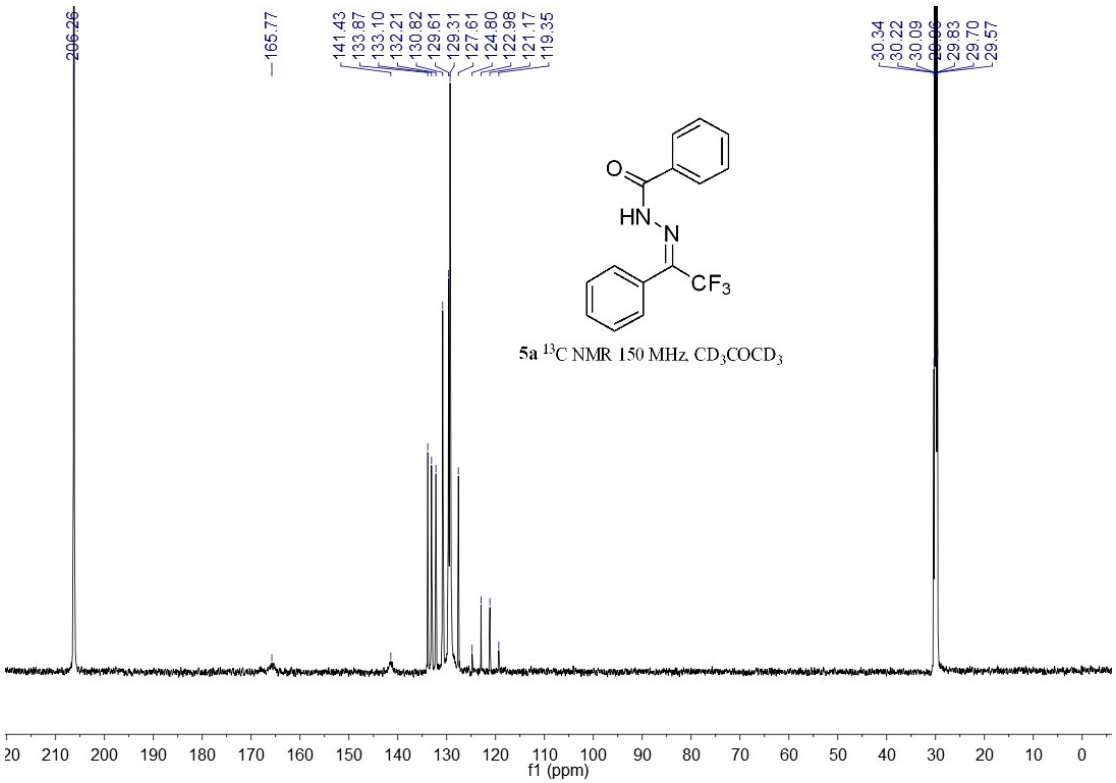
1. Spectra copies of all compounds and control experiments.

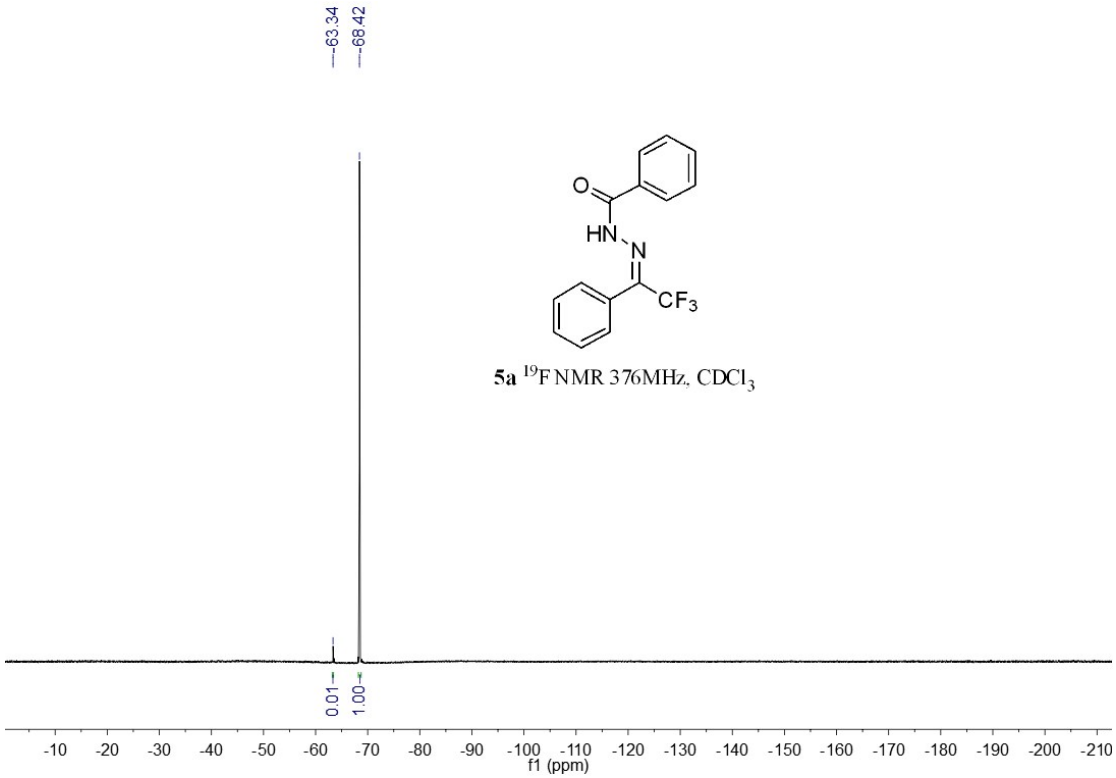
NMR copies of major product of compound **5a**:



5a ^{1}H NMR 600 MHz, $CDCl_3$

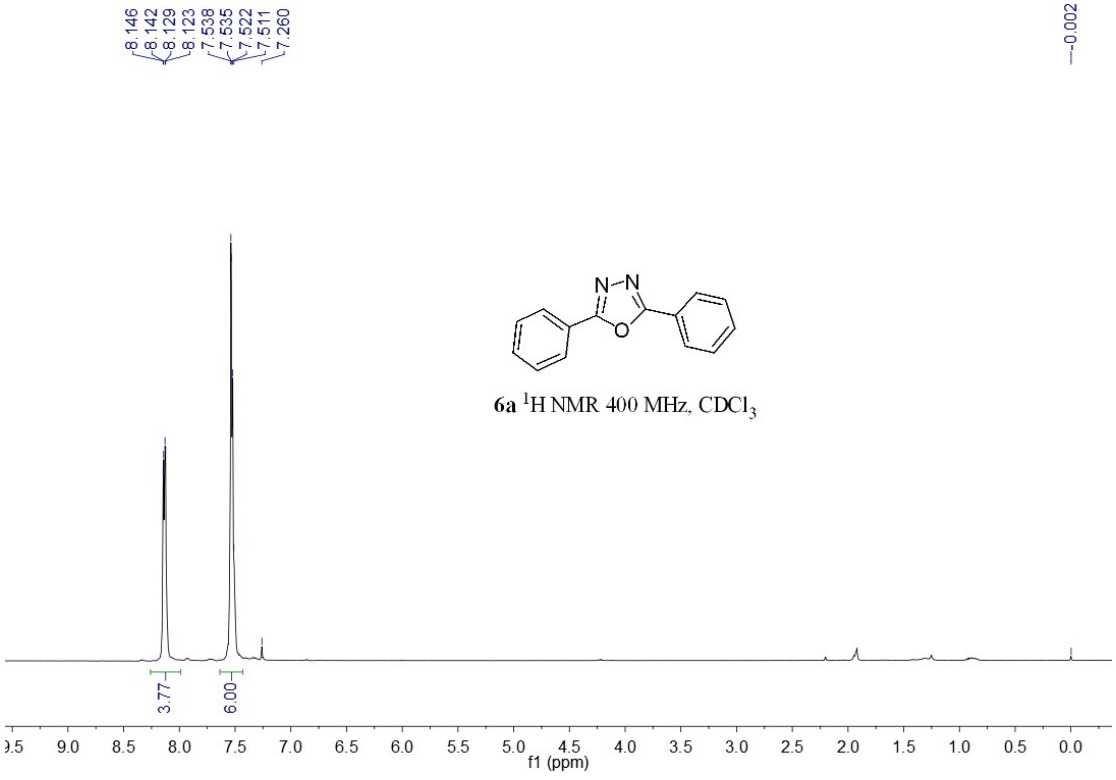


5a ^{13}C NMR 150 MHz, CD_3COCD_3

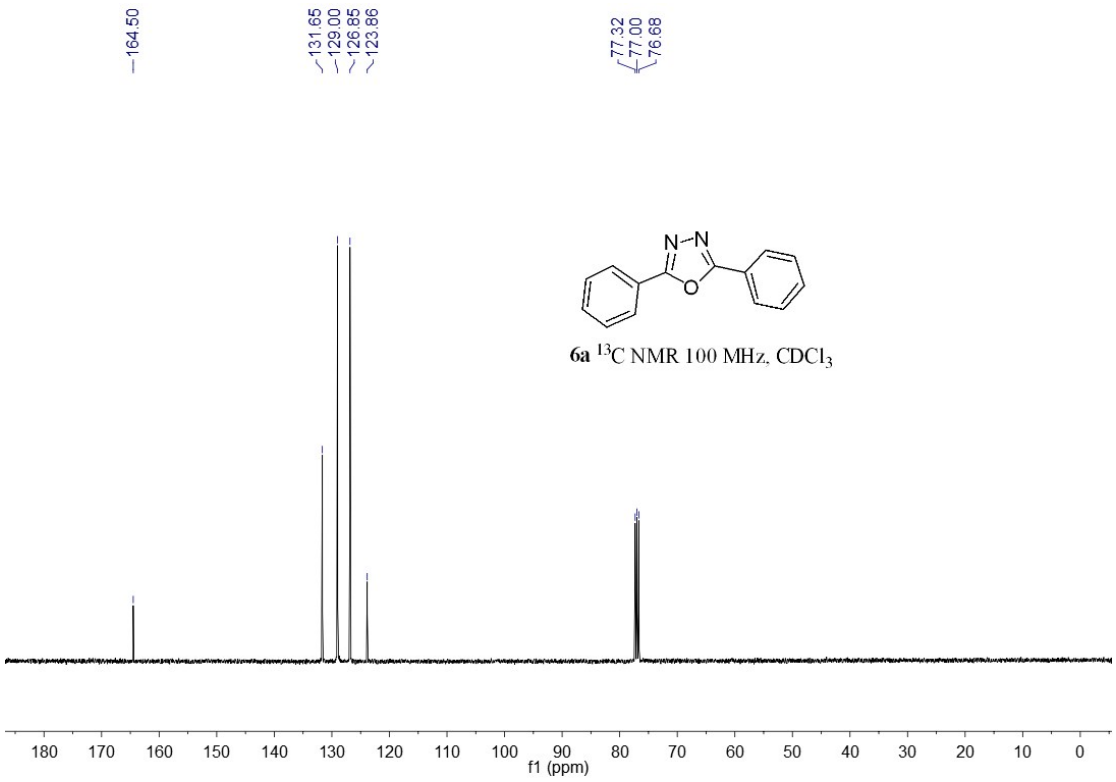


5a ^{19}F NMR 376MHz, $CDCl_3$

NMR copies of major product of compound **6a**:

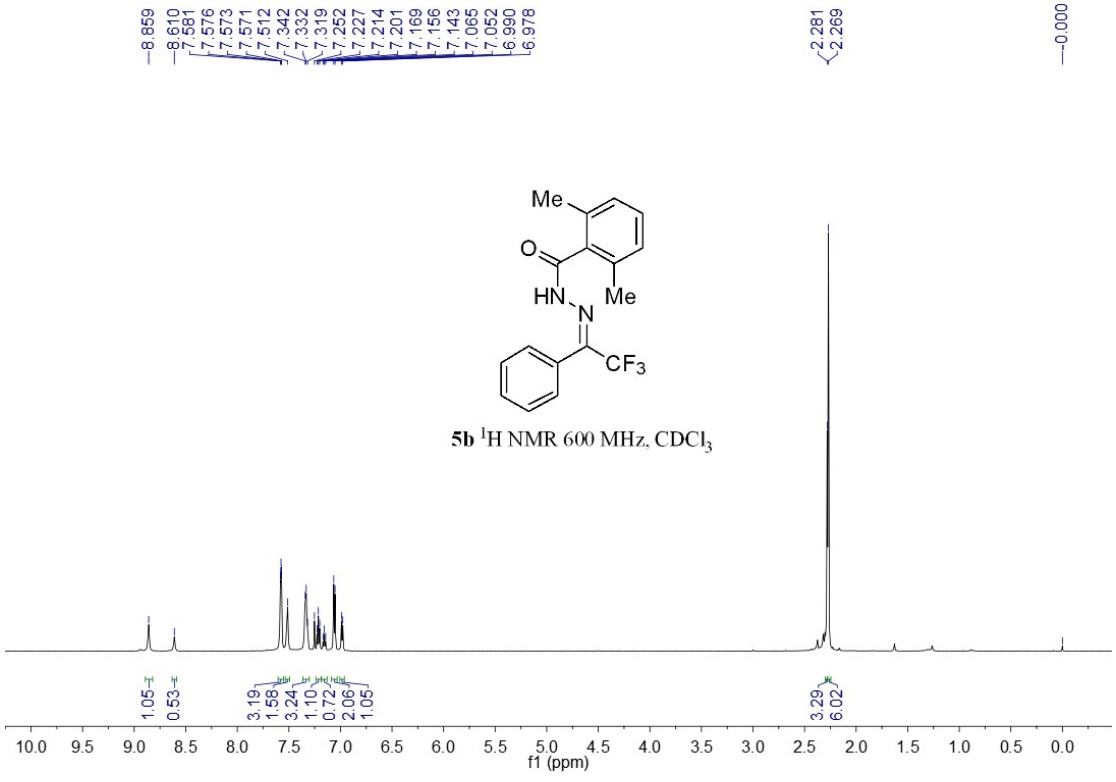


6a ^{1}H NMR 400 MHz, $CDCl_3$

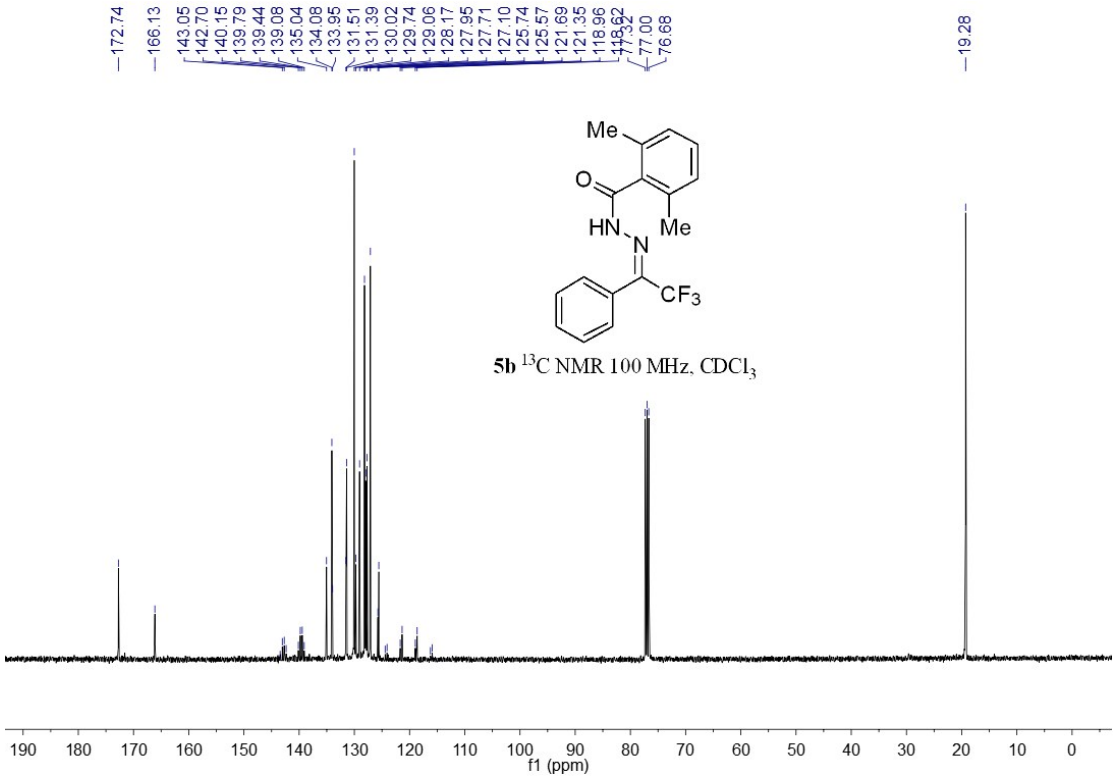


6a ^{13}C NMR 100 MHz, $CDCl_3$

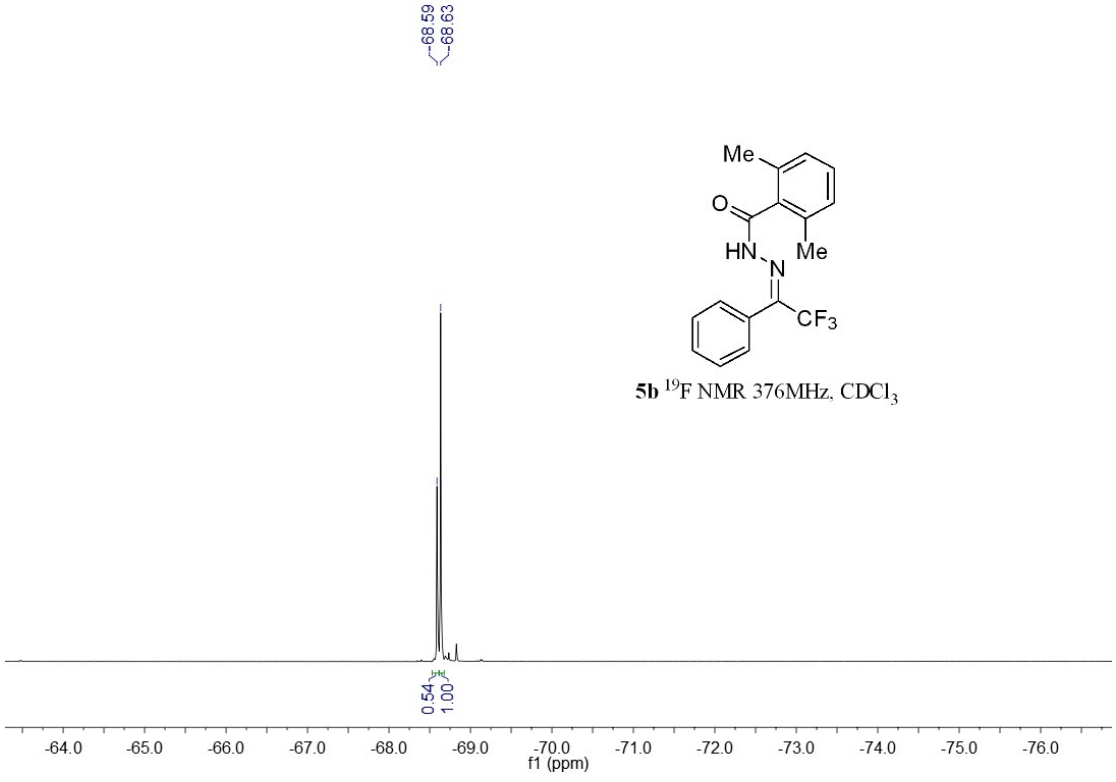
NMR copies of major product of compound **5b**:



5b ^{1}H NMR 600 MHz, $CDCl_3$

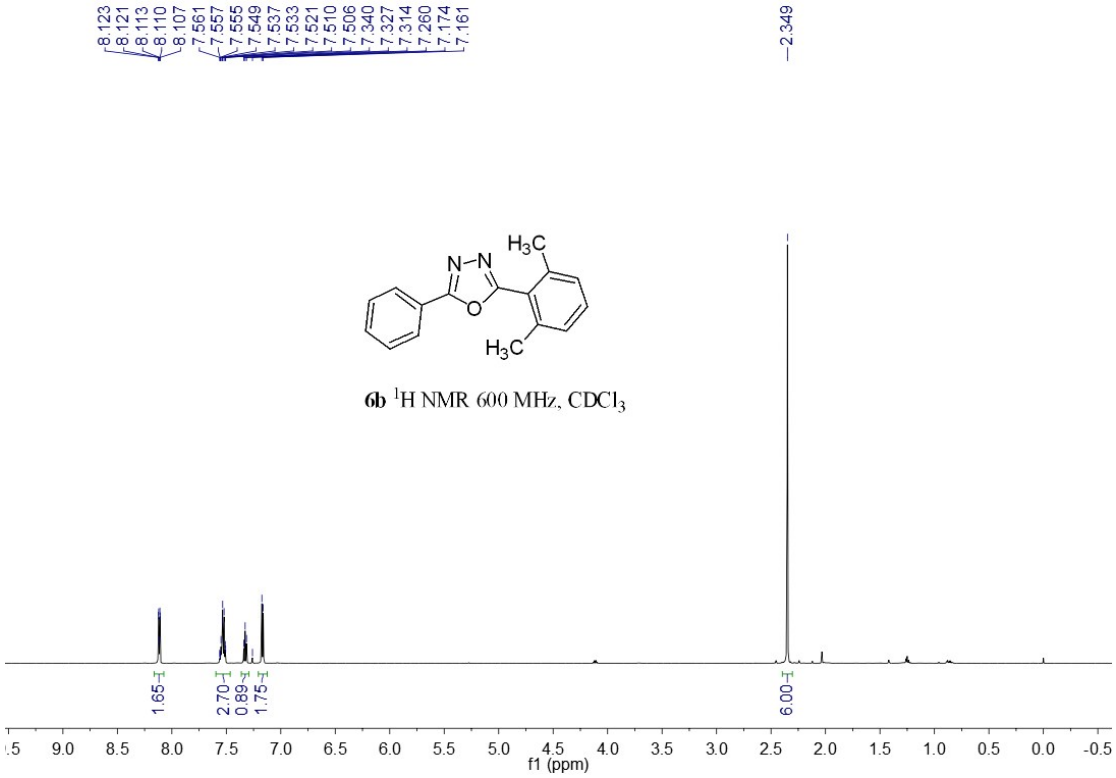


5b ^{13}C NMR 100 MHz, $CDCl_3$

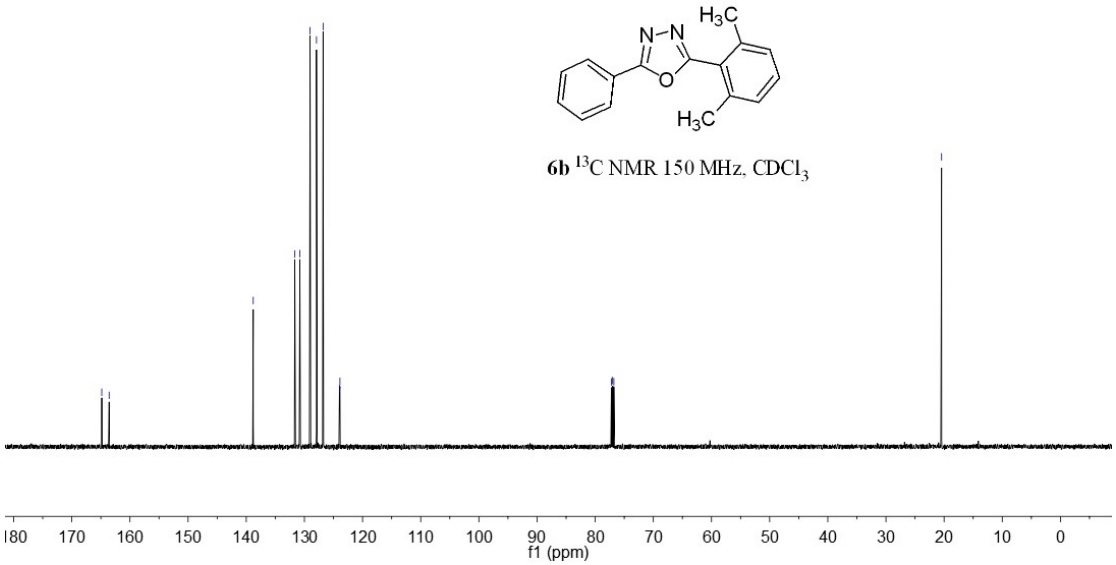


5b ^{19}F NMR 376MHz, $CDCl_3$

NMR copies of major product of compound **6b**:

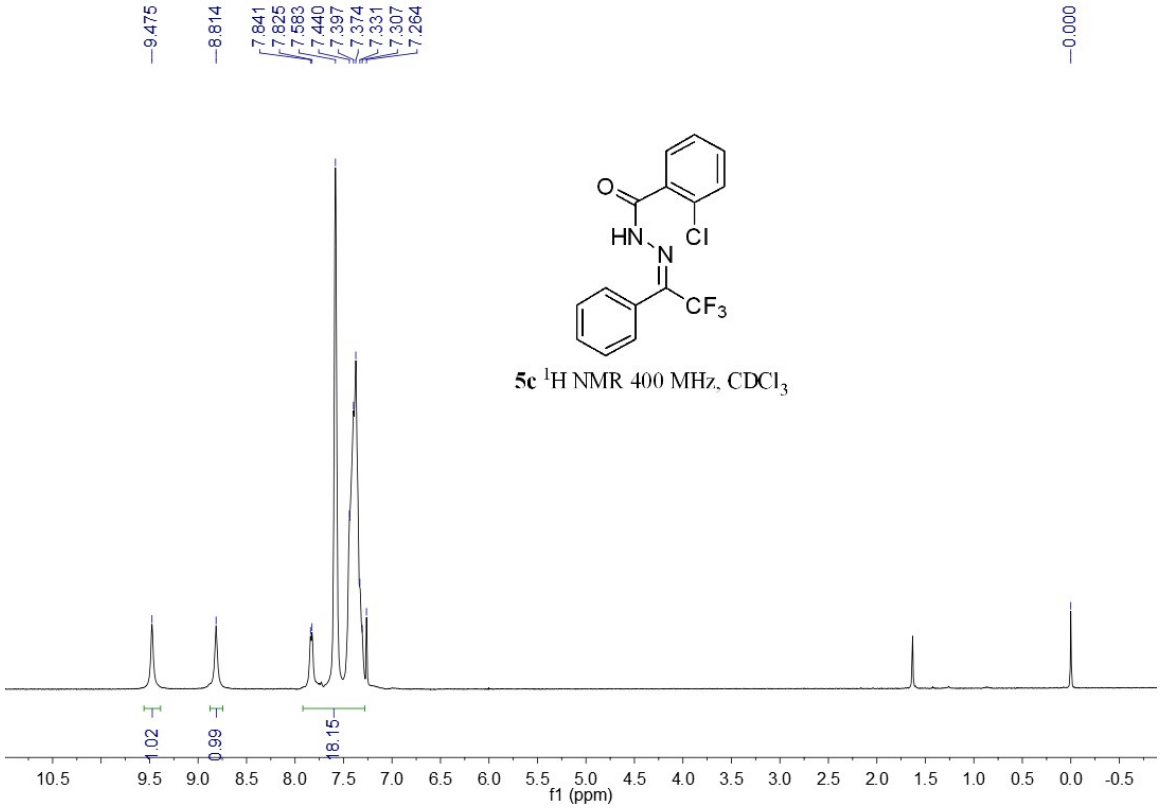


6b ^{1}H NMR 600 MHz, $CDCl_3$

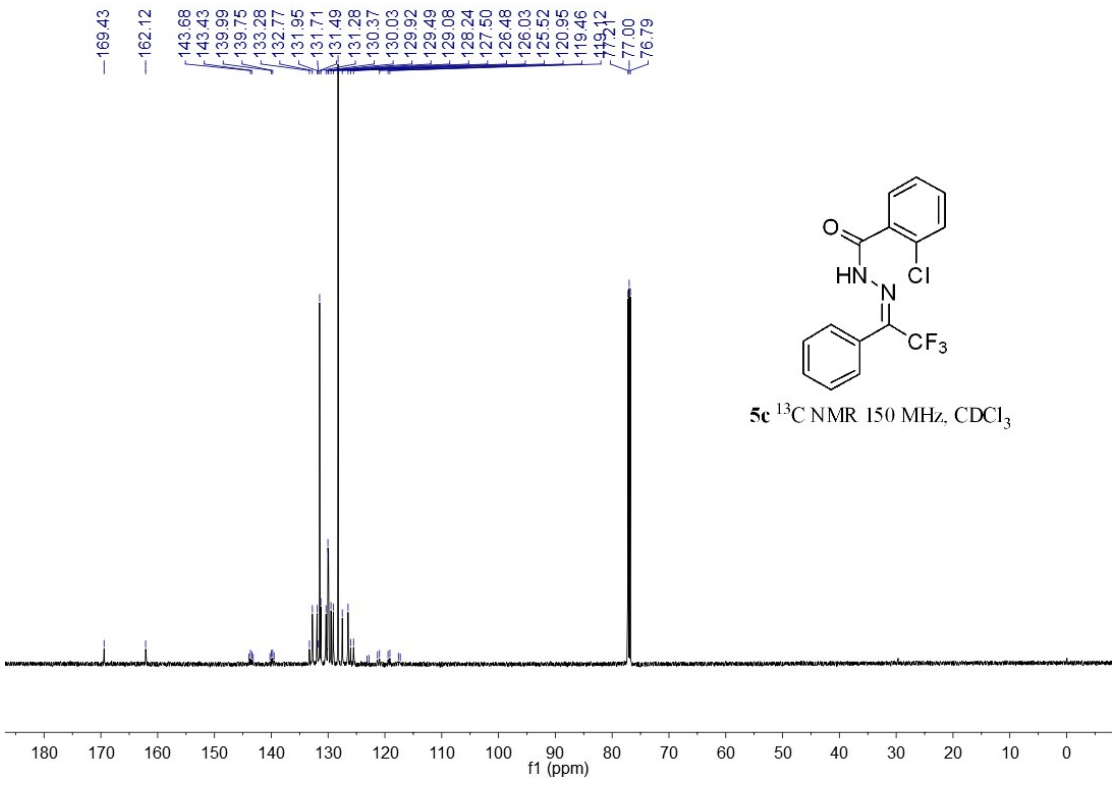


6b ^{13}C NMR 150 MHz, $CDCl_3$

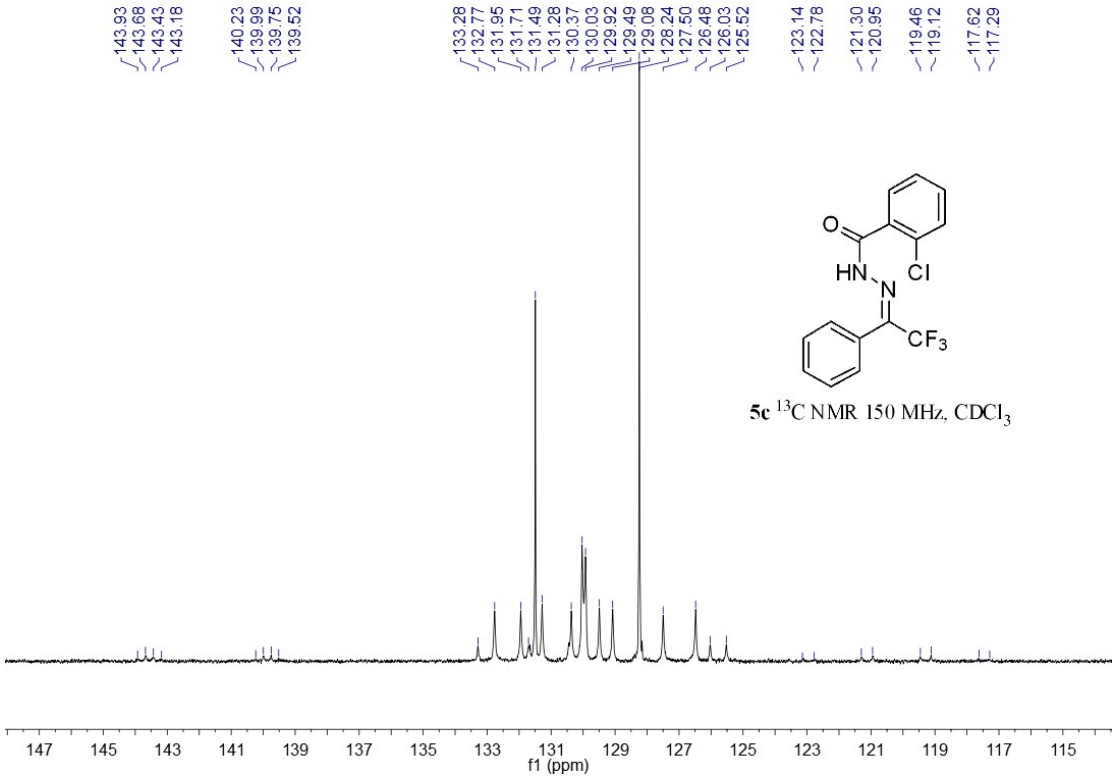
NMR copies of major product of compound **5c**:



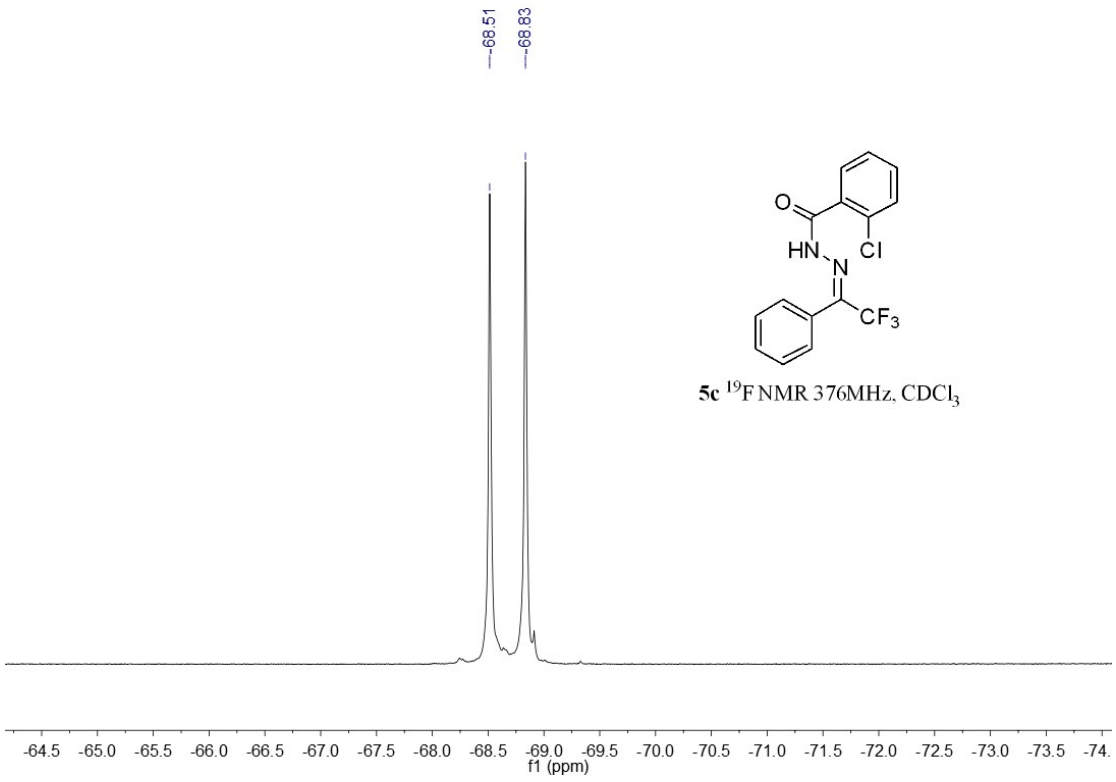
5c ^{1}H NMR 400 MHz, $CDCl_3$



5c ^{13}C NMR 150 MHz, $CDCl_3$

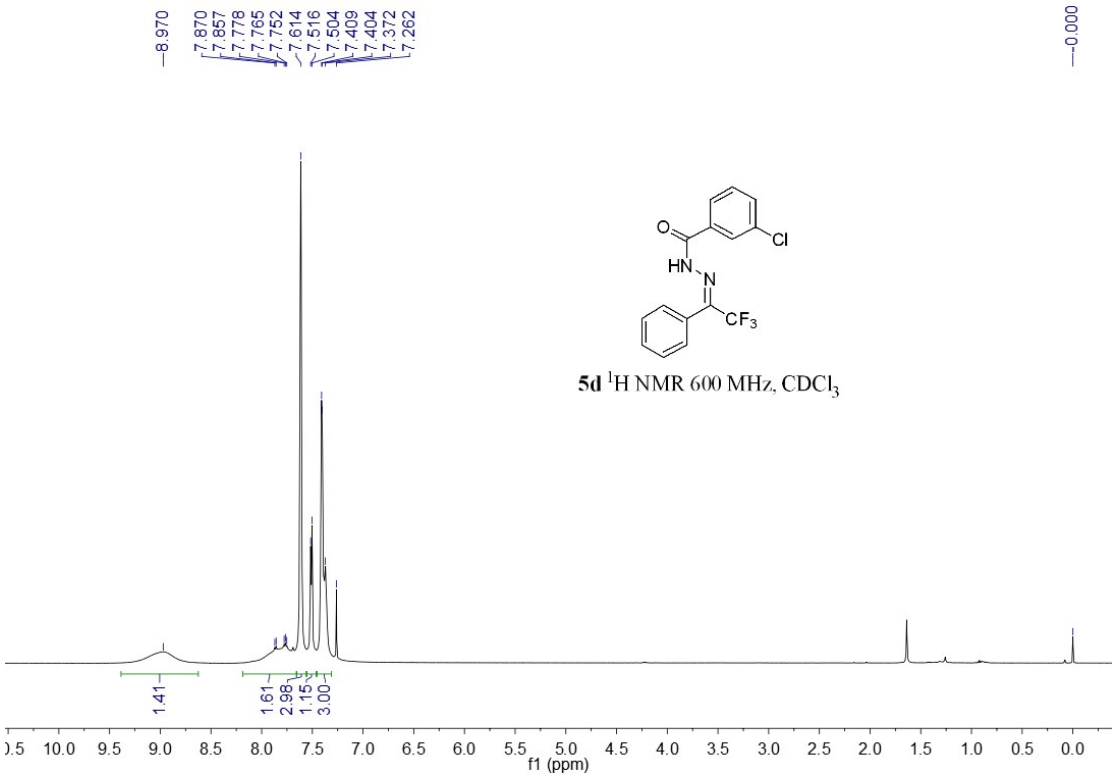


5c ^{13}C NMR 150 MHz, $CDCl_3$

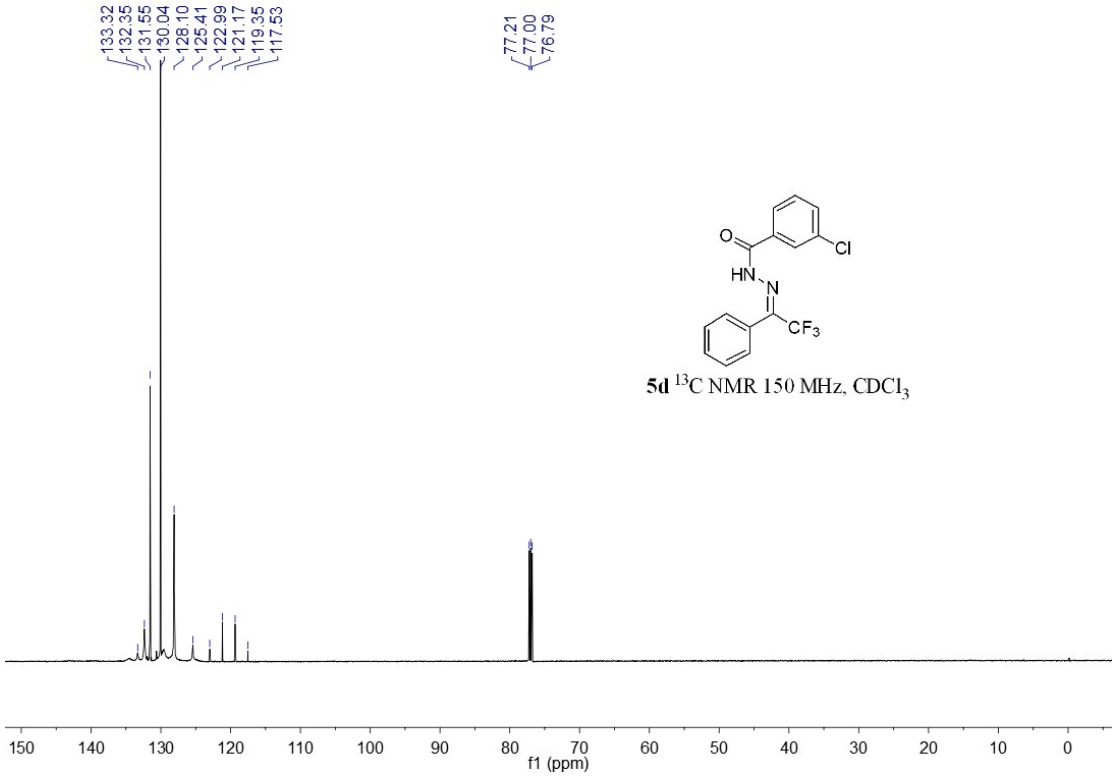


5c ^{19}F NMR 376MHz, $CDCl_3$

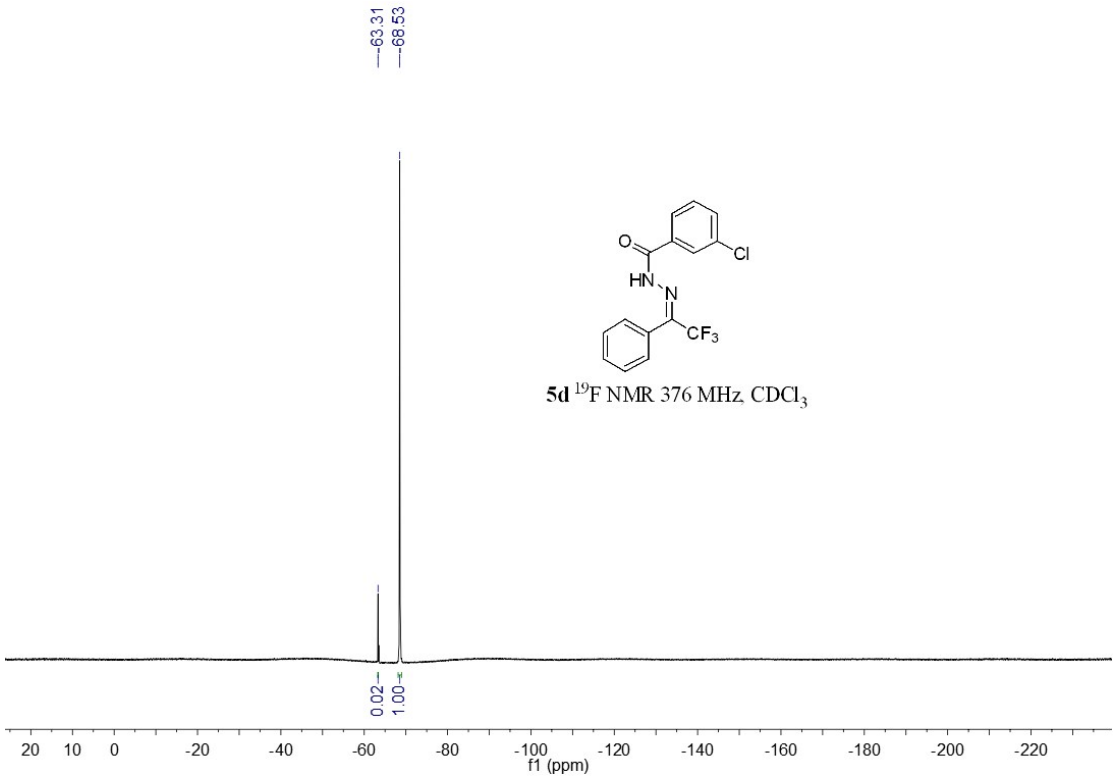
NMR copies of major product of compound **5d**:



5d ^{1}H NMR 600 MHz, $CDCl_3$

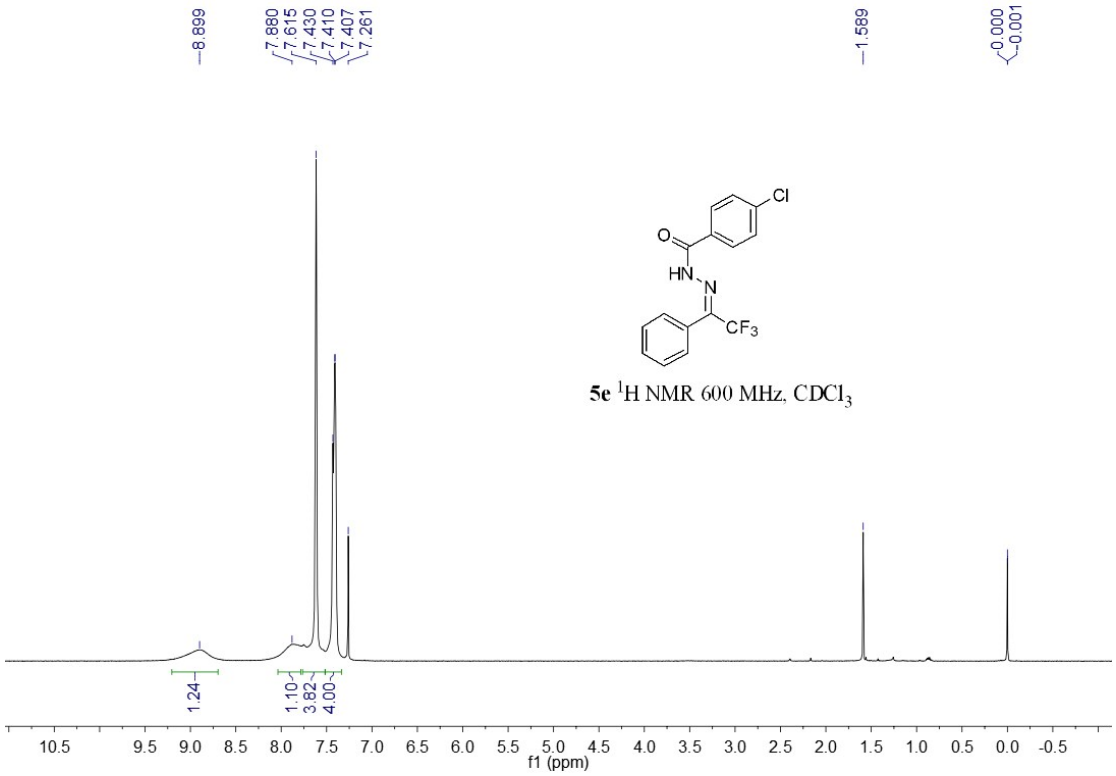


5d ^{13}C NMR 150 MHz, $CDCl_3$

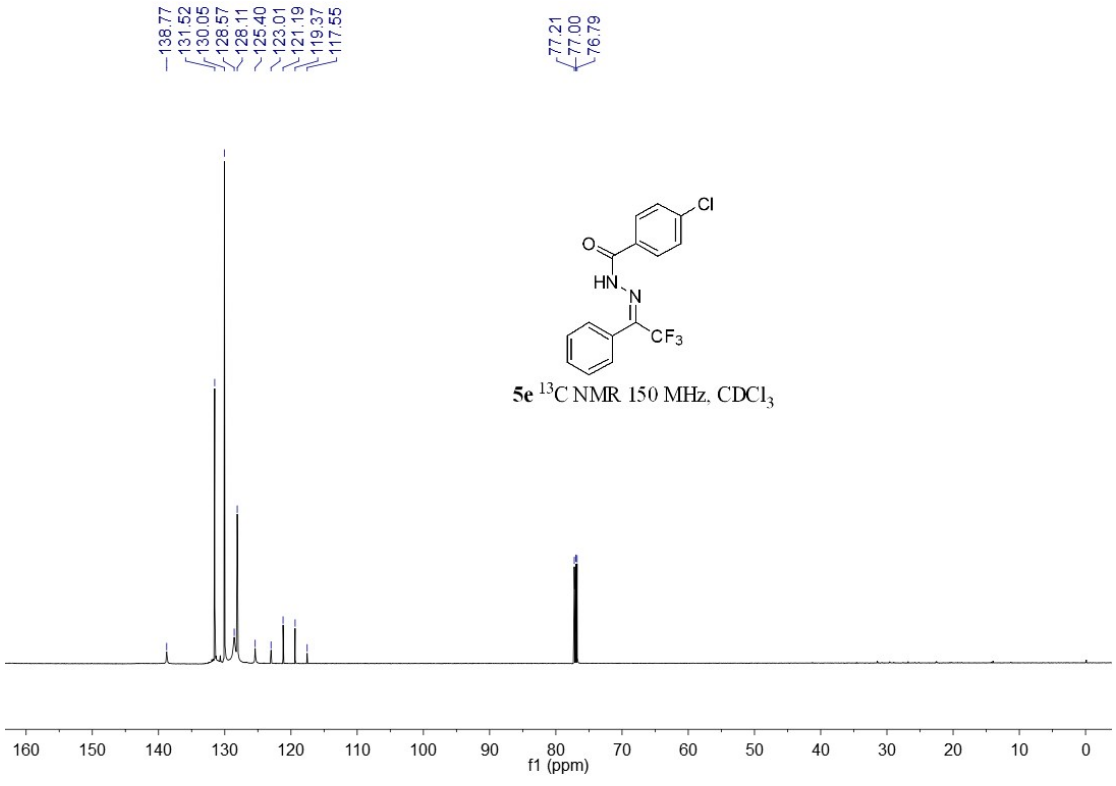


5d ^{19}F NMR 376 MHz, $CDCl_3$

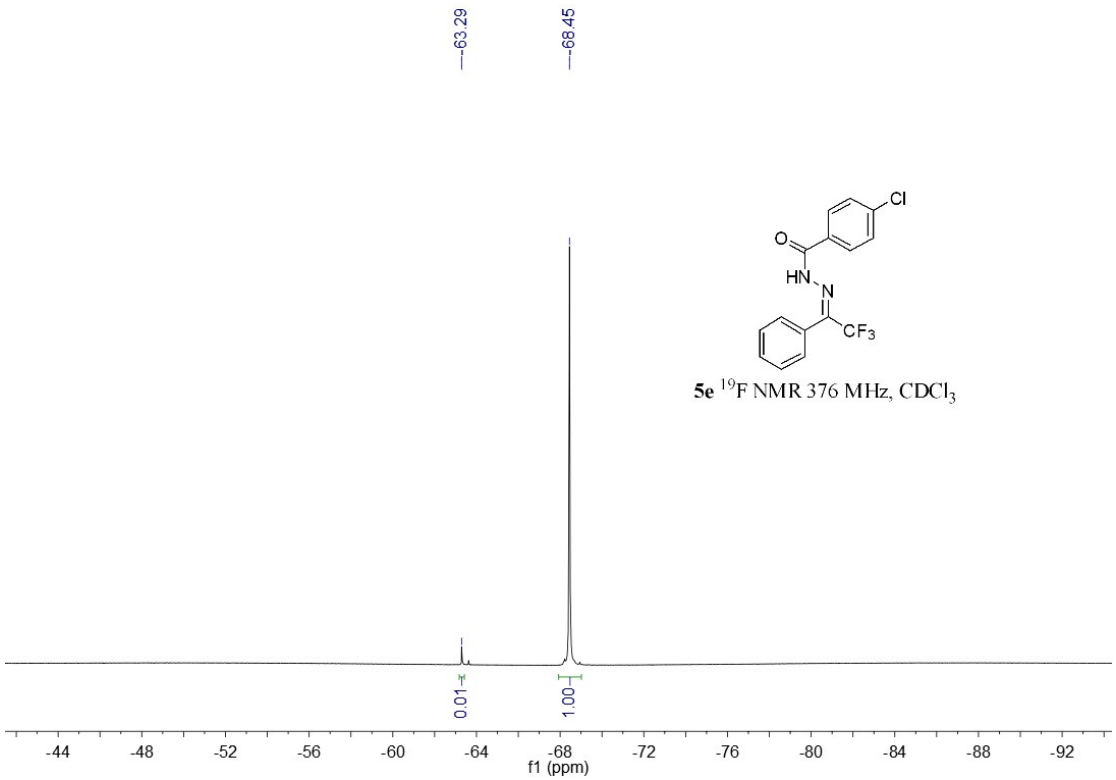
NMR copies of major product of compound **5e**:



5e ^{1}H NMR 600 MHz, $CDCl_3$

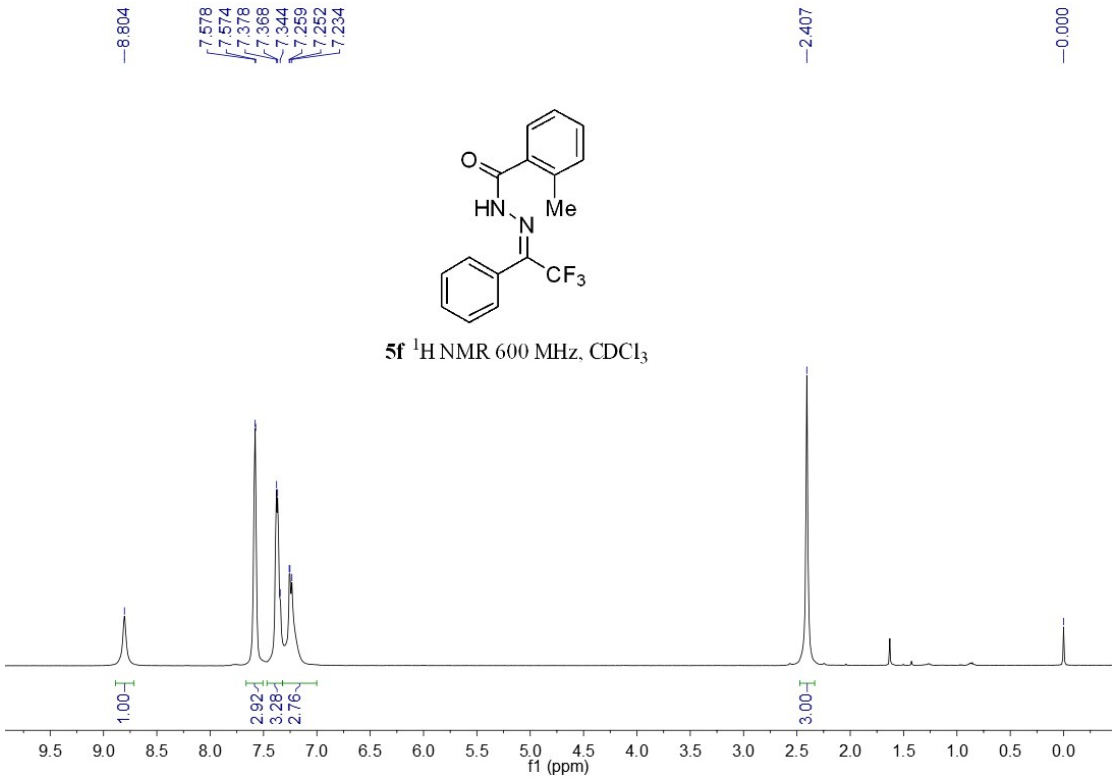


5e ^{13}C NMR 150 MHz, $CDCl_3$

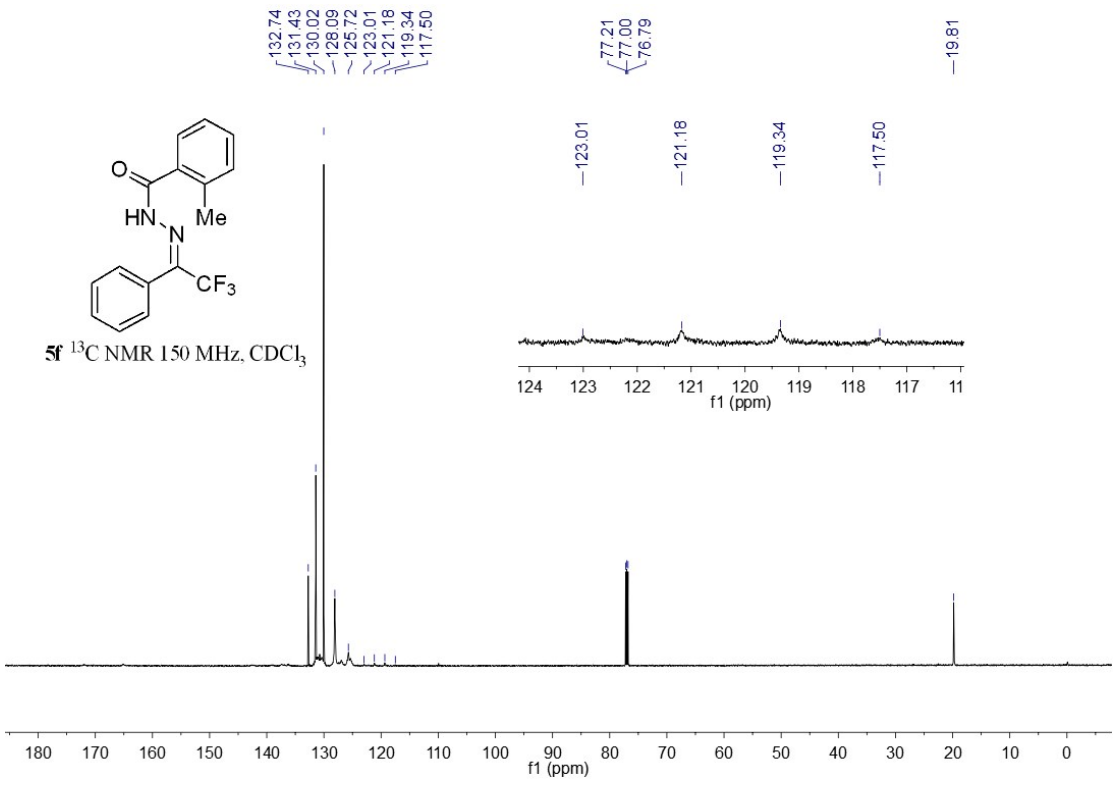


5e ^{19}F NMR 376 MHz, $CDCl_3$

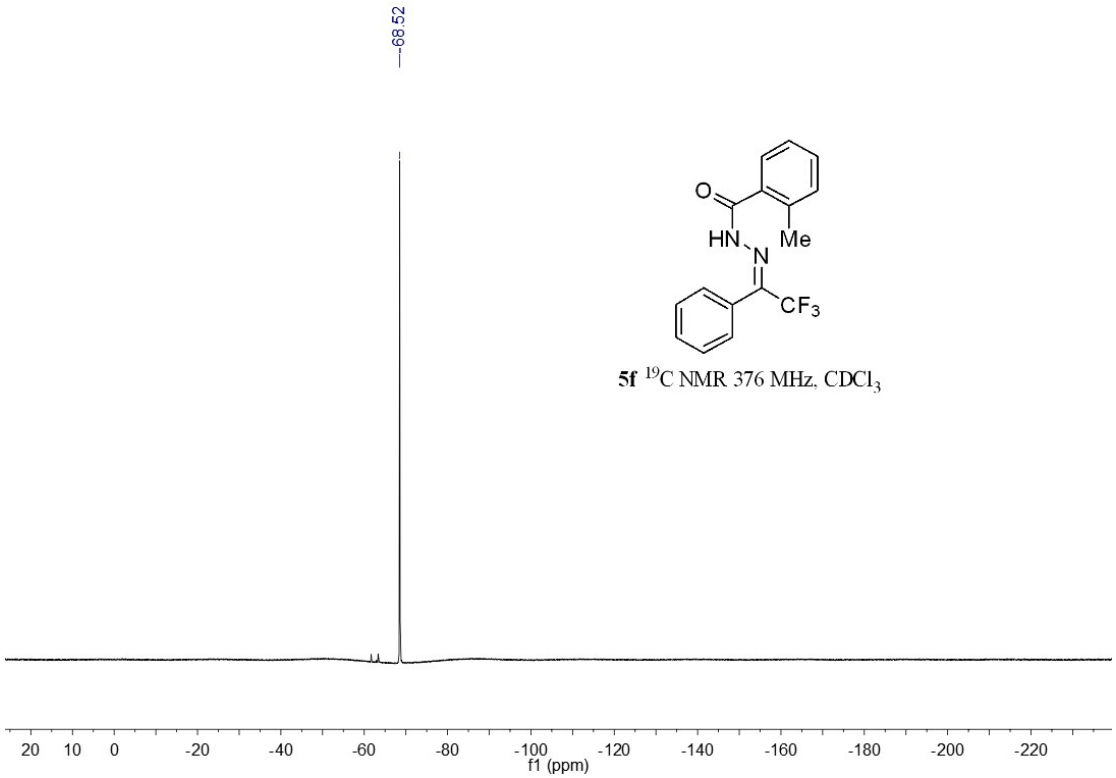
NMR copies of major product of compound **5f**:



5f ^{1}H NMR 600 MHz, $CDCl_3$

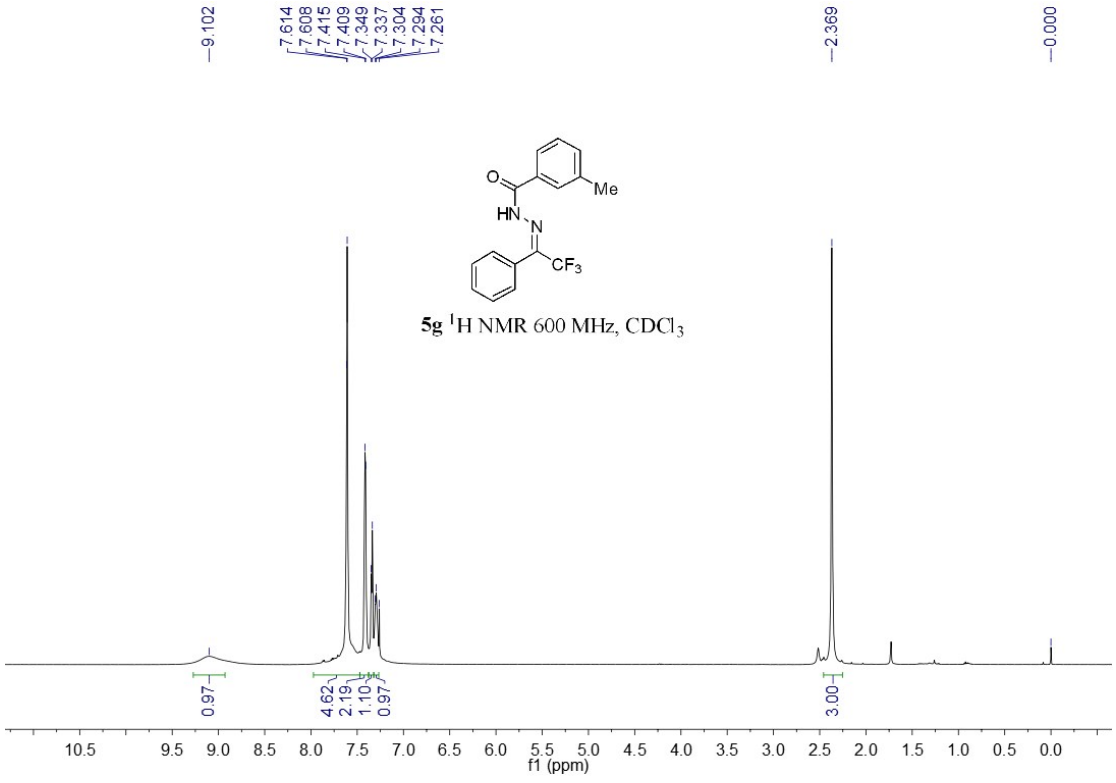


5f ^{13}C NMR 150 MHz, $CDCl_3$

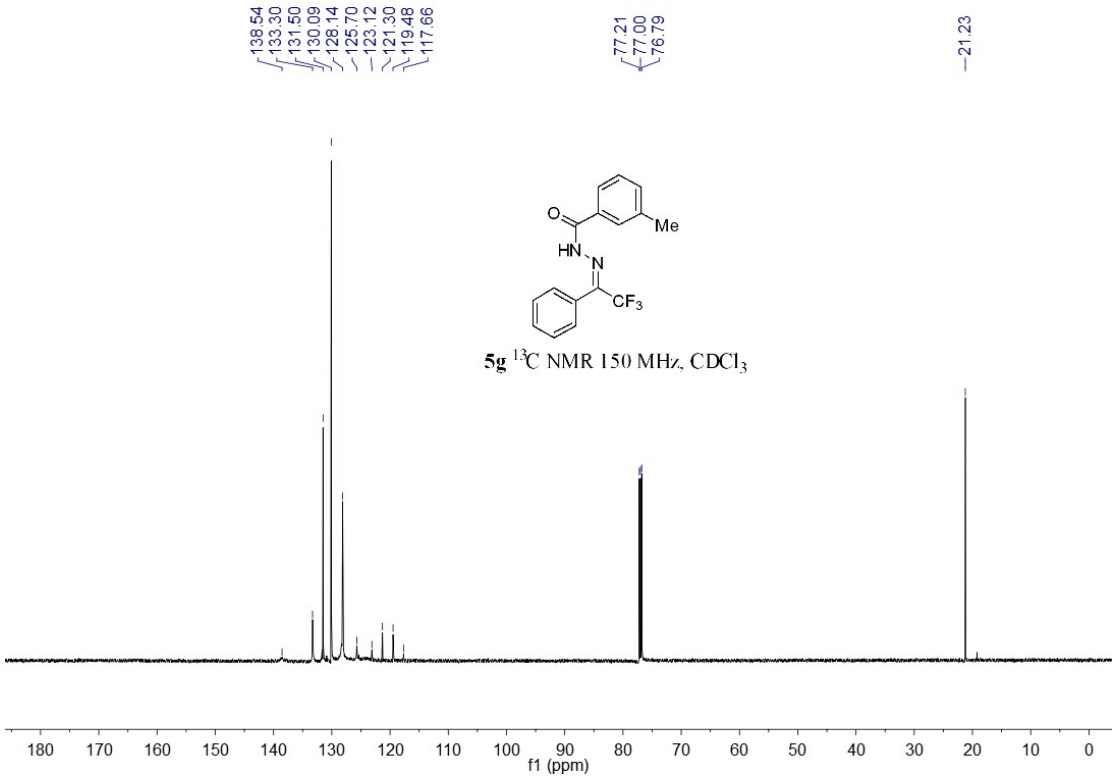


5f ^{19}C NMR 376 MHz, $CDCl_3$

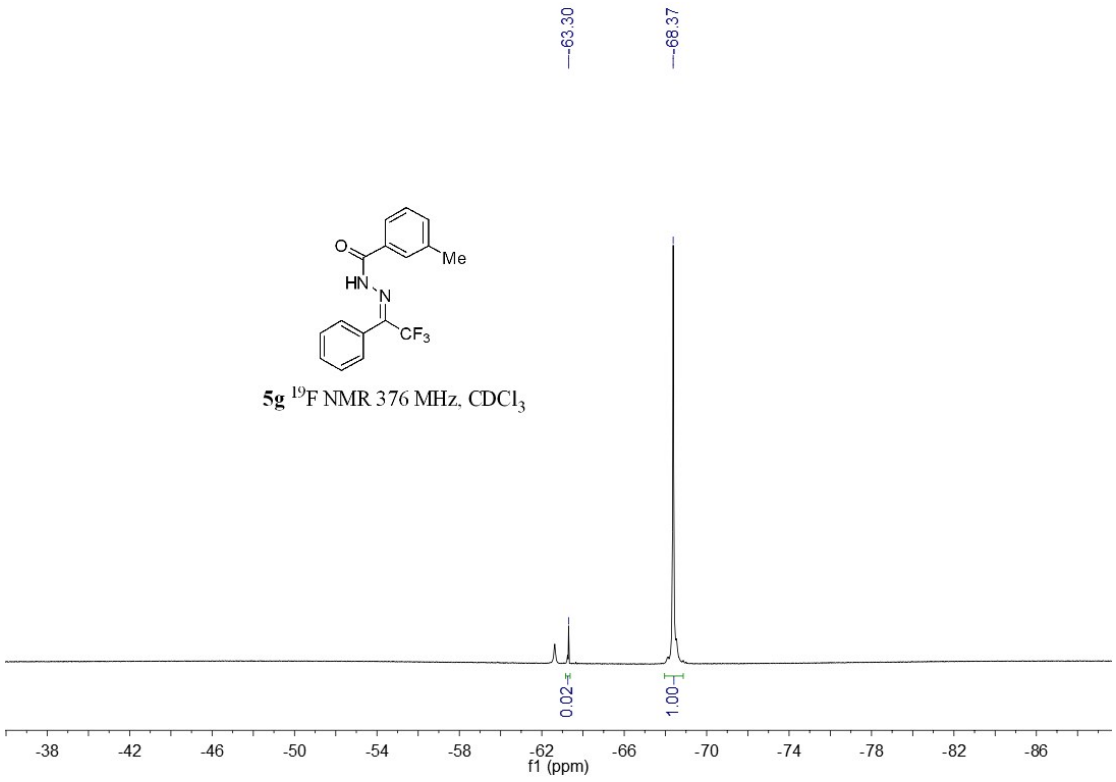
NMR copies of major product of compound **5g**:



5g ^{1}H NMR 600 MHz, $CDCl_3$

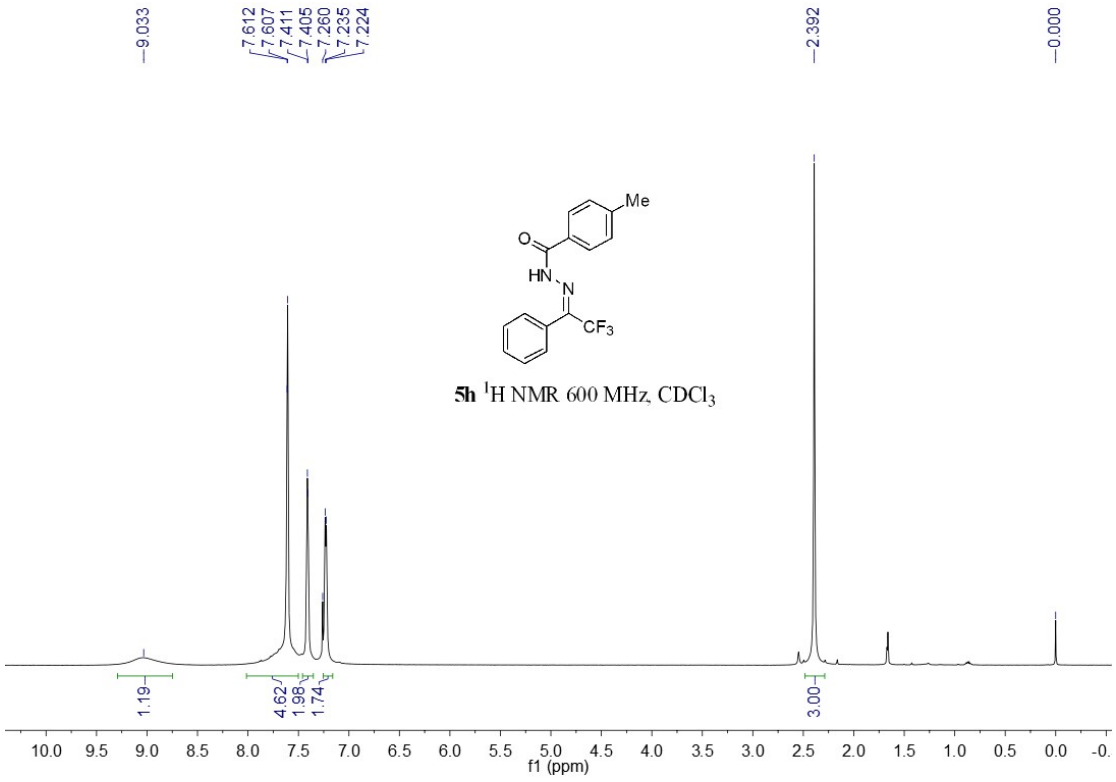


5g ^{13}C NMR 150 MHz, $CDCl_3$

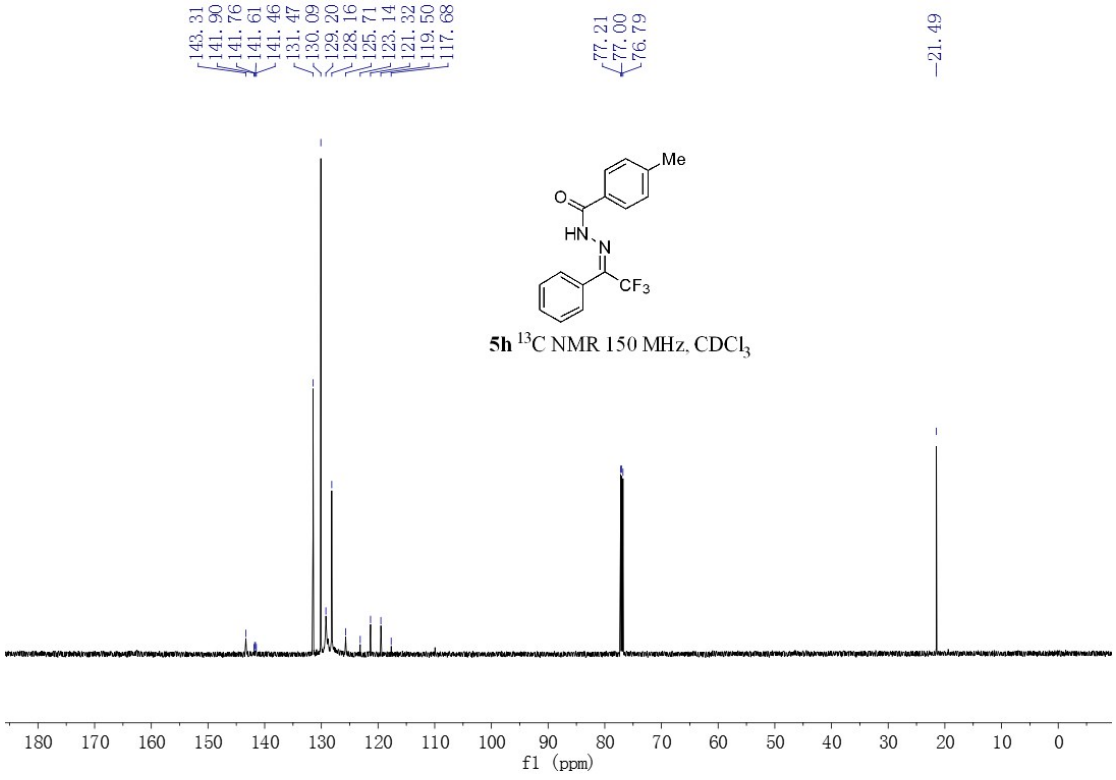


5g ^{19}F NMR 376 MHz, $CDCl_3$

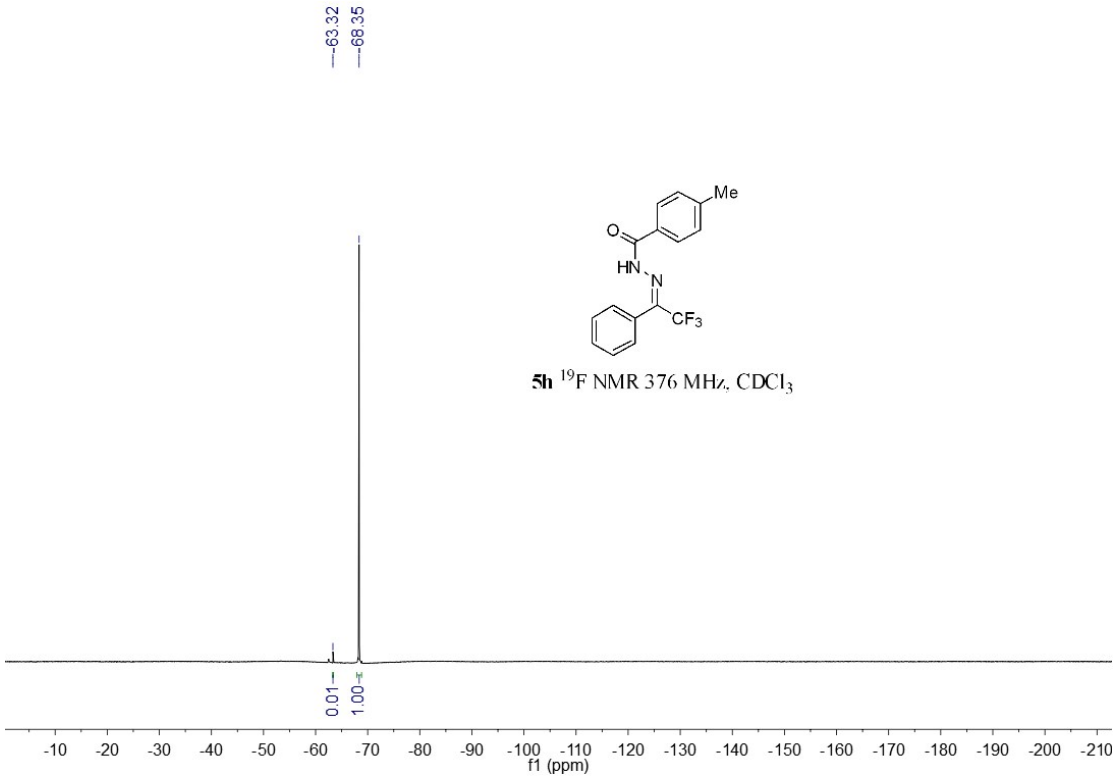
NMR copies of major product of compound **5h**:



5h ^{1}H NMR 600 MHz, $CDCl_3$

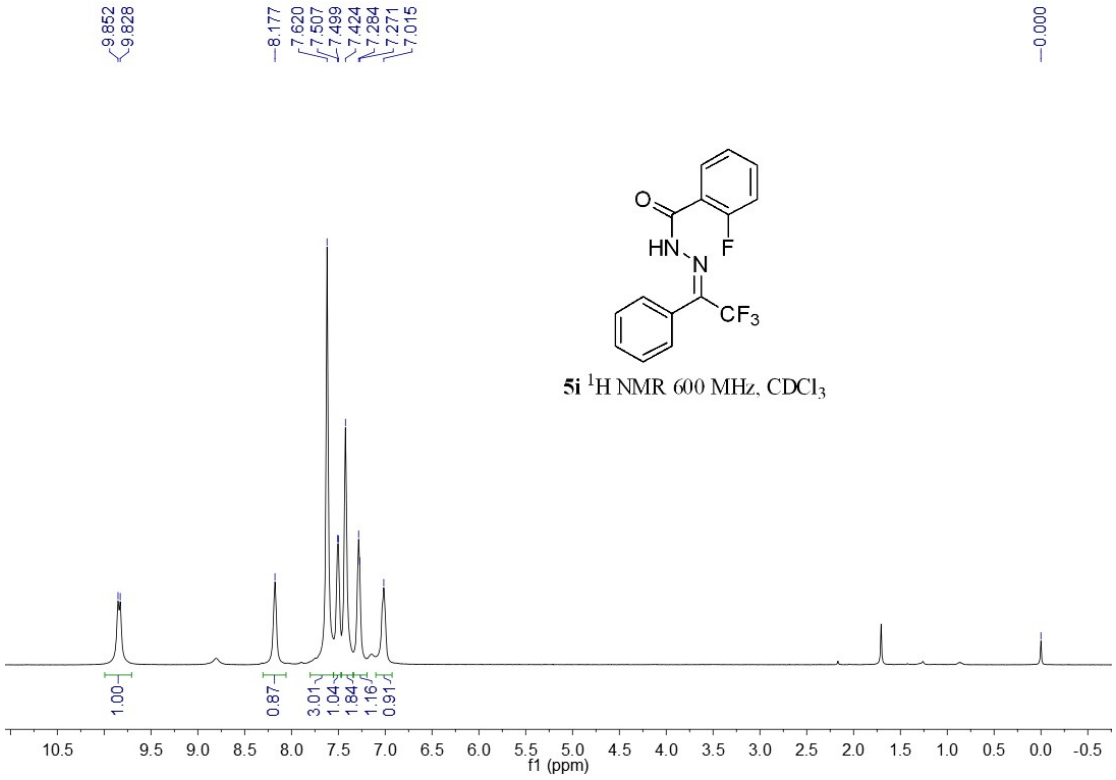


5h ^{13}C NMR 150 MHz, $CDCl_3$

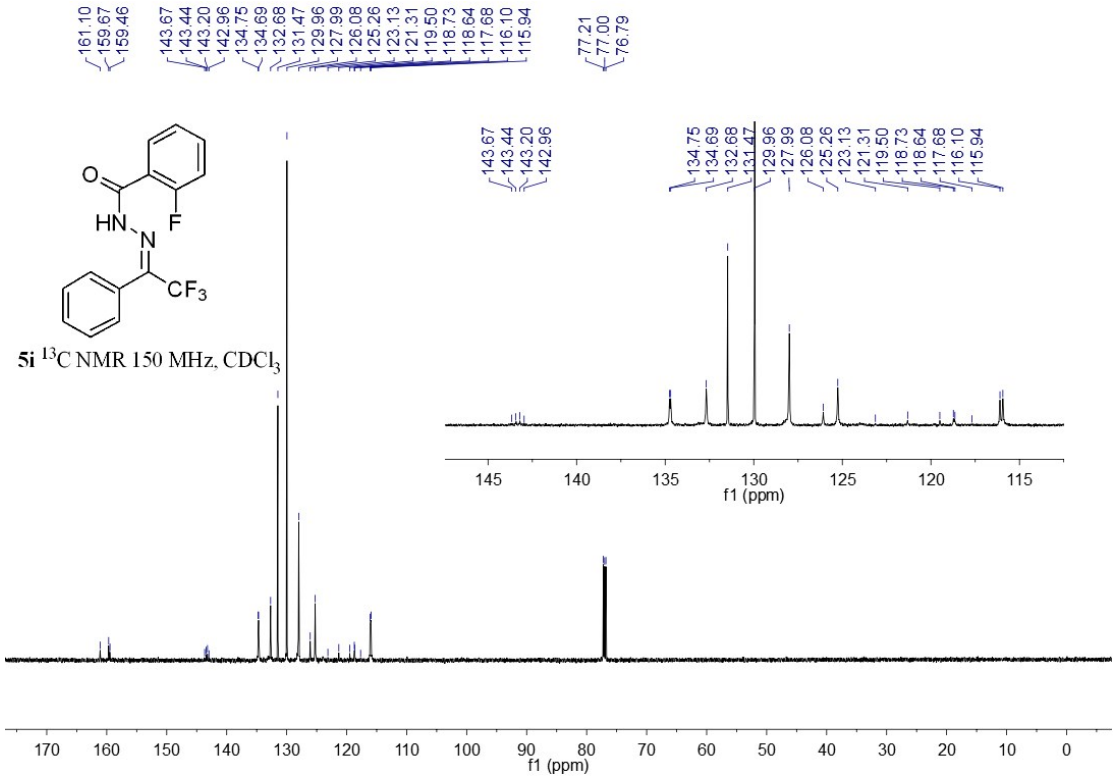


5h ^{19}F NMR 376 MHz, $CDCl_3$

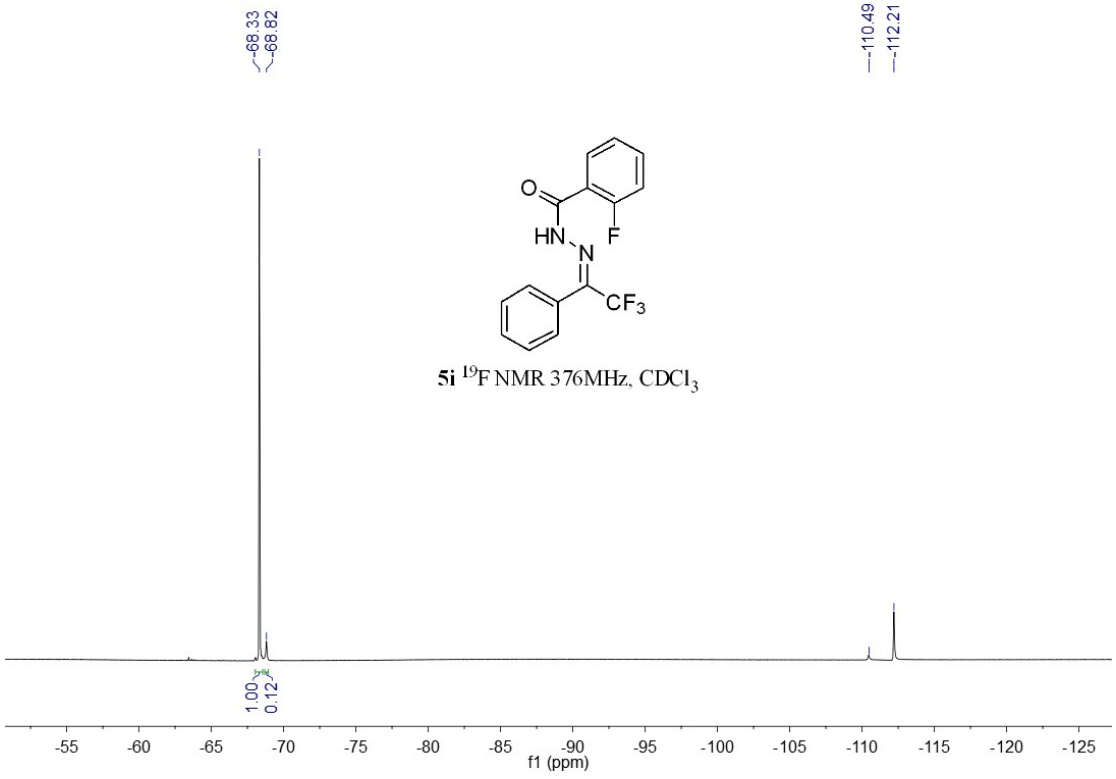
NMR copies of major product of compound **5i**:



5i ^{1}H NMR 600 MHz, $CDCl_3$

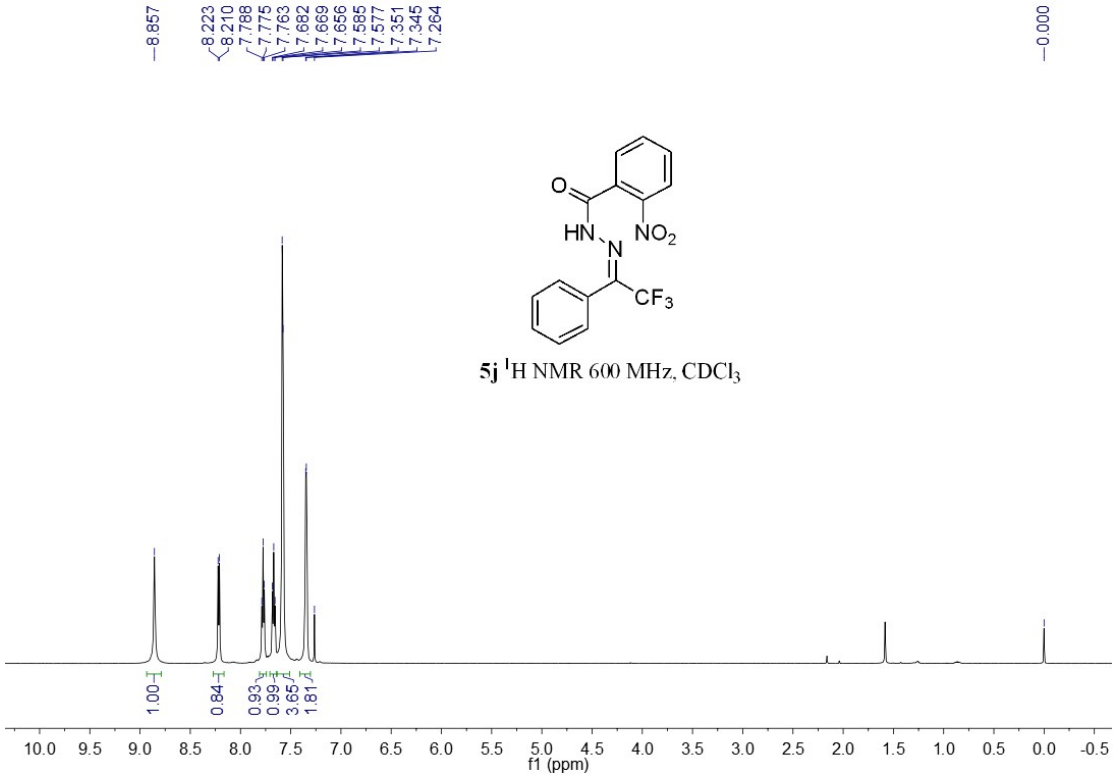


5i ^{13}C NMR 150 MHz, $CDCl_3$

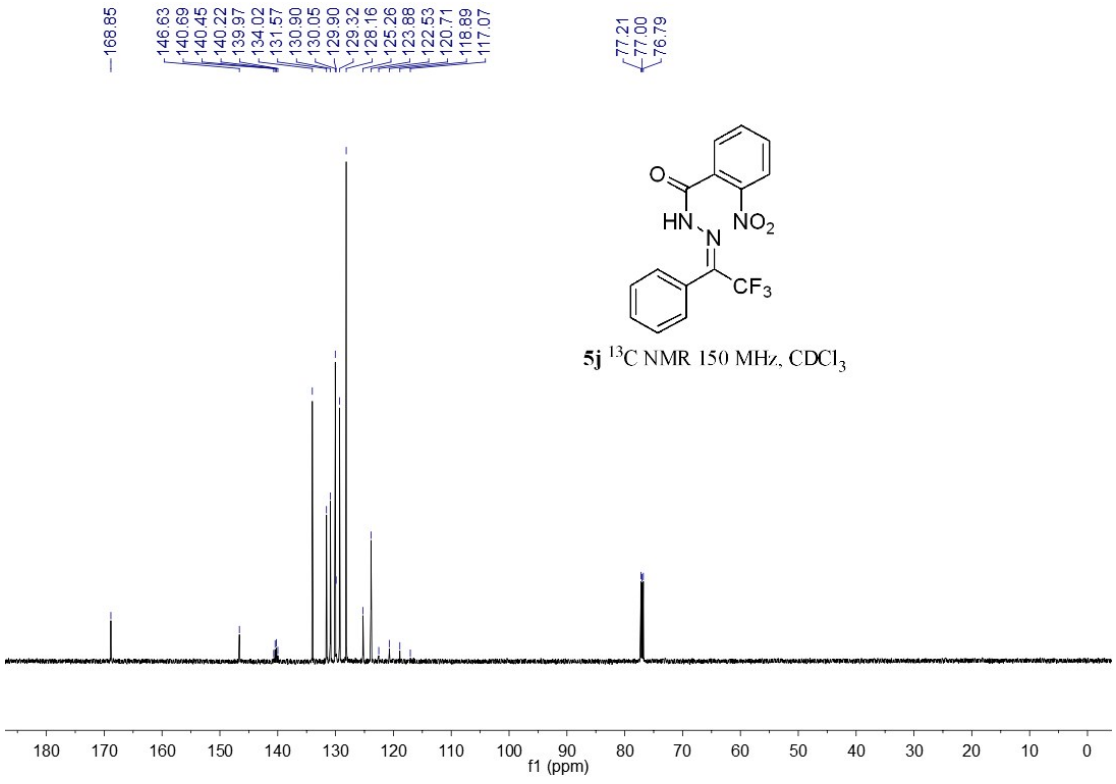


5i ^{19}F NMR 376MHz, $CDCl_3$

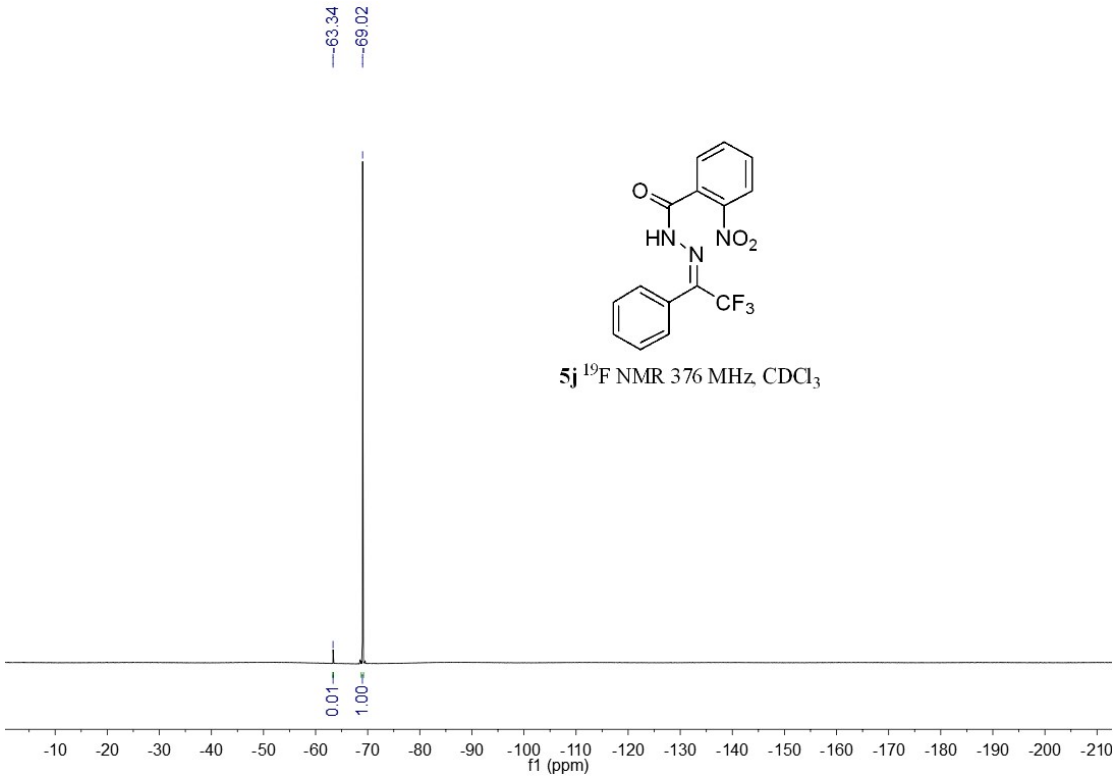
NMR copies of major product of compound **5j**:



5j ^{1}H NMR 600 MHz, $CDCl_3$

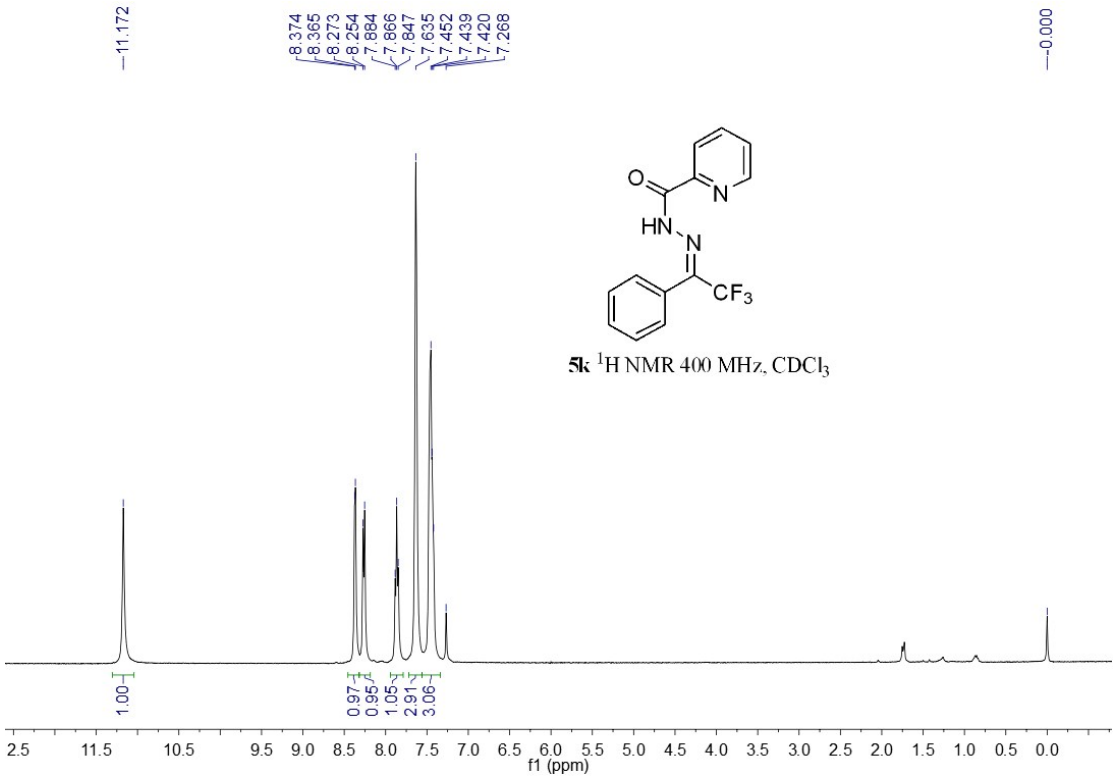


5j ^{13}C NMR 150 MHz, $CDCl_3$

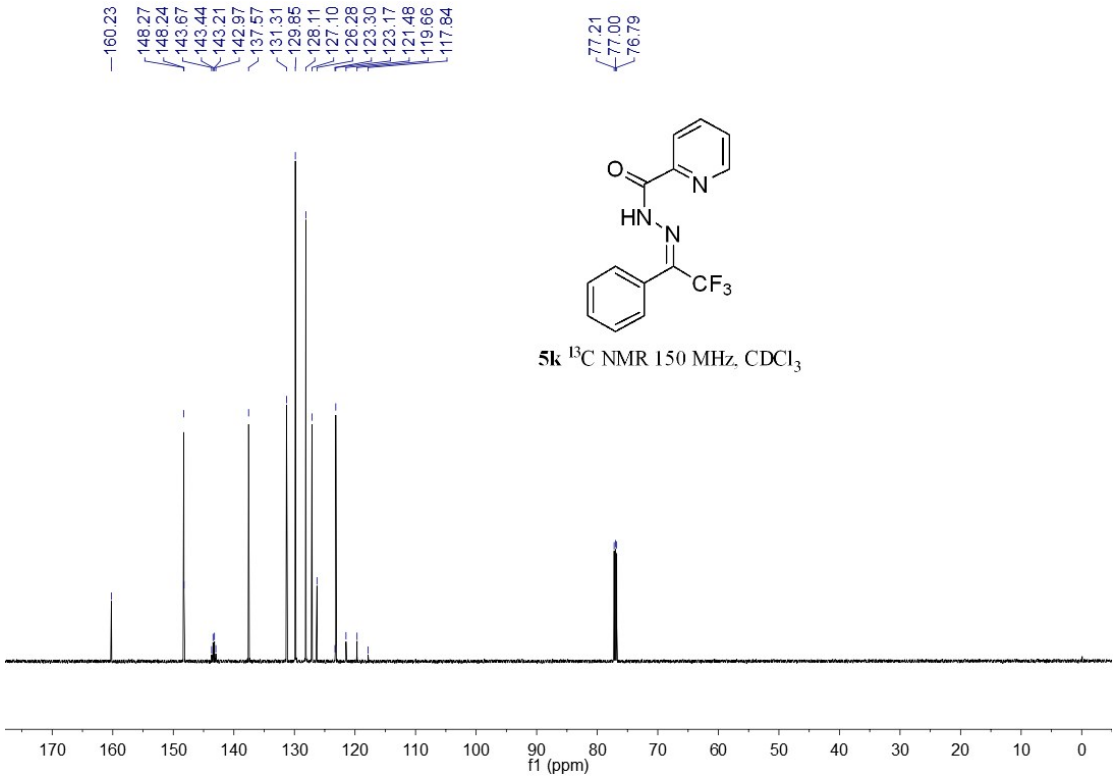


5j ^{19}F NMR 376 MHz, $CDCl_3$

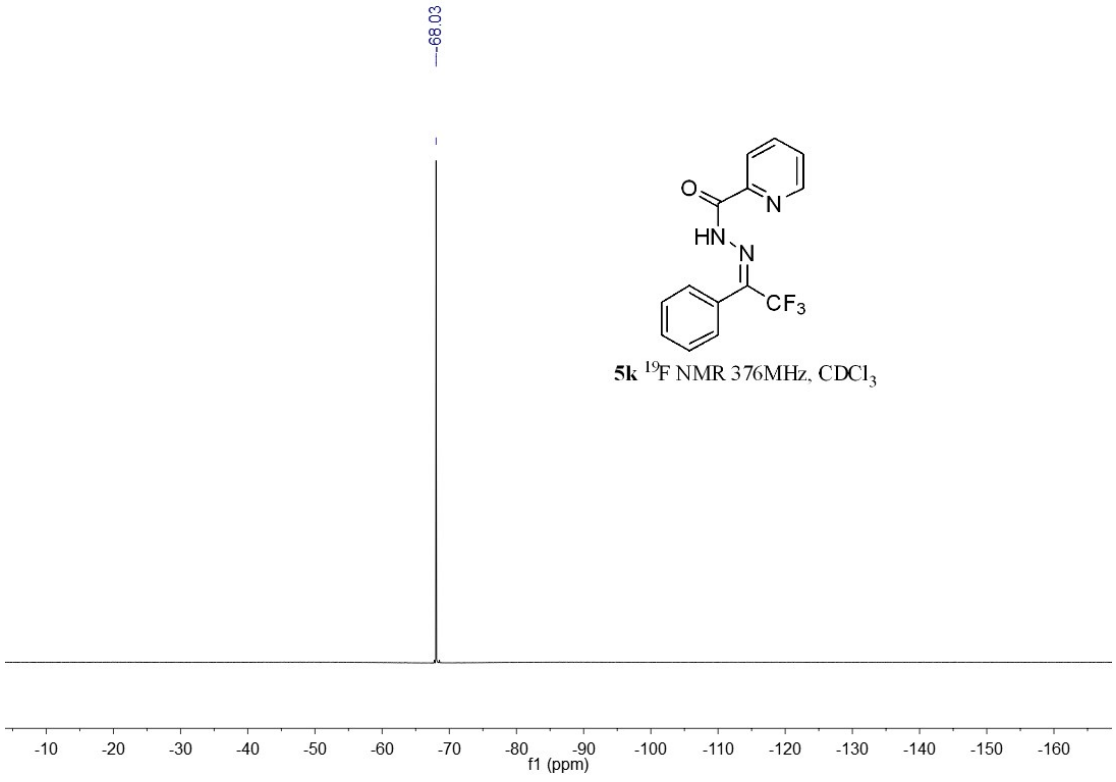
NMR copies of major product of compound **5k**:



5k ^{1}H NMR 400 MHz, $CDCl_3$

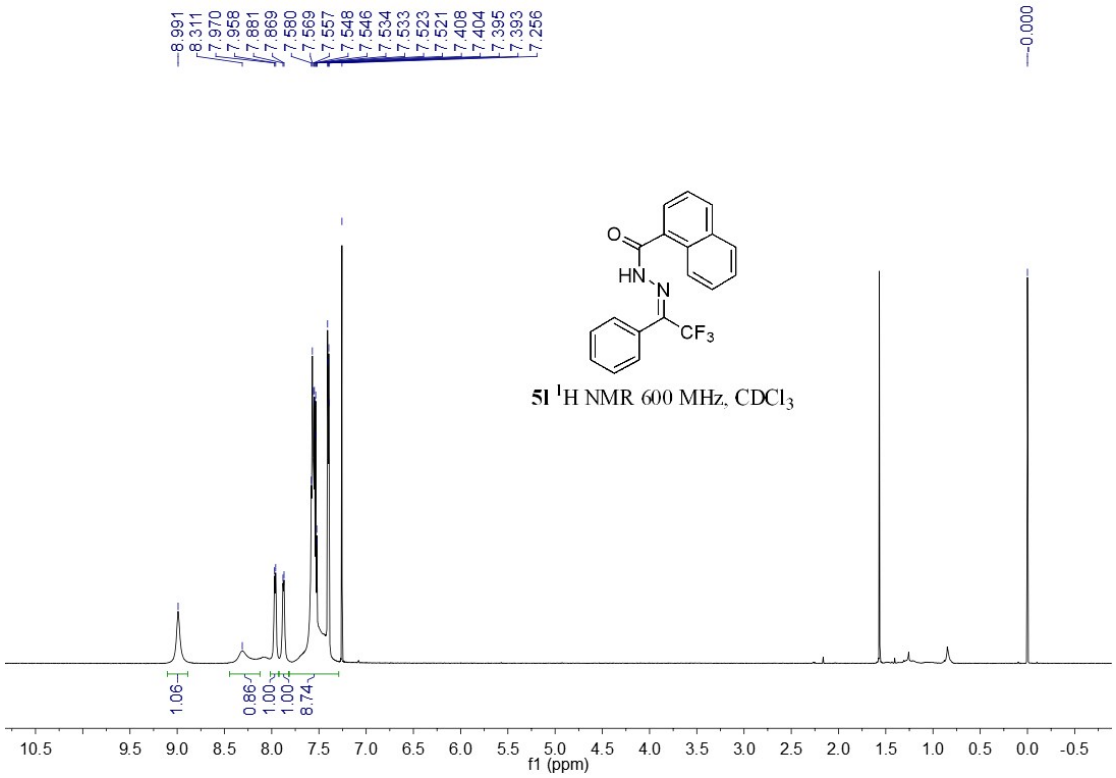


5k ^{13}C NMR 150 MHz, $CDCl_3$

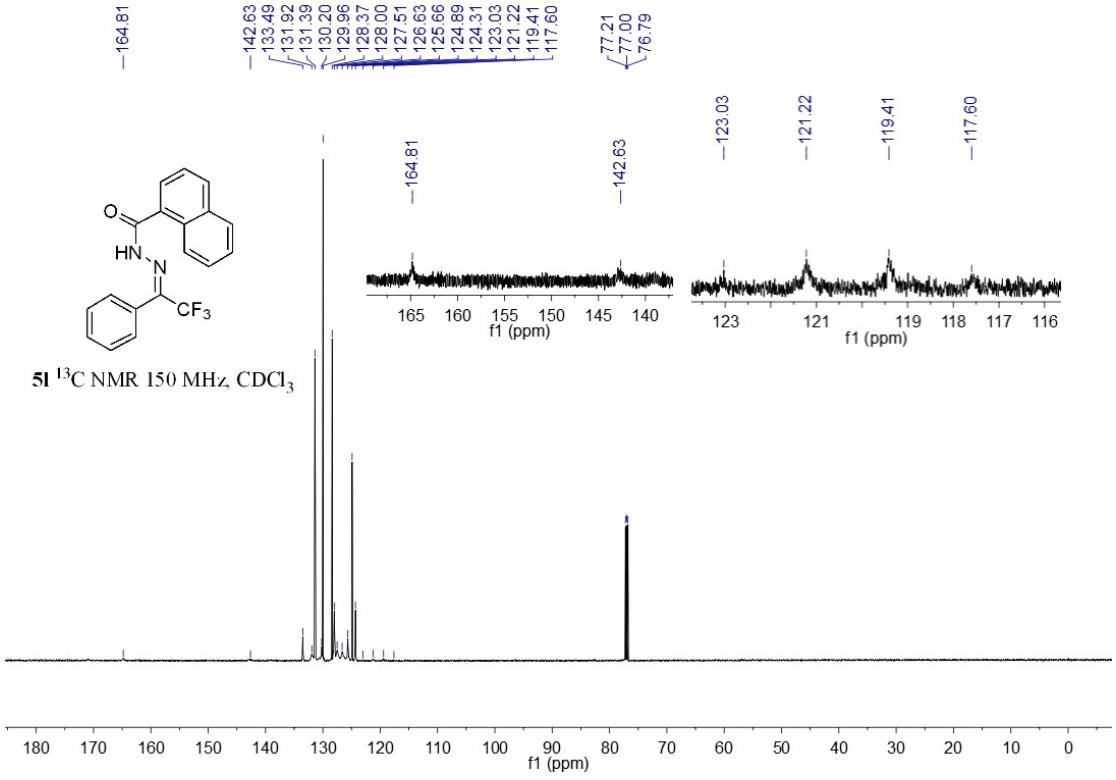


5k ^{19}F NMR 376MHz, $CDCl_3$

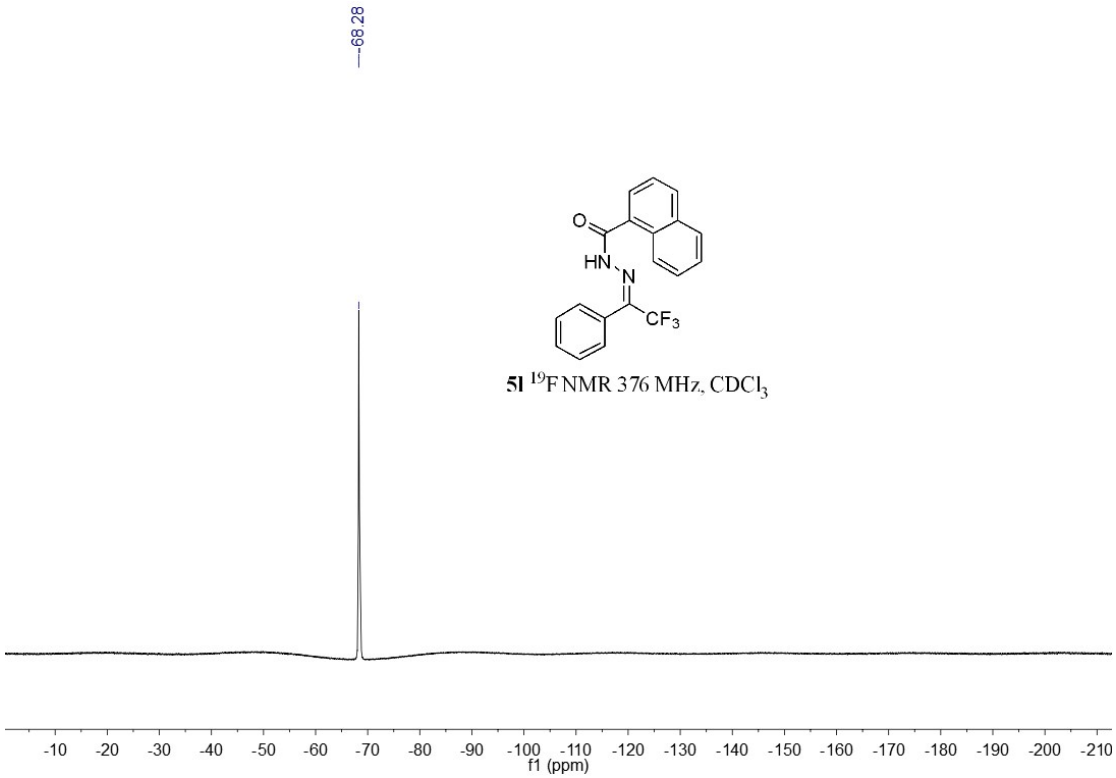
NMR copies of major product of compound **5l**:



5l ^{1}H NMR 600 MHz, $CDCl_3$

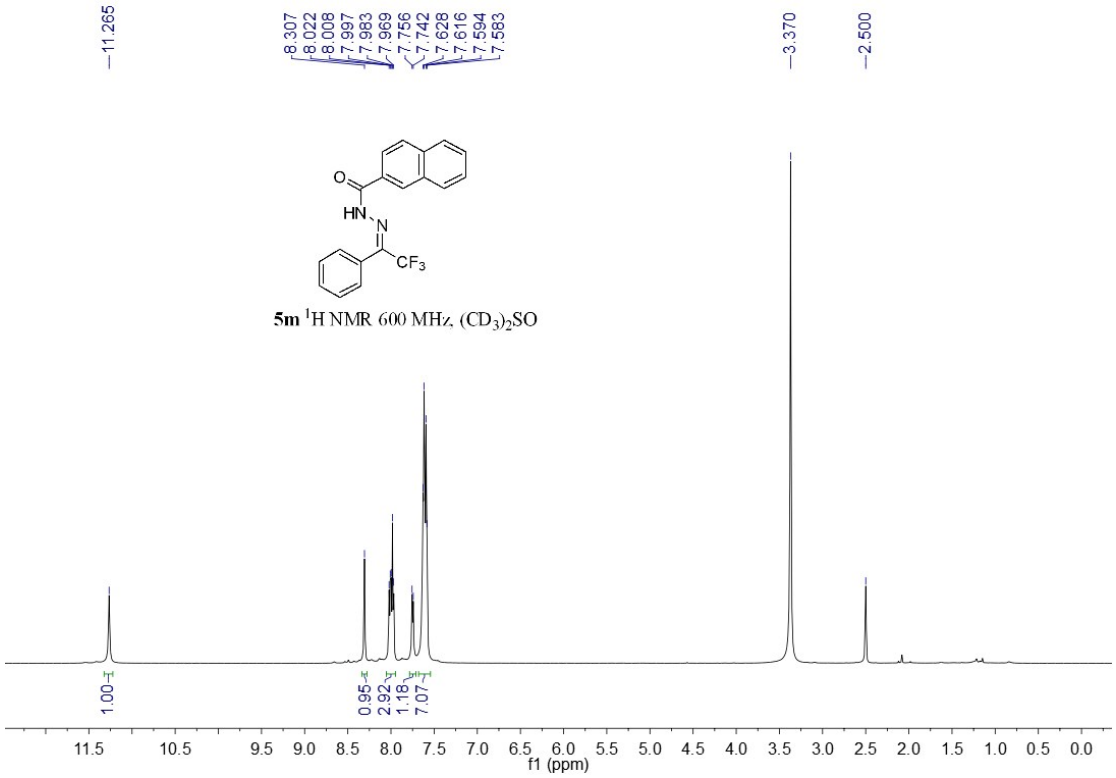


5l ^{13}C NMR 150 MHz, $CDCl_3$

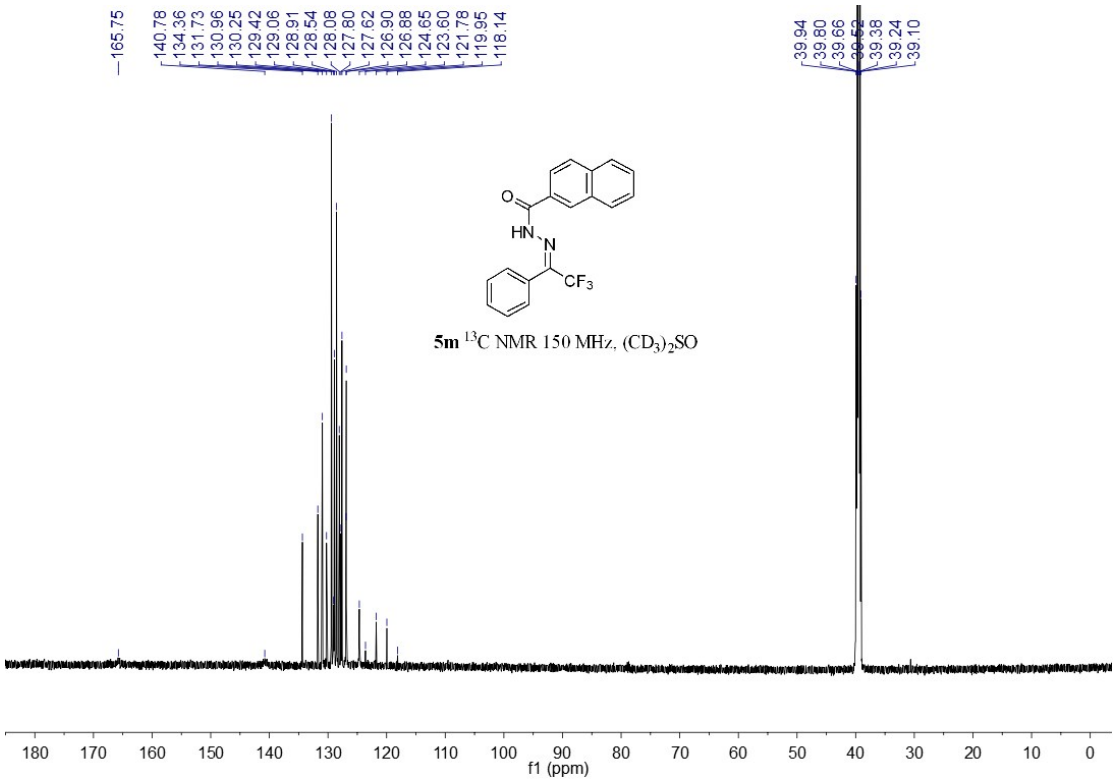


5l ^{19}F NMR 376 MHz, $CDCl_3$

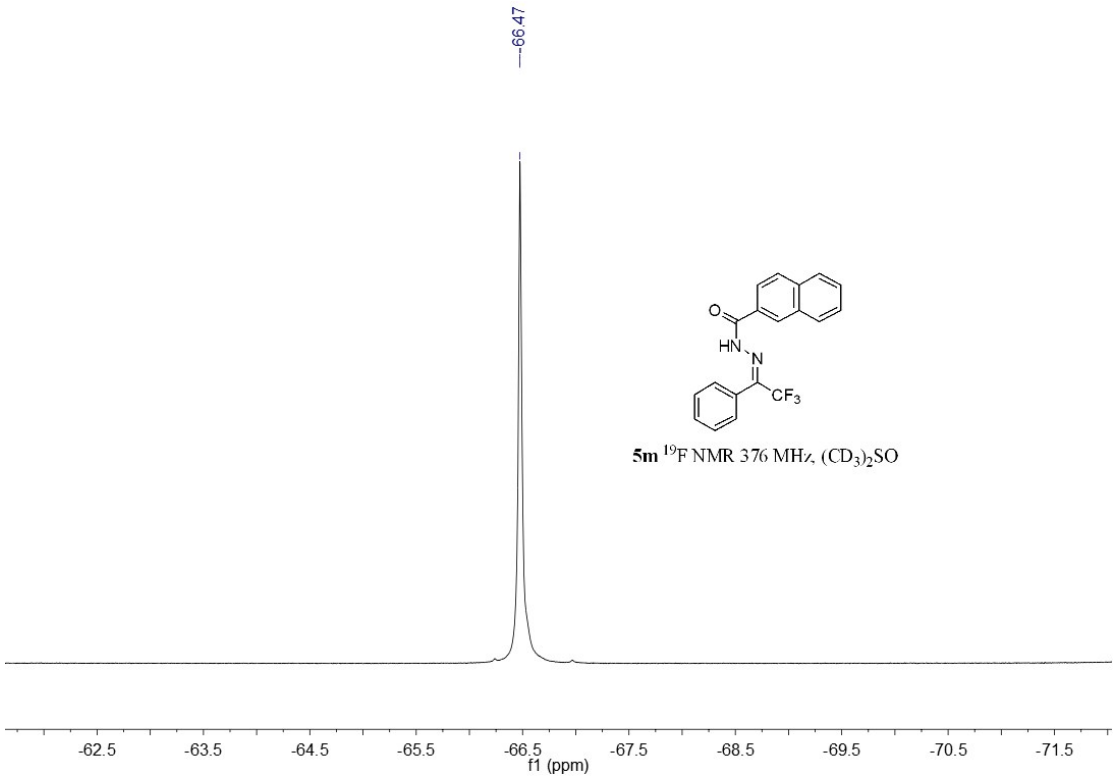
NMR copies of major product of compound **5m**:



5m ^{1}H NMR 600 MHz, $(CD_3)_2SO$

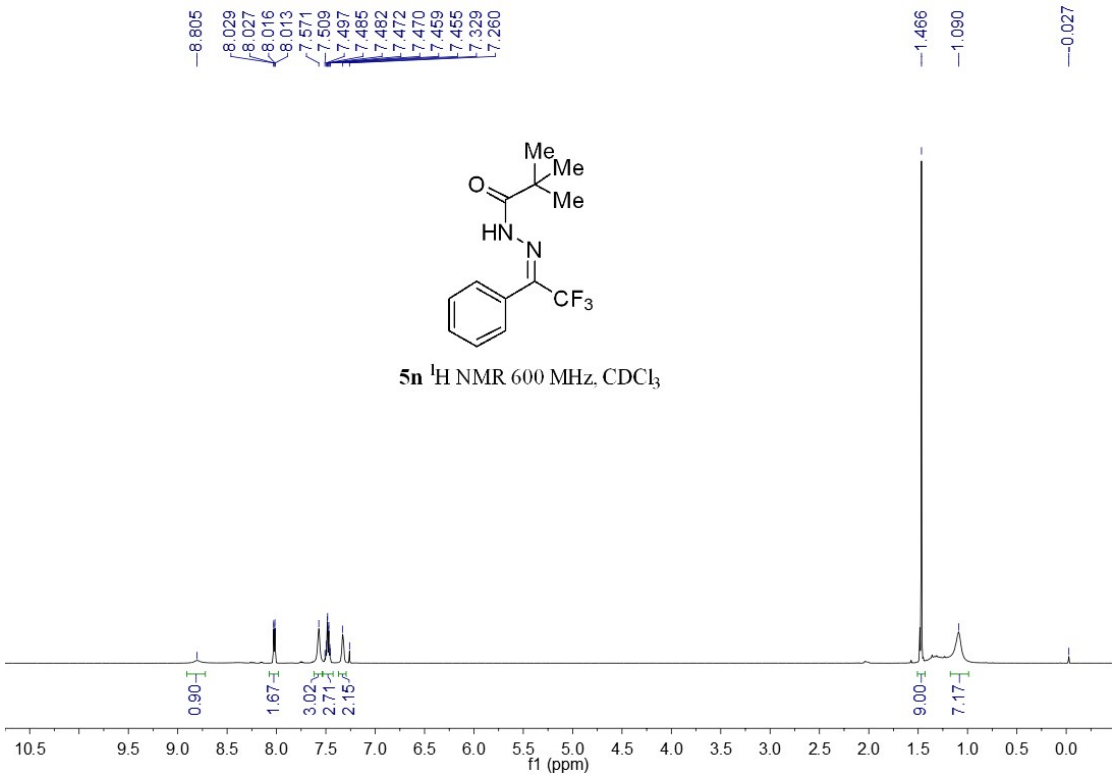


5m ^{13}C NMR 150 MHz, $(CD_3)_2SO$

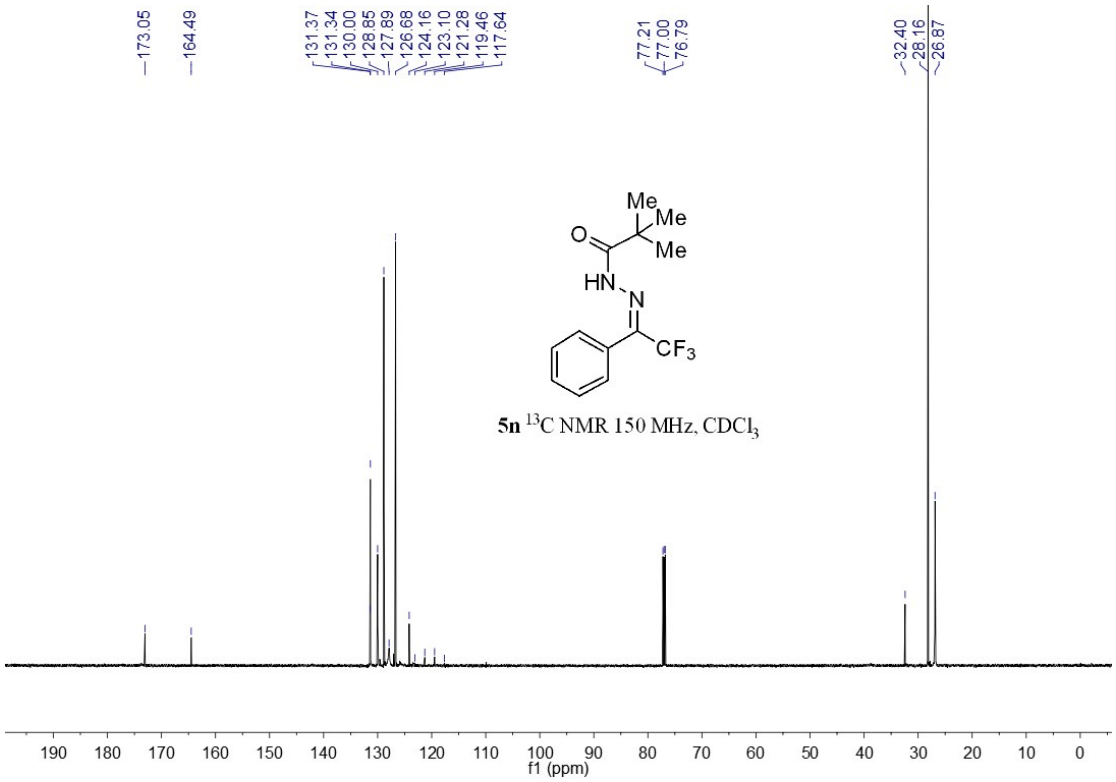


5m ^{19}F NMR 376 MHz, $(CD_3)_2SO$

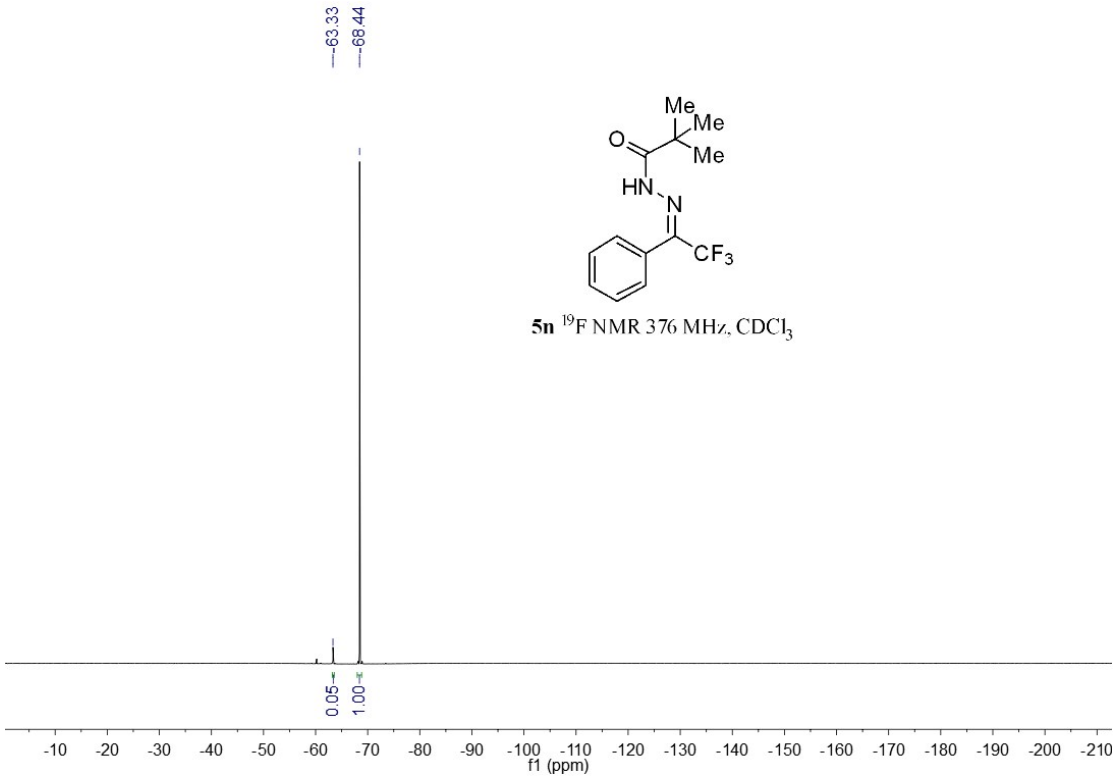
NMR copies of major product of compound **5n**:



5n ^{1}H NMR 600 MHz, $CDCl_3$

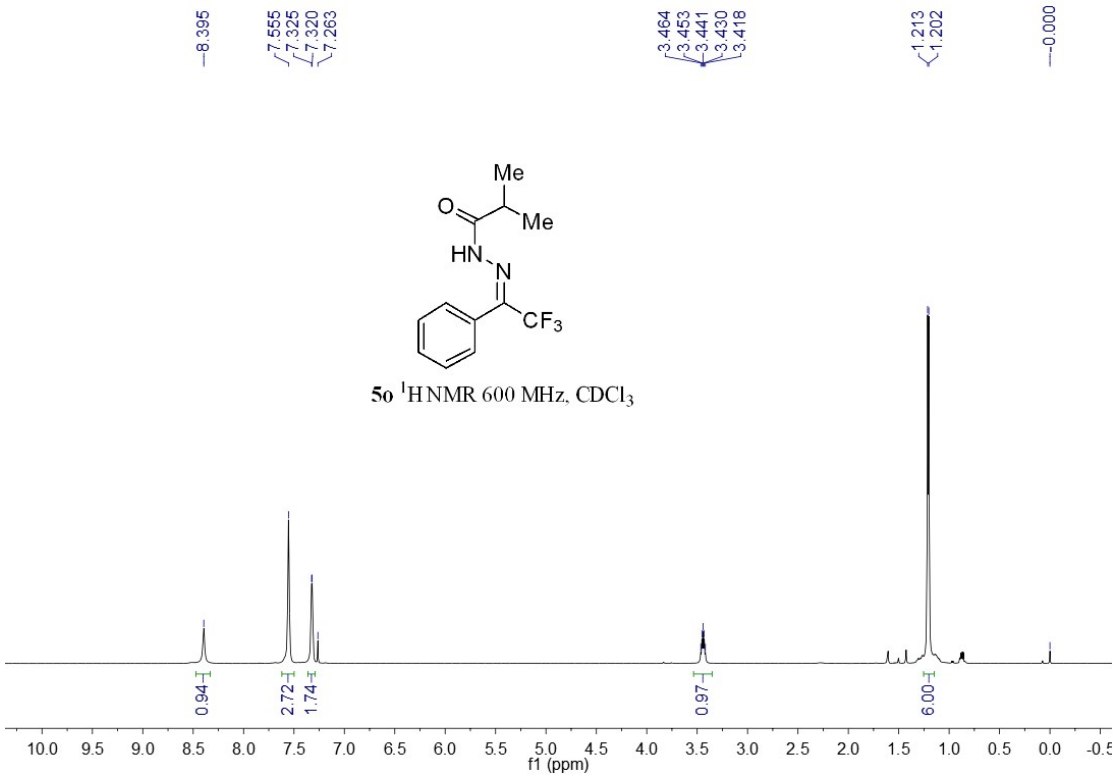


5n ^{13}C NMR 150 MHz, $CDCl_3$

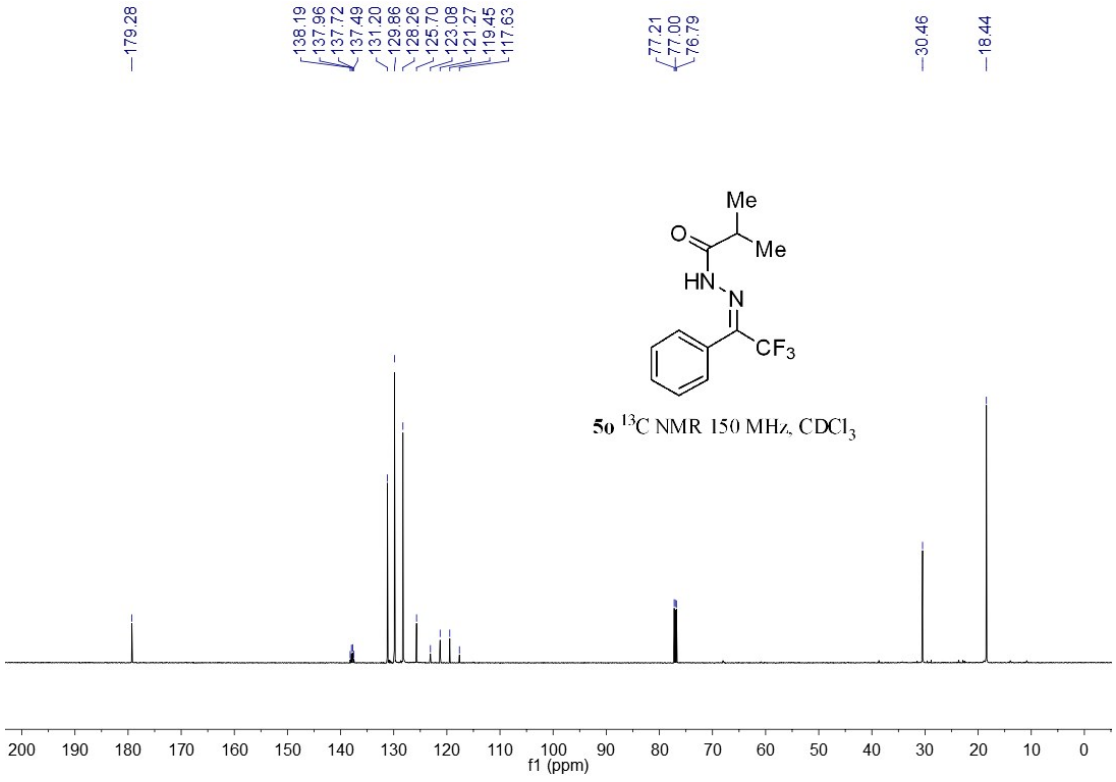


5n ^{19}F NMR 376 MHz, $CDCl_3$

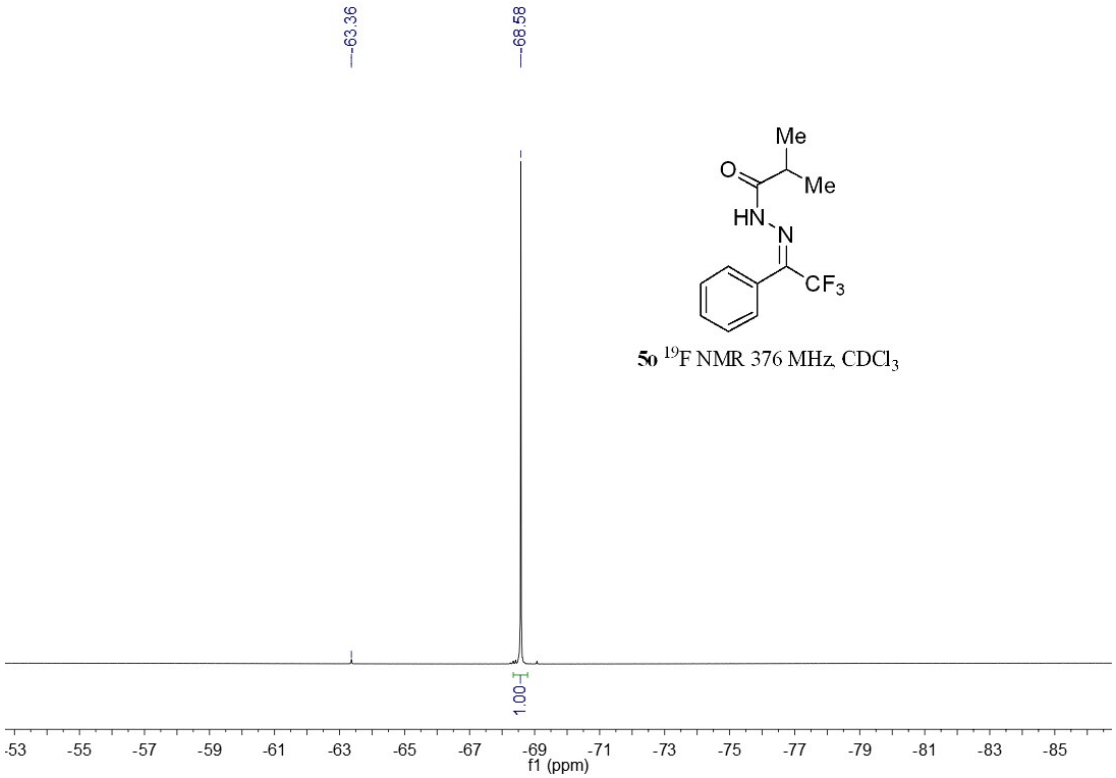
NMR copies of major product of compound **5o**:



5o ^{1}H NMR 600 MHz, $CDCl_3$

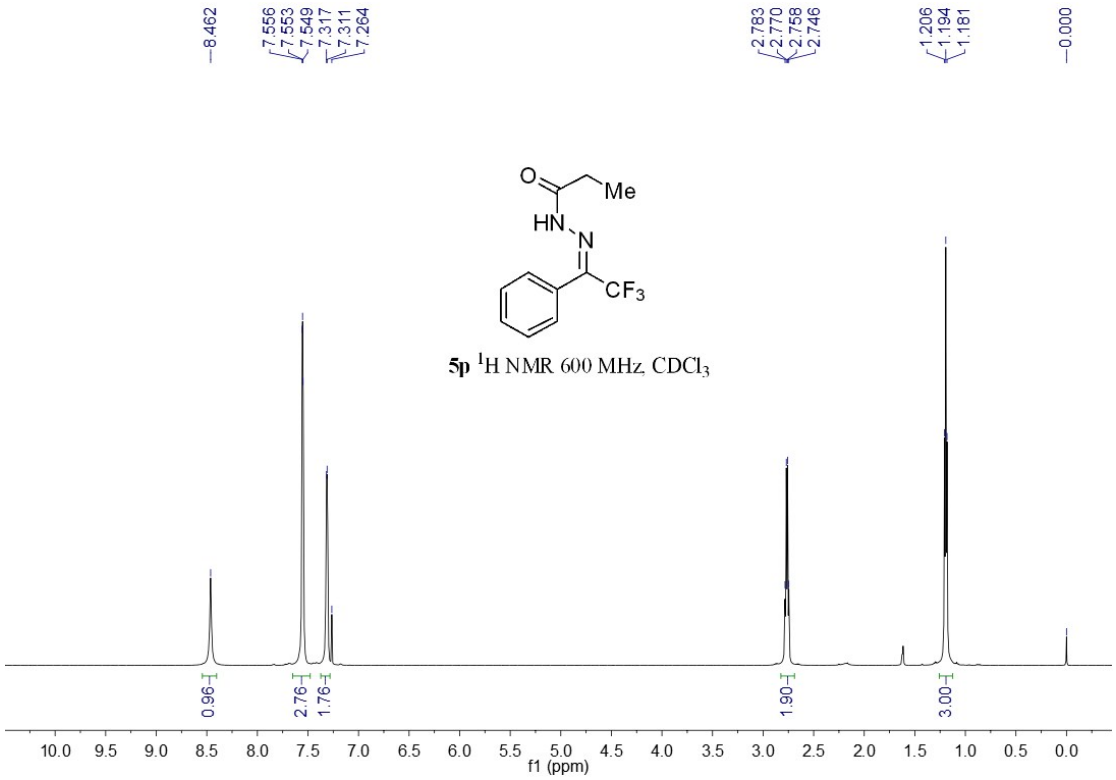


5o ^{13}C NMR 150 MHz, $CDCl_3$

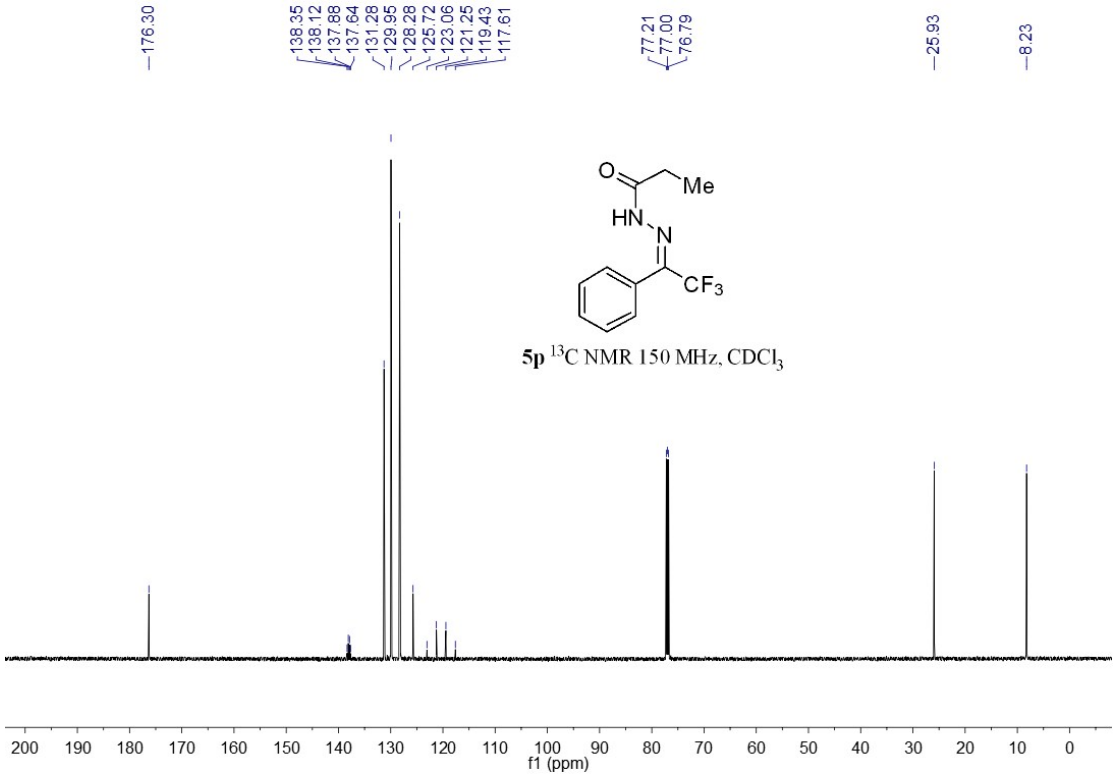


50 ^{19}F NMR 376 MHz, $CDCl_3$

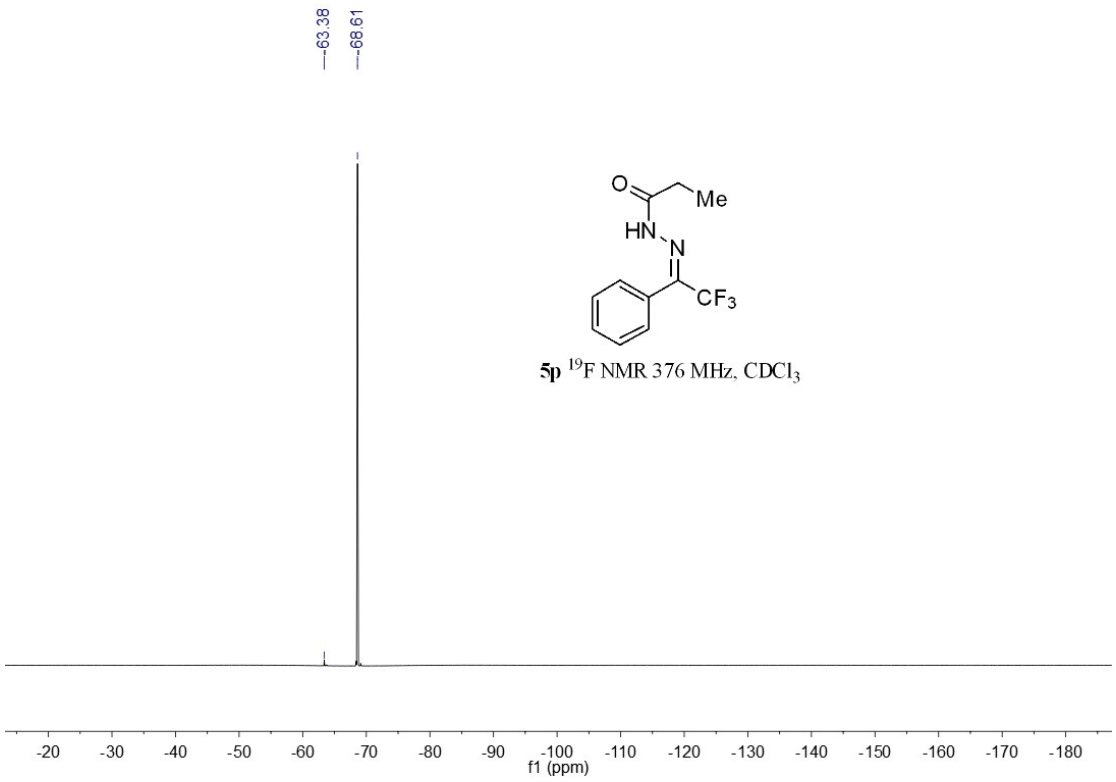
NMR copies of major product of compound **5p**:



5p ^{1}H NMR 600 MHz, $CDCl_3$

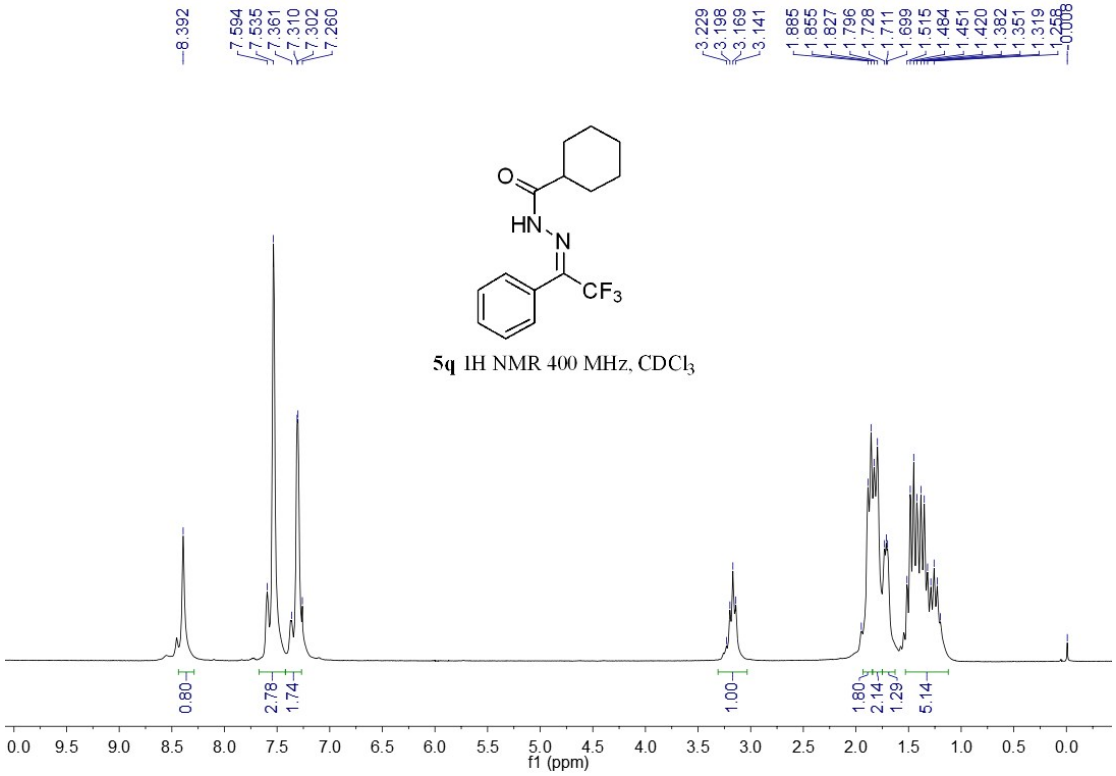


5p ^{13}C NMR 150 MHz, $CDCl_3$

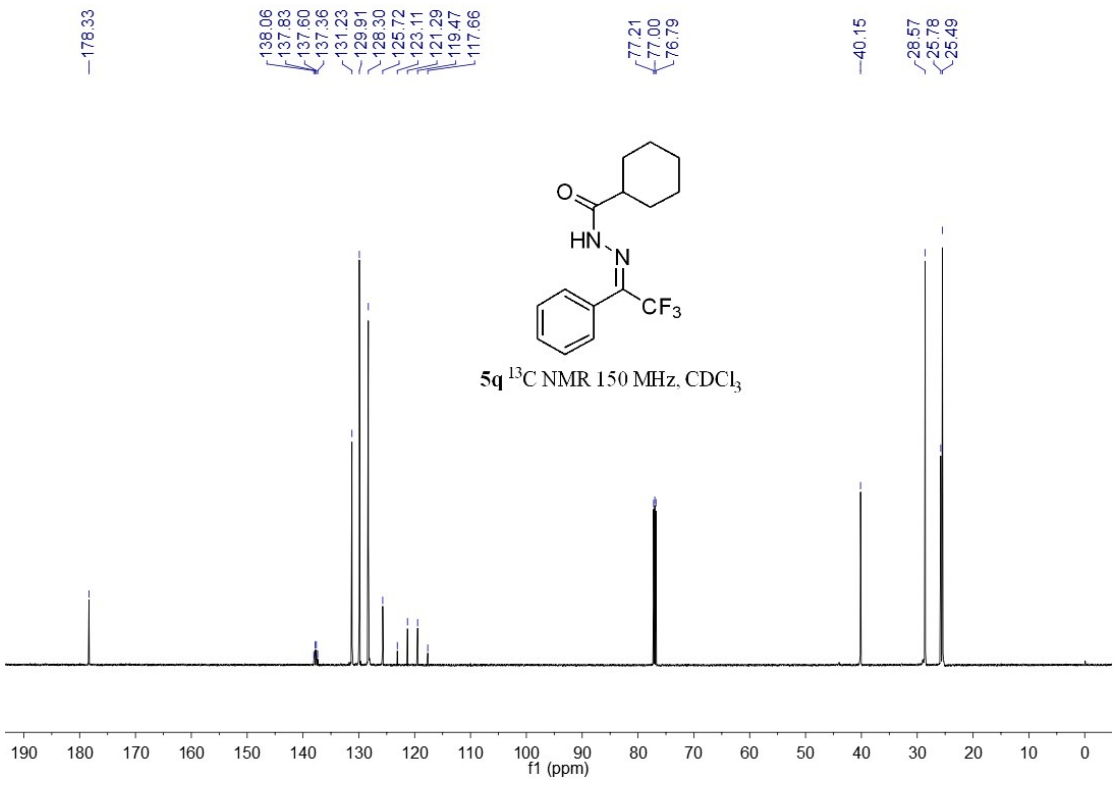


5p ^{19}F NMR 376 MHz, $CDCl_3$

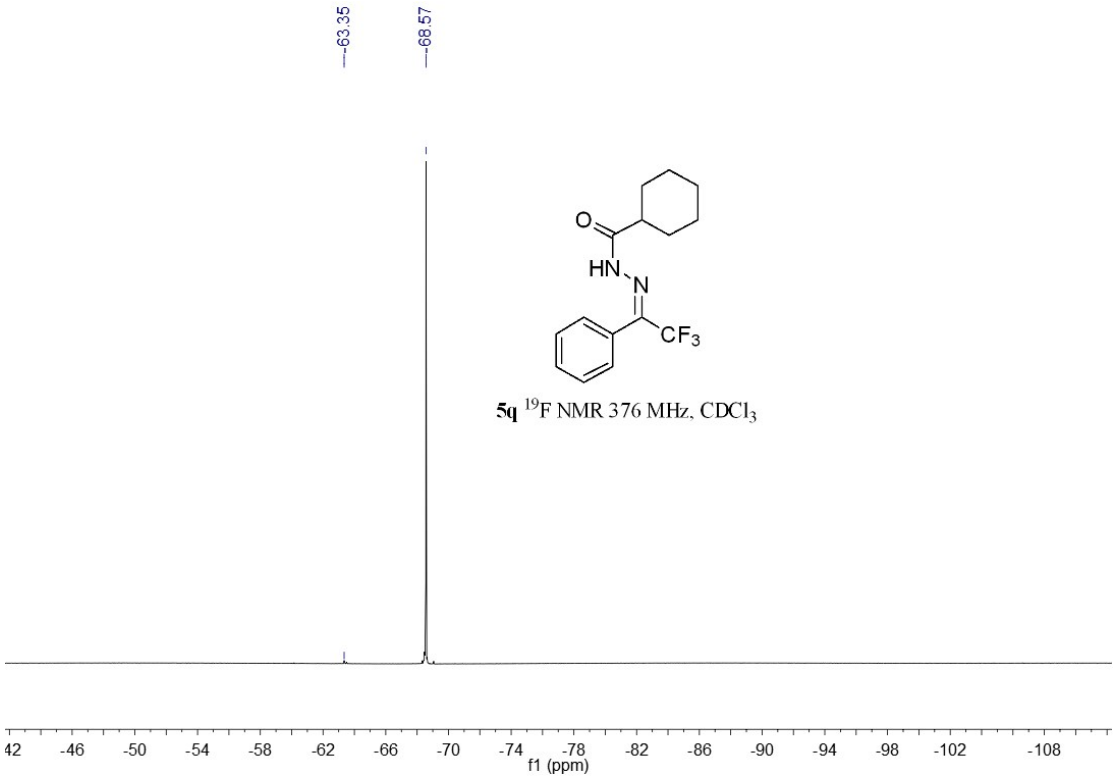
NMR copies of major product of compound **5q**:



5q 1H NMR 400 MHz, $CDCl_3$

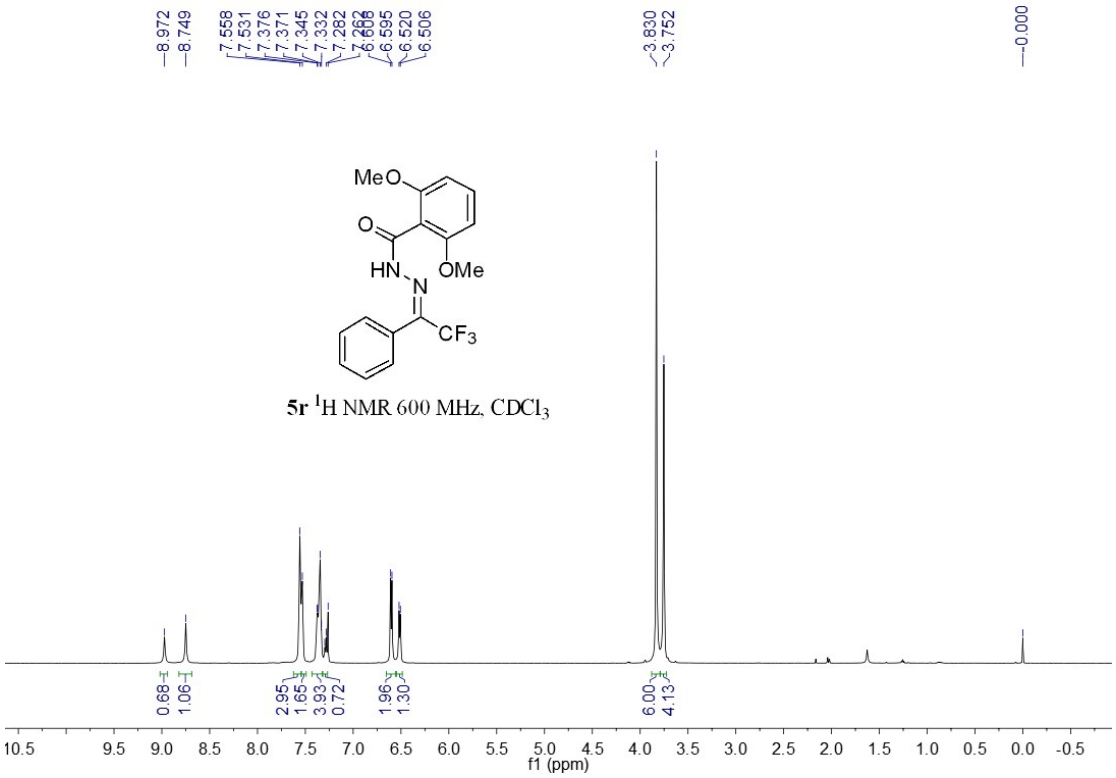


5q ^{13}C NMR 150 MHz, $CDCl_3$

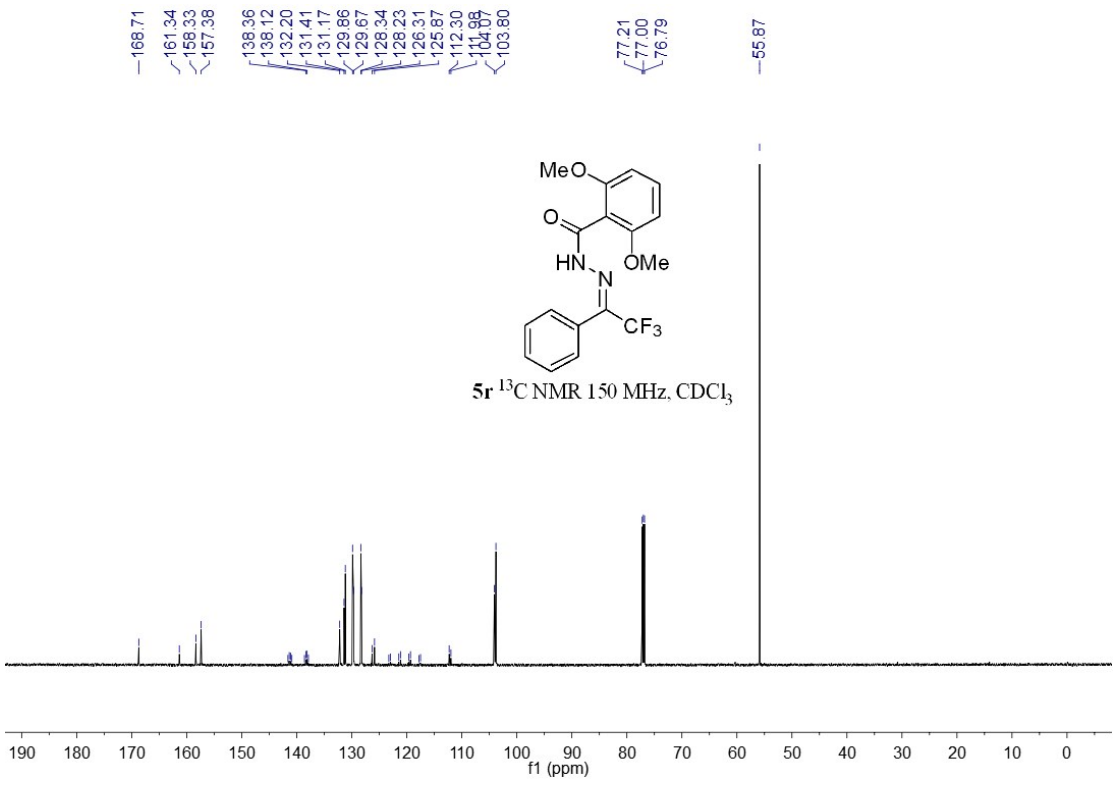


5q ^{19}F NMR 376 MHz, $CDCl_3$

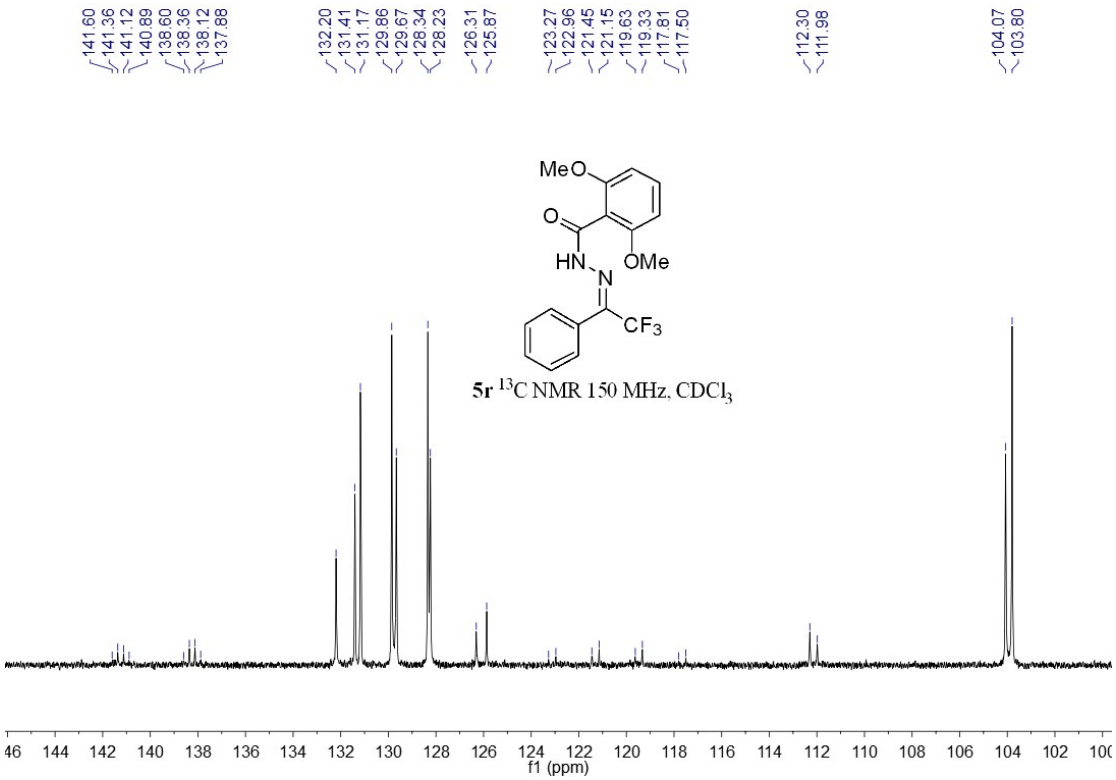
NMR copies of major product of compound **5r**:



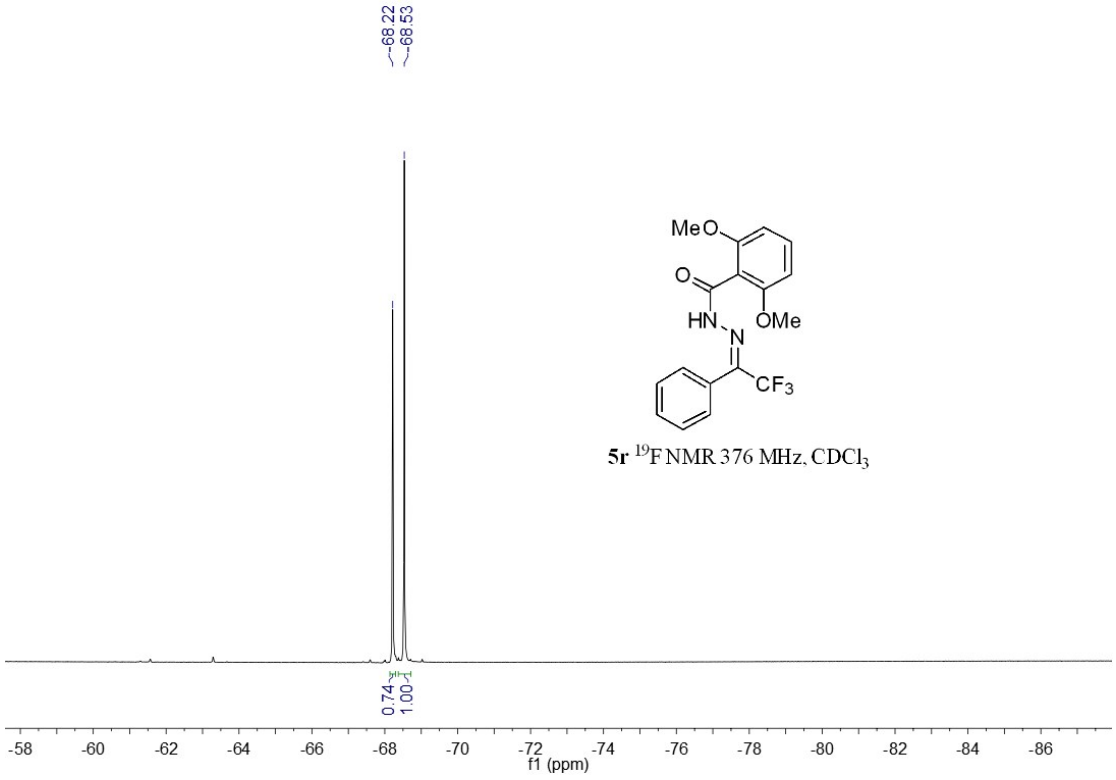
5r ^{1}H NMR 600 MHz, $CDCl_3$



5r ^{13}C NMR 150 MHz, $CDCl_3$

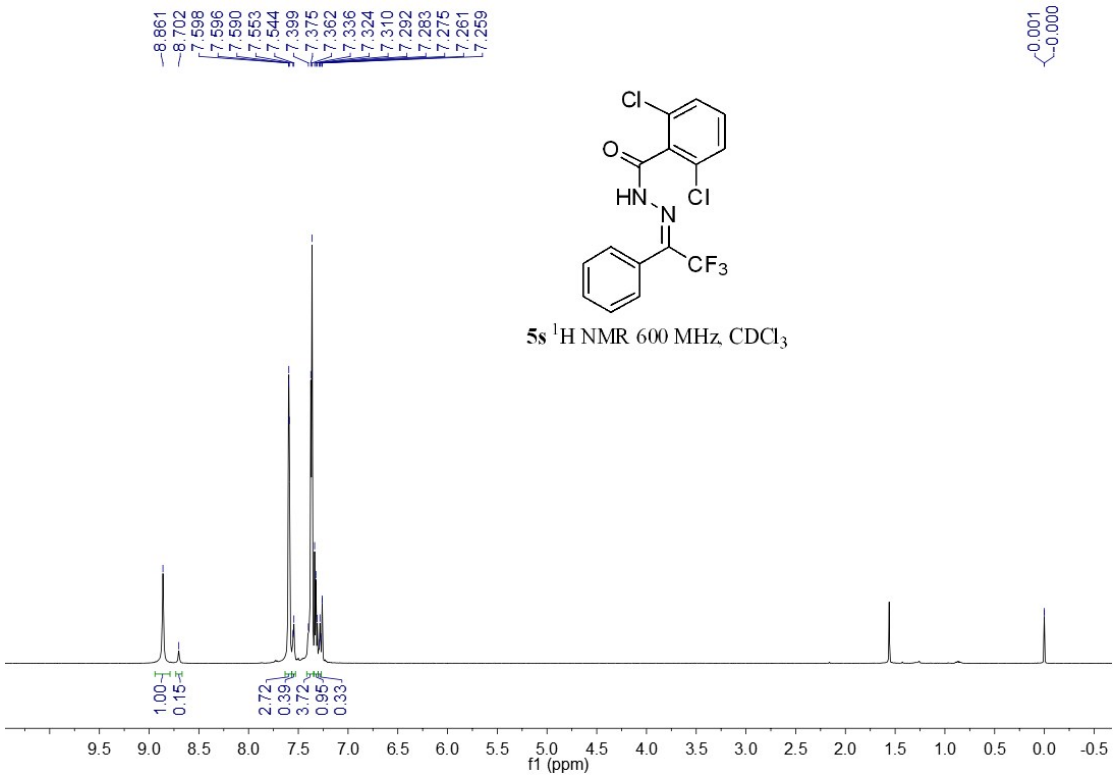


5r ^{13}C NMR 150 MHz, $CDCl_3$

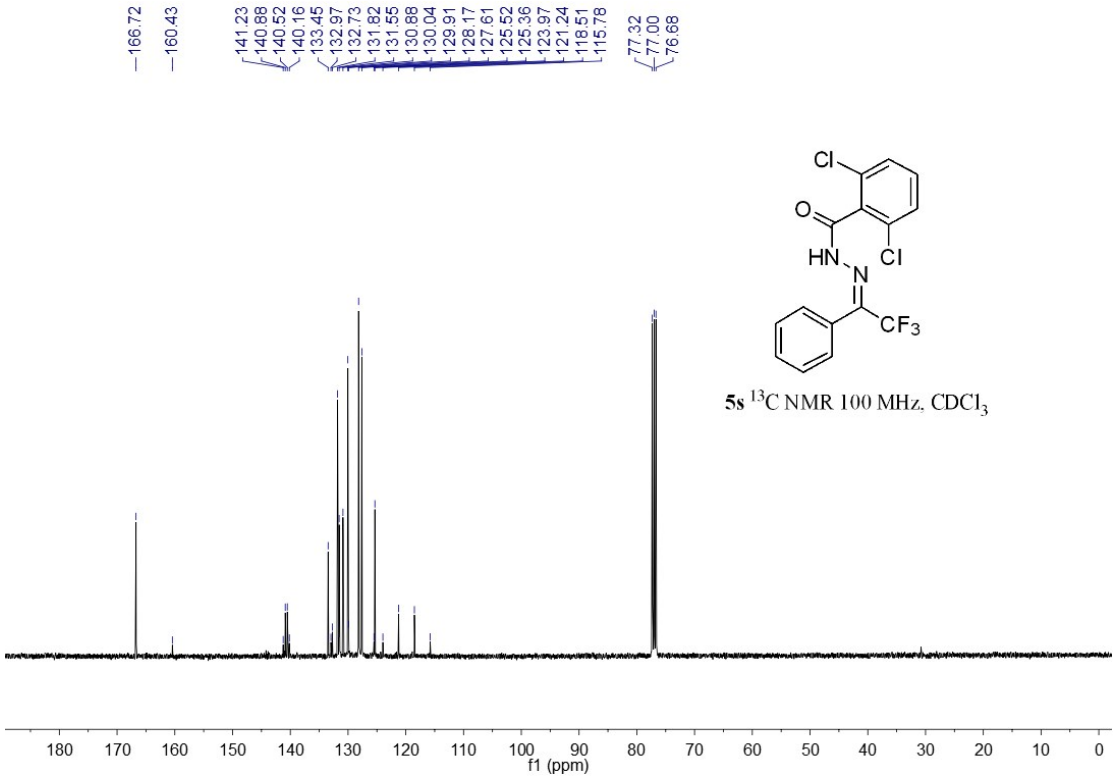


5r ^{19}F NMR 376 MHz, $CDCl_3$

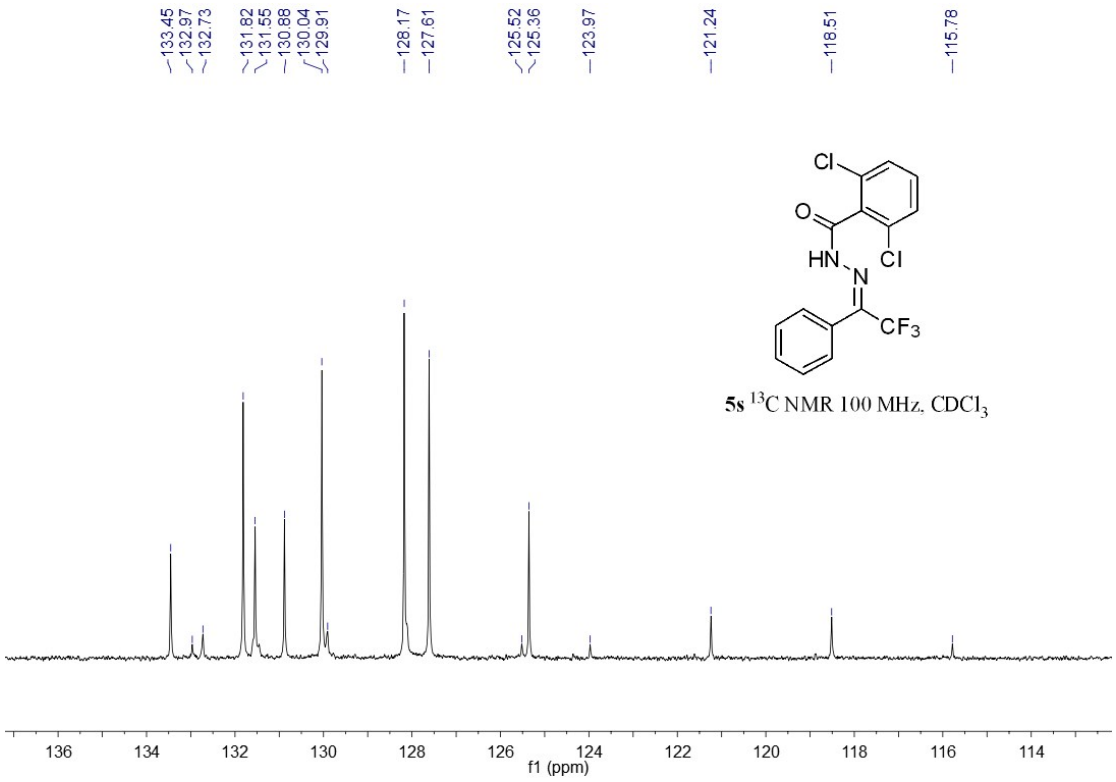
NMR copies of major product of compound **5s**:



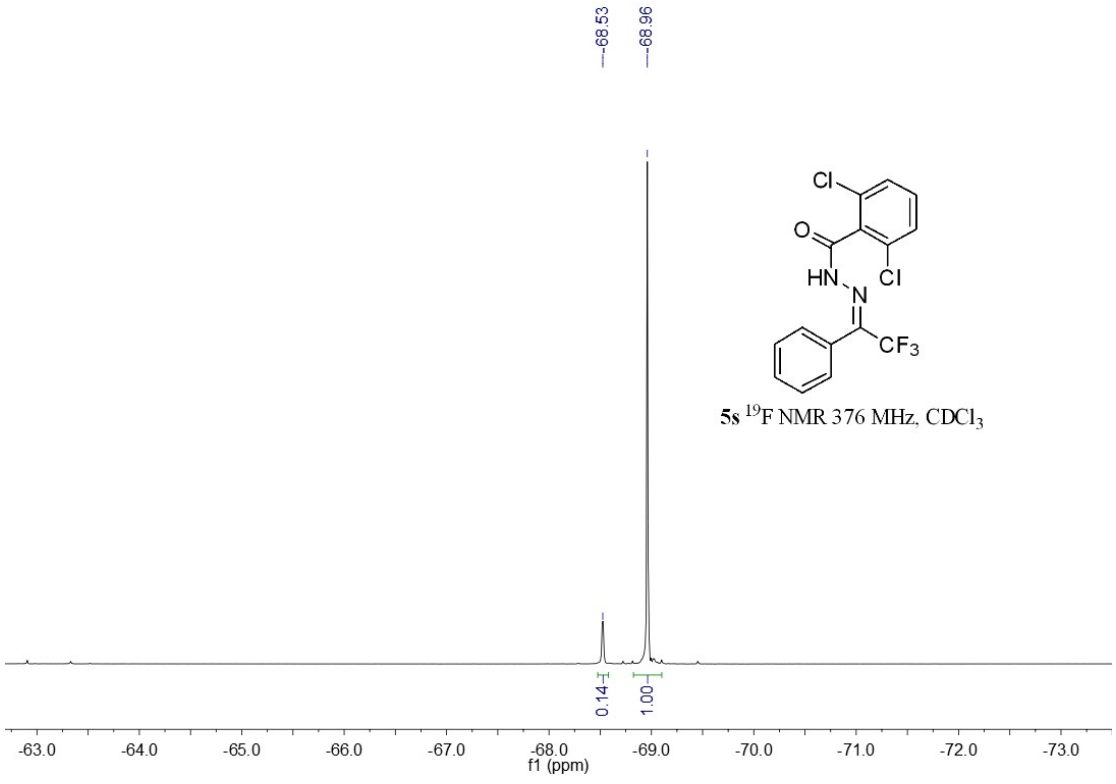
5s ^{1}H NMR 600 MHz, $CDCl_3$



5s ^{13}C NMR 100 MHz, $CDCl_3$

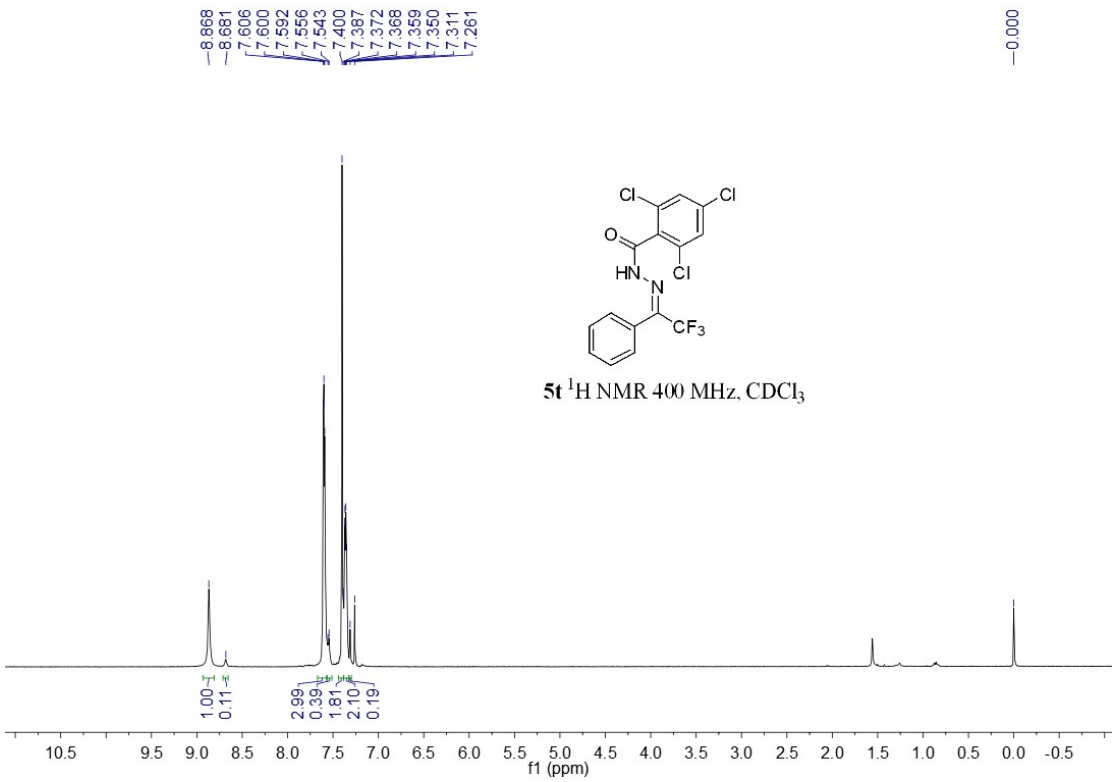


5s ^{13}C NMR 100 MHz, $CDCl_3$

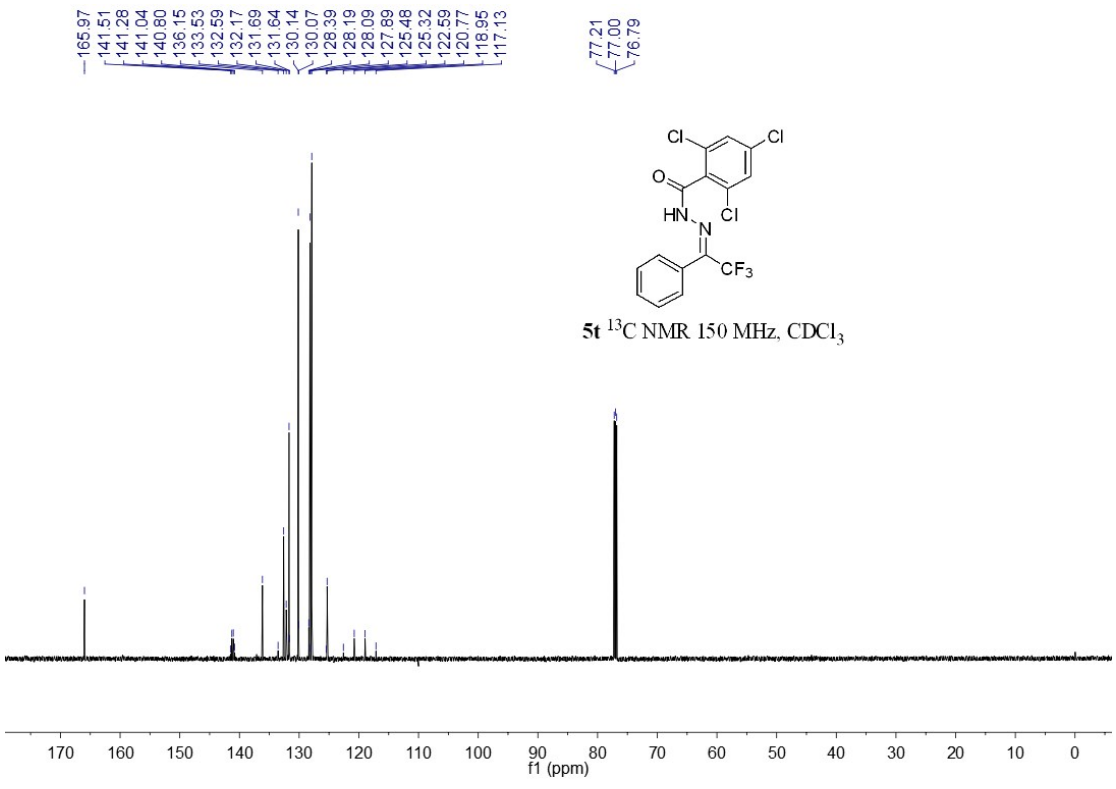


5s ^{19}F NMR 376 MHz, $CDCl_3$

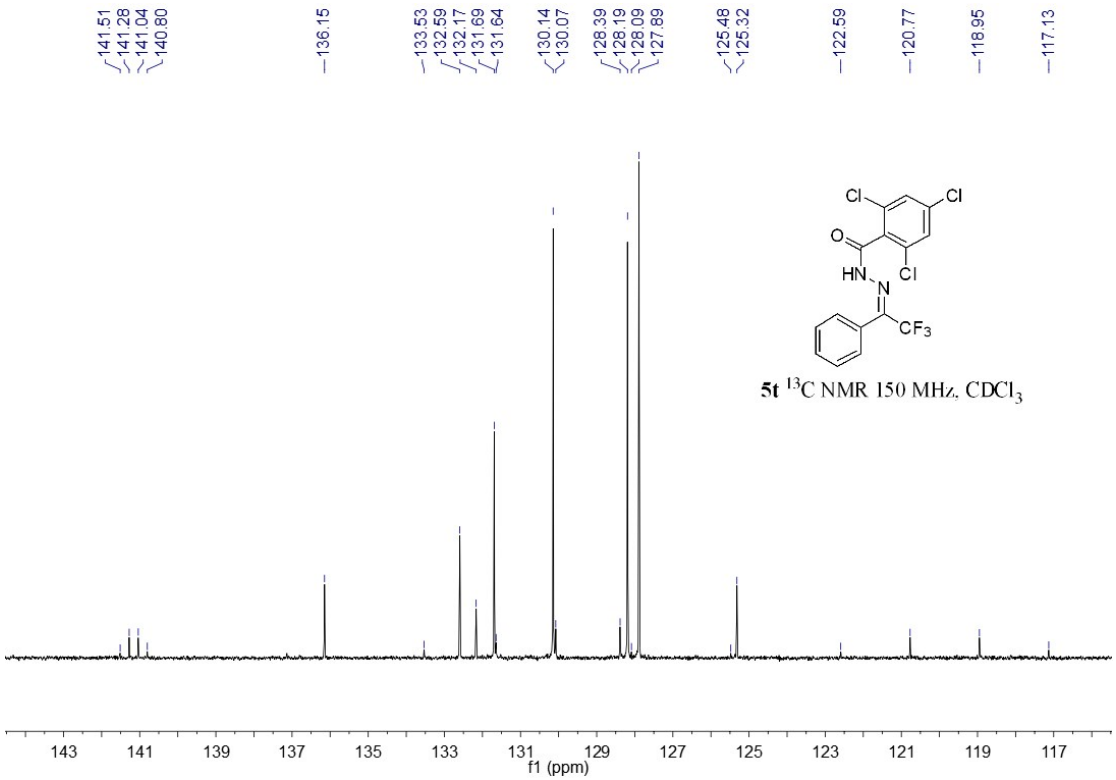
NMR copies of major product of compound **5t**:



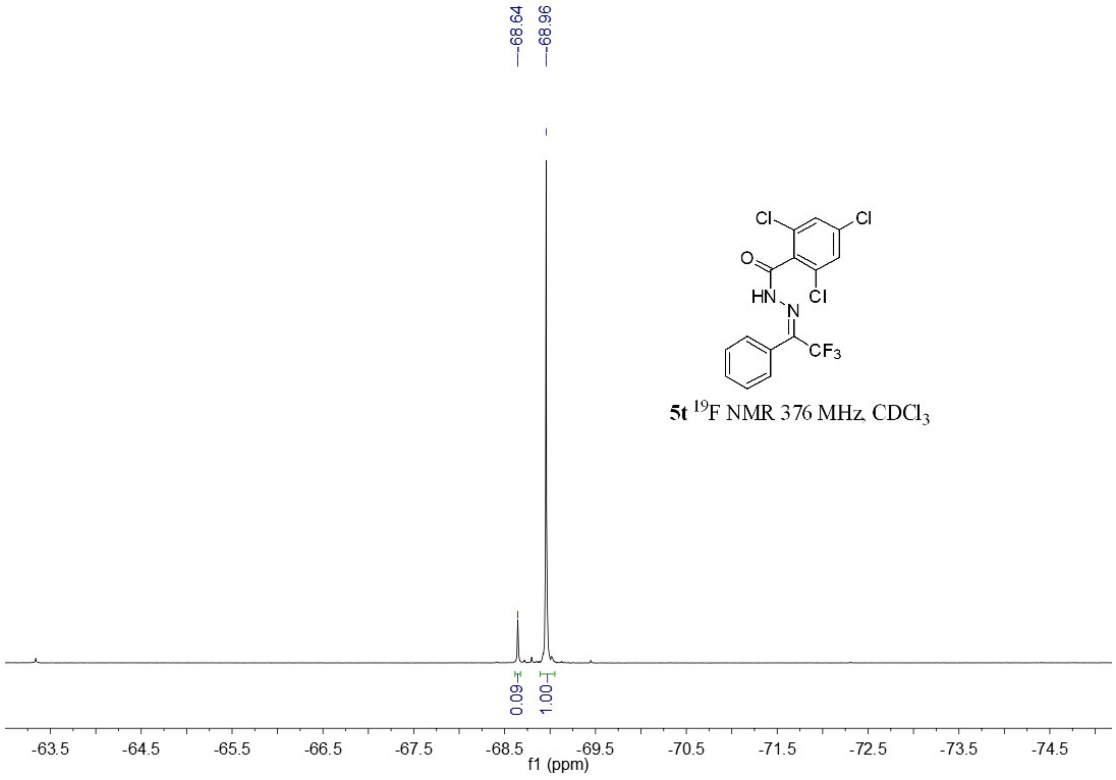
5t ^{1}H NMR 400 MHz, $CDCl_3$



5t ^{13}C NMR 150 MHz, $CDCl_3$

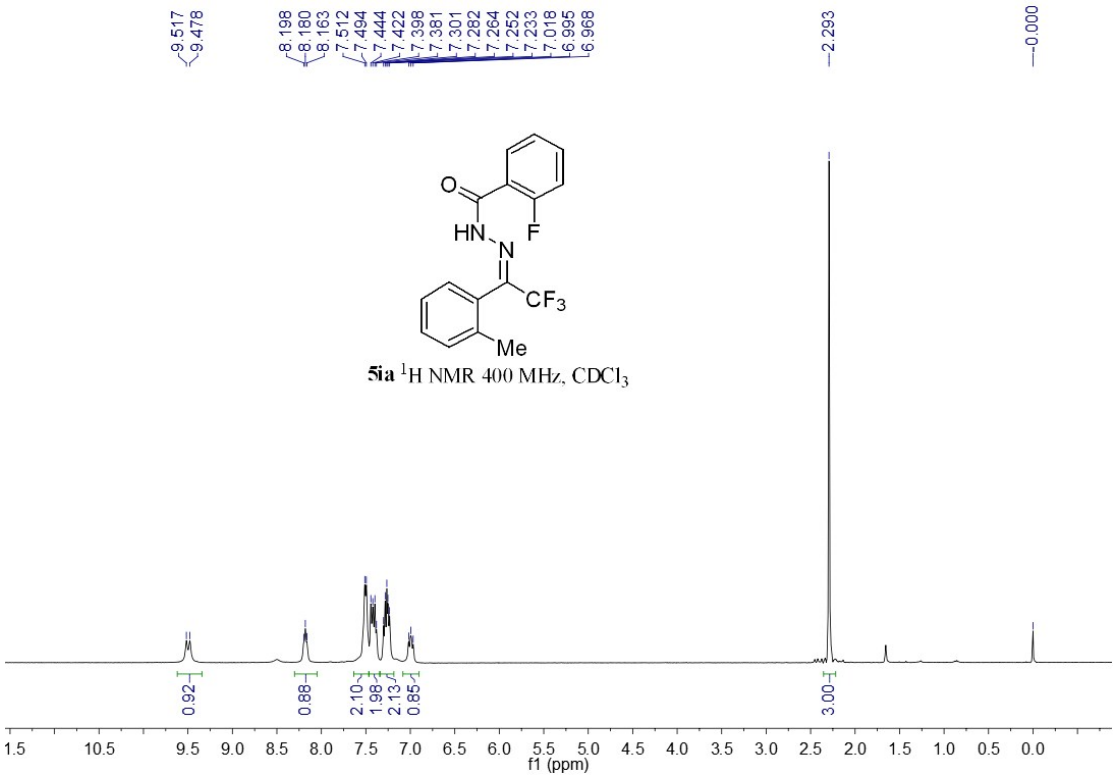


5t ^{13}C NMR 150 MHz, $CDCl_3$

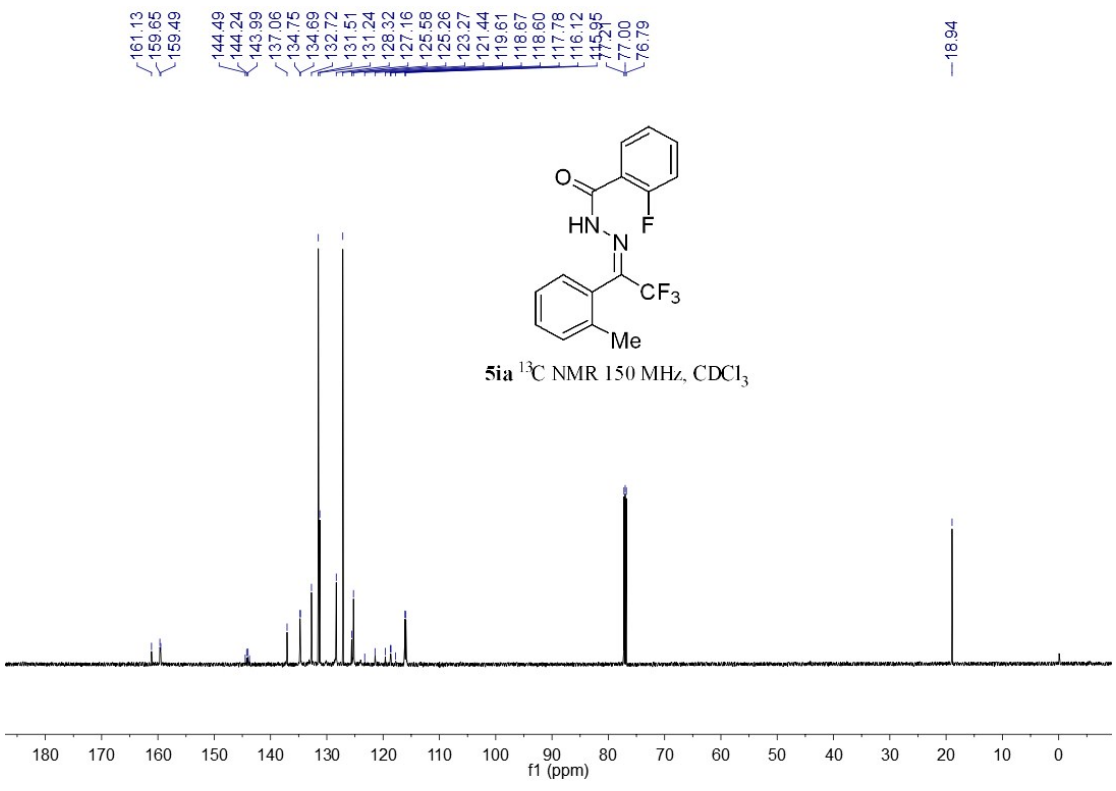


5t ^{19}F NMR 376 MHz, $CDCl_3$

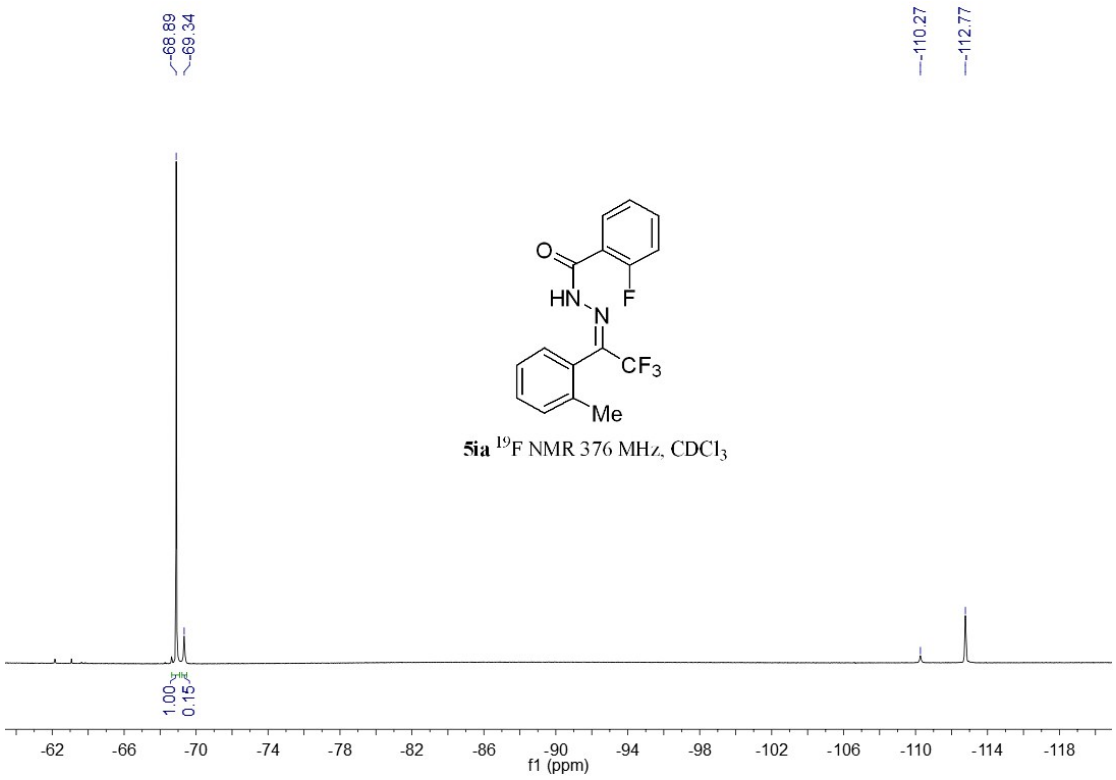
NMR copies of major product of compound **5ia**:



5ia ^{1}H NMR 400 MHz, $CDCl_3$

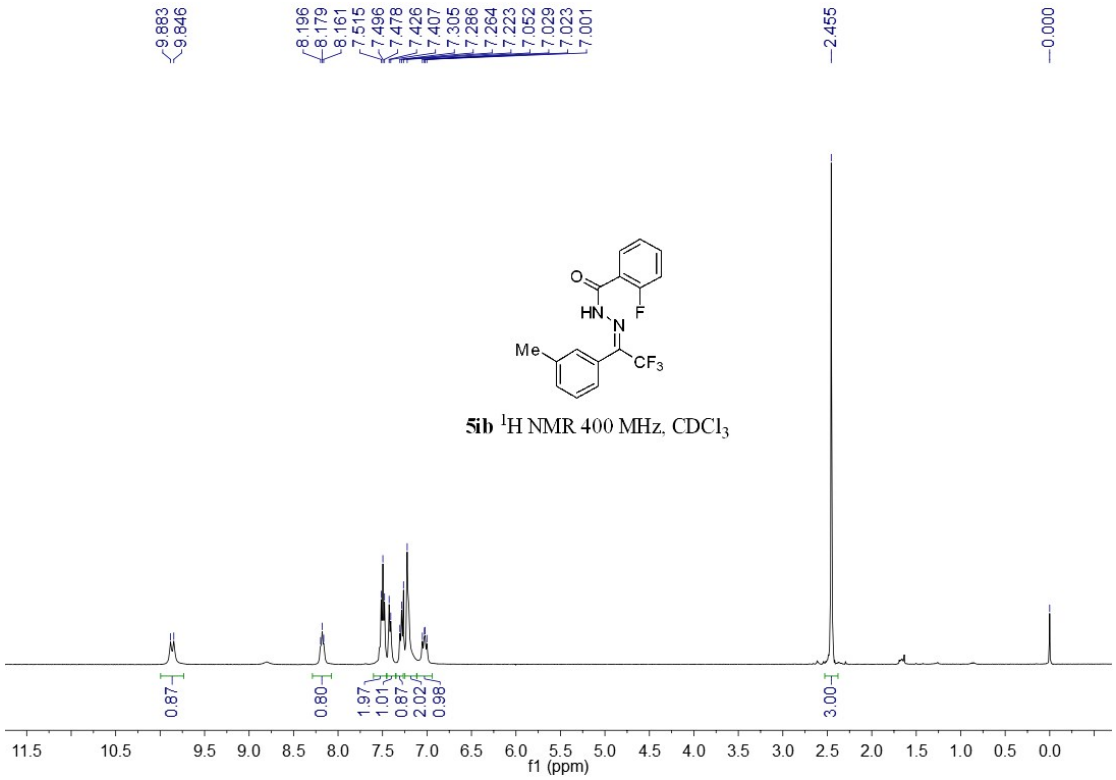


5ia ^{13}C NMR 150 MHz, $CDCl_3$

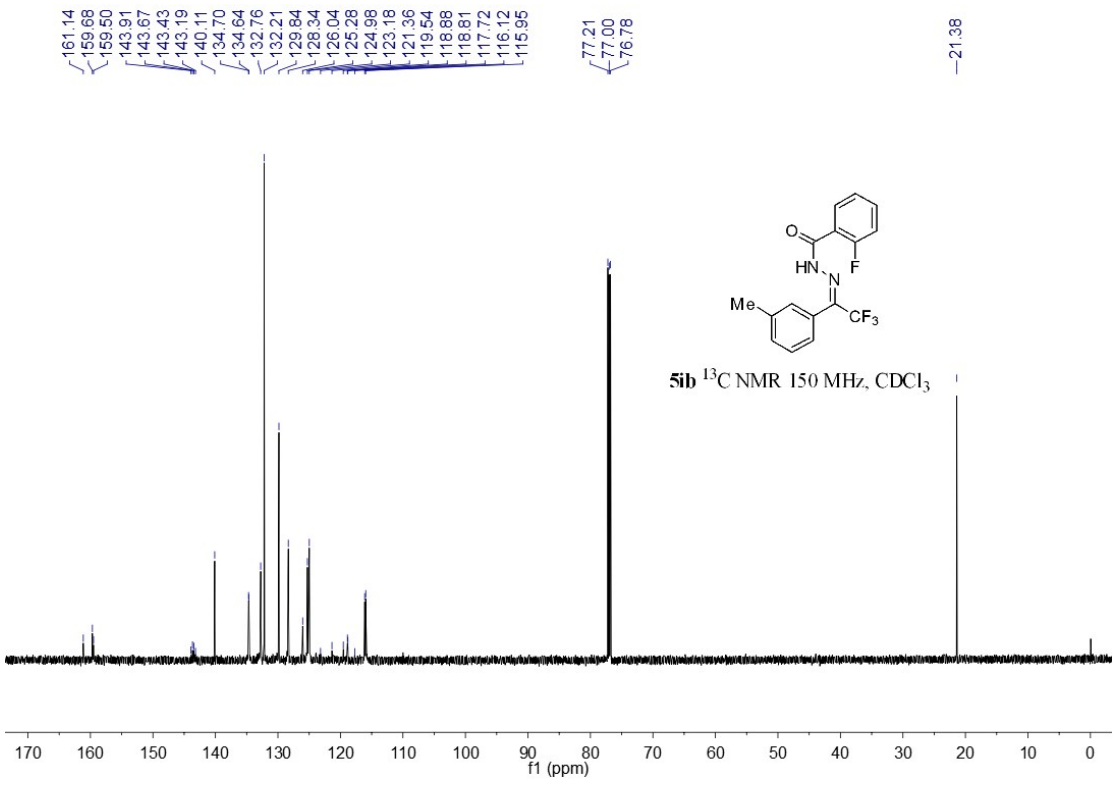


5ia ^{19}F NMR 376 MHz, $CDCl_3$

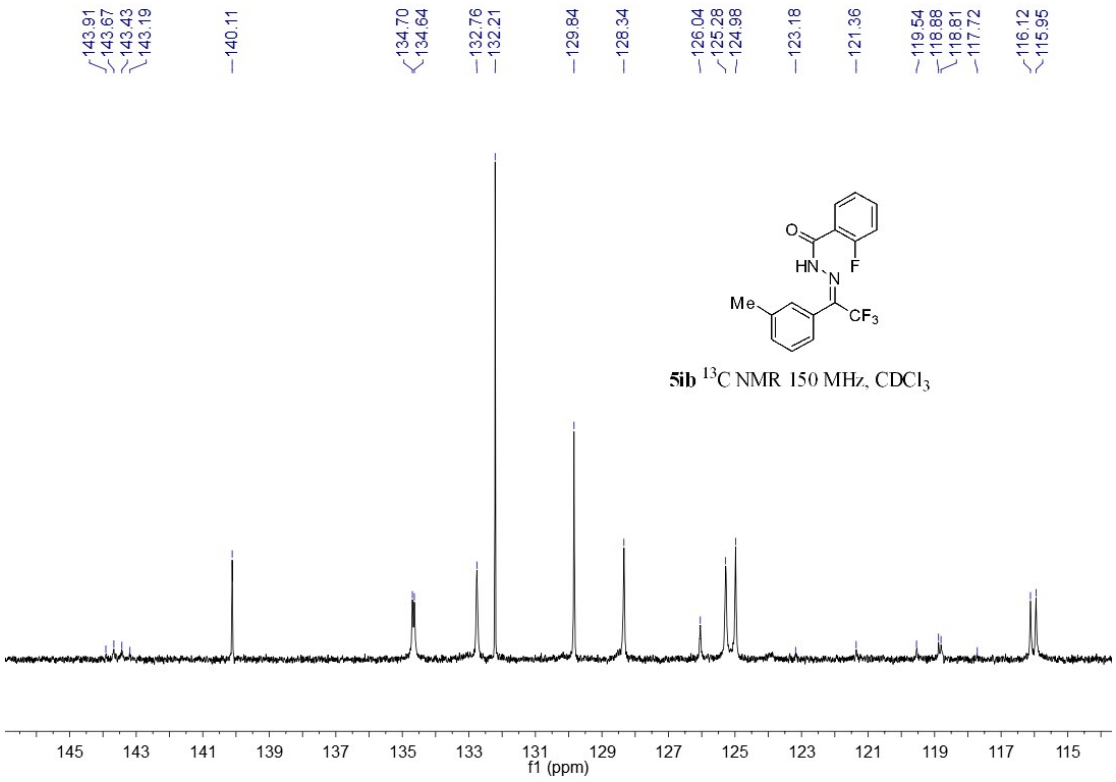
NMR copies of major product of compound **5ib**:



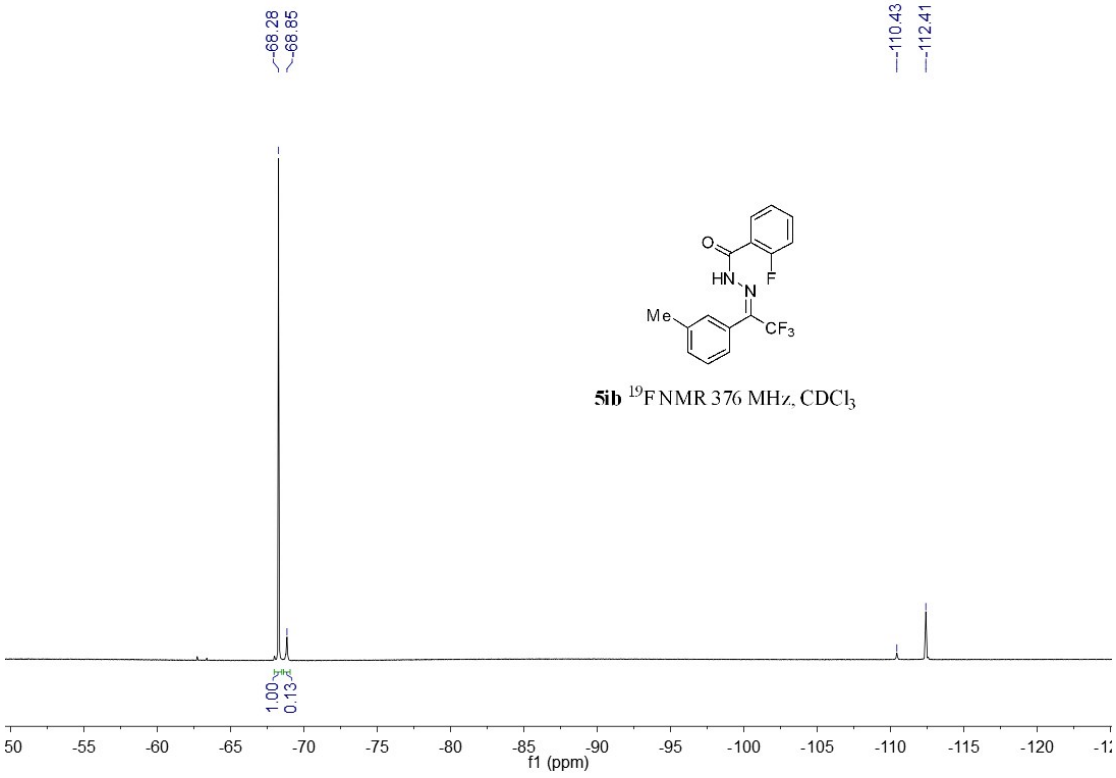
5ib ^{1}H NMR 400 MHz, $CDCl_3$



5ib ^{13}C NMR 150 MHz, $CDCl_3$

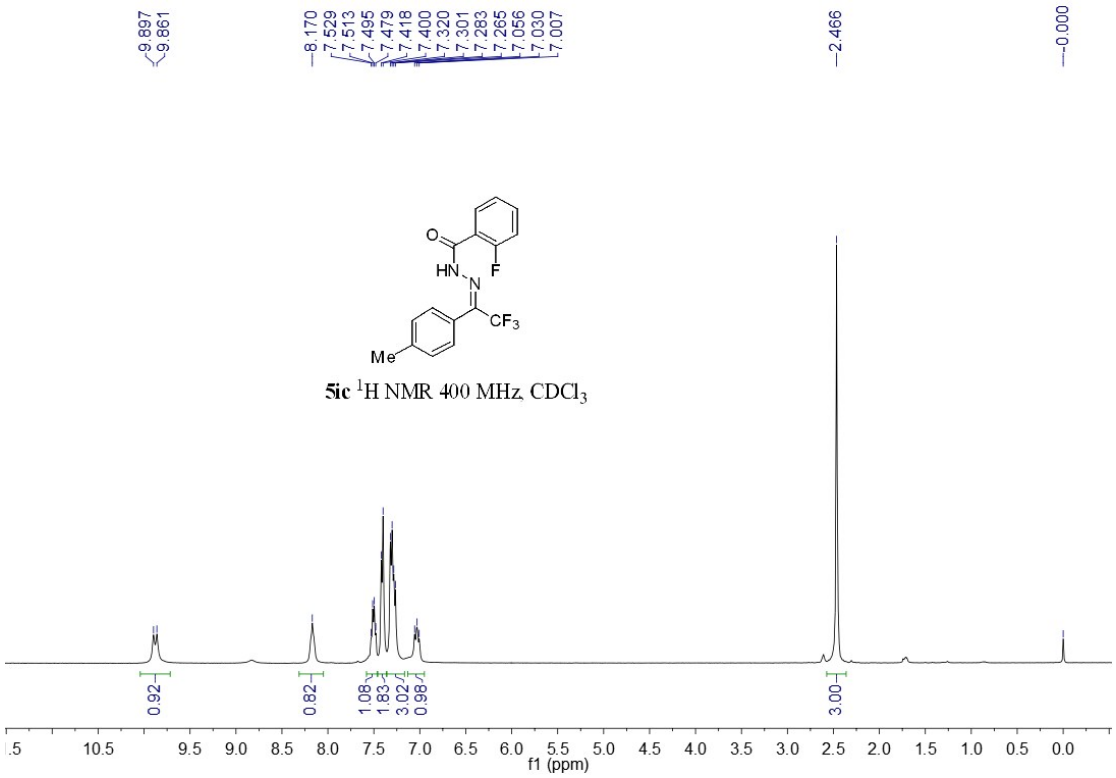


5ib ^{13}C NMR 150 MHz, $CDCl_3$

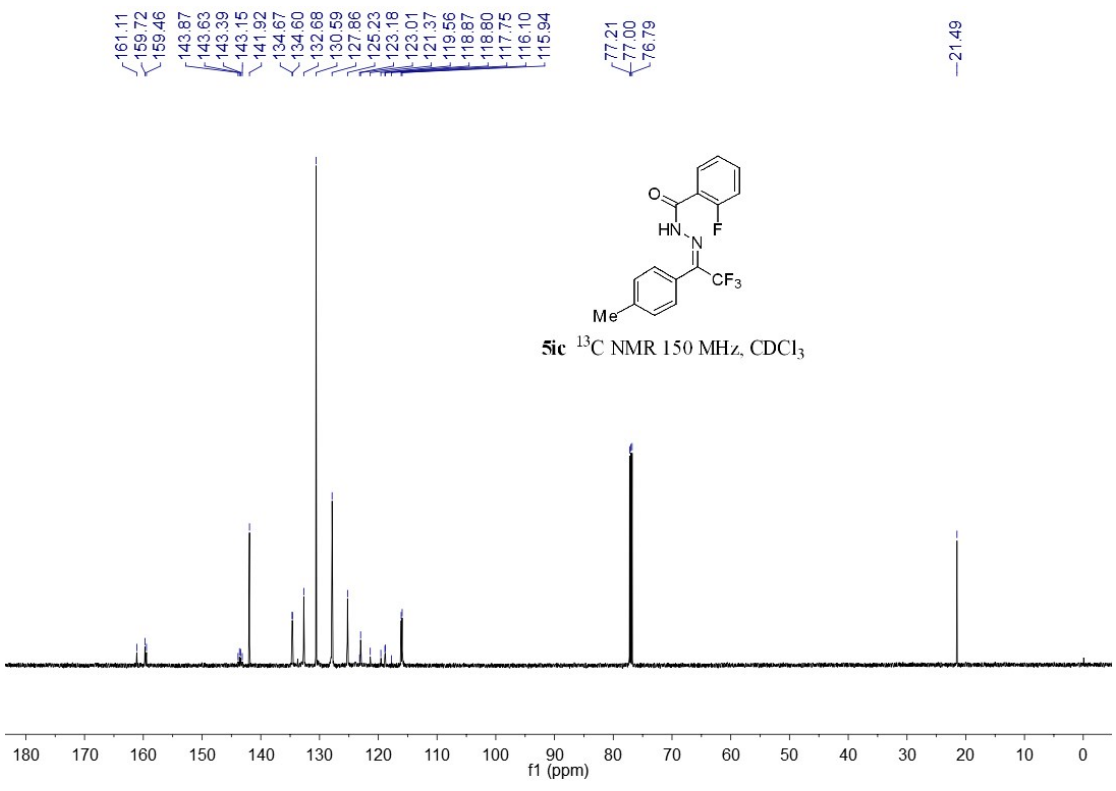


5ib ^{19}F NMR 376 MHz, $CDCl_3$

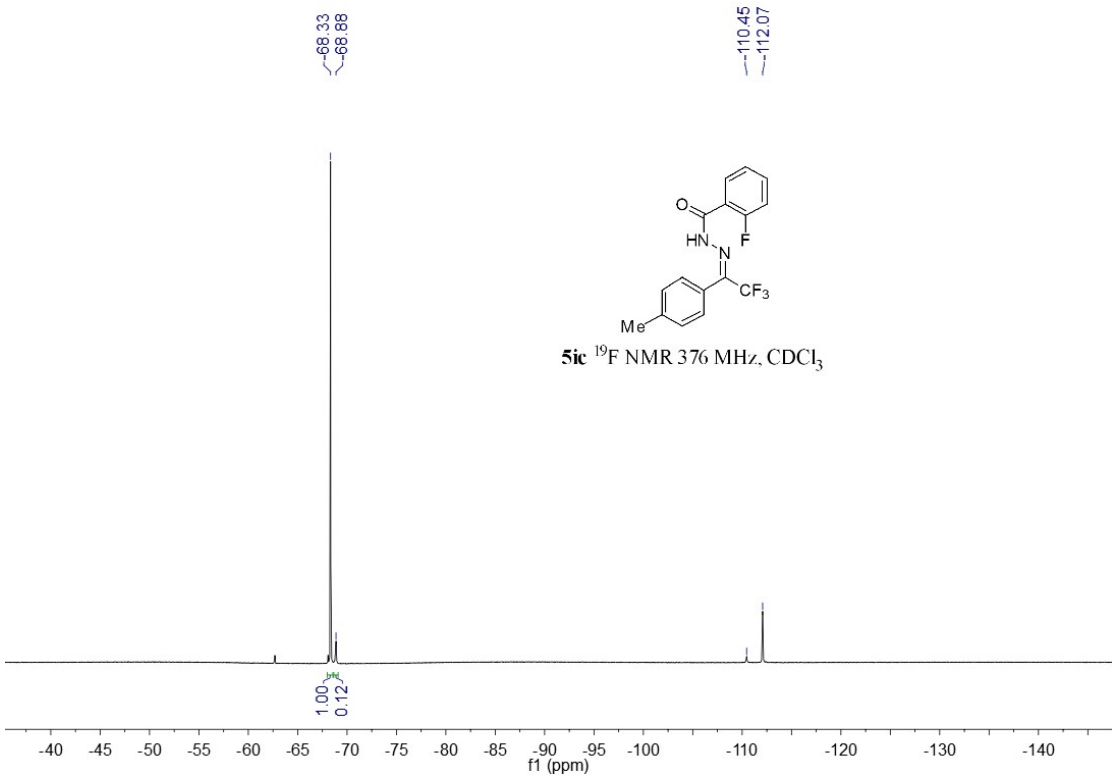
NMR copies of major product of compound **5ic**:



5ic ^{1}H NMR 400 MHz, $CDCl_3$

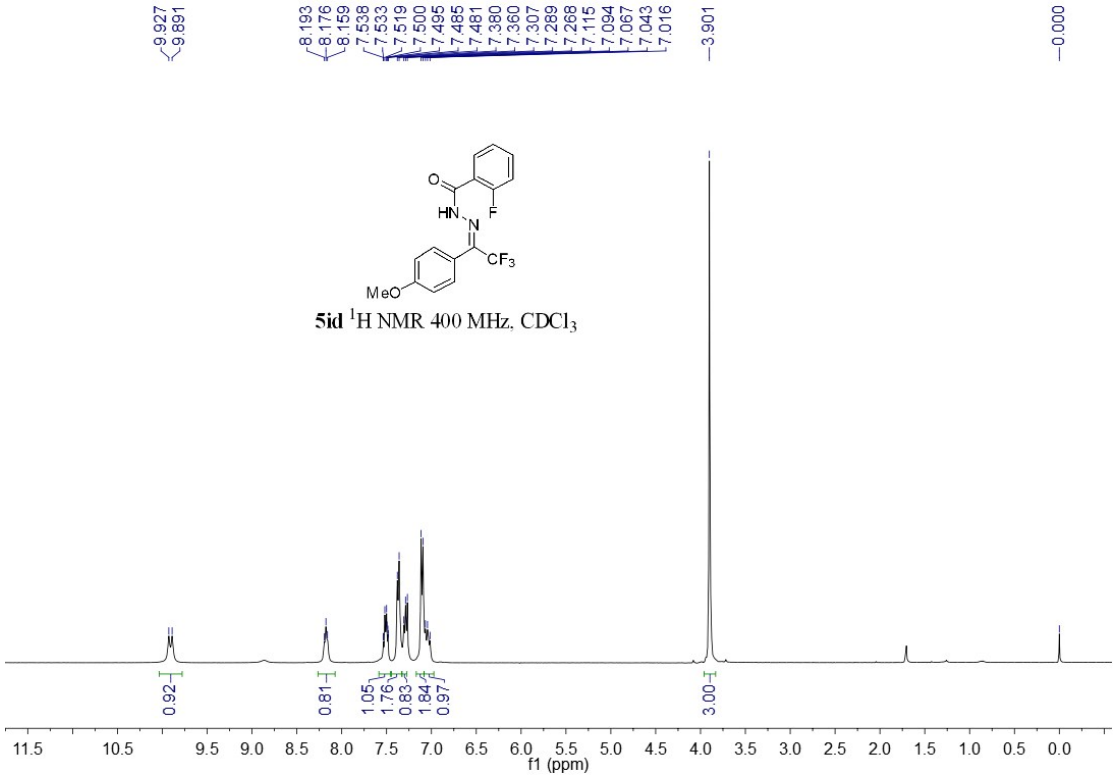


5ic ^{13}C NMR 150 MHz, $CDCl_3$

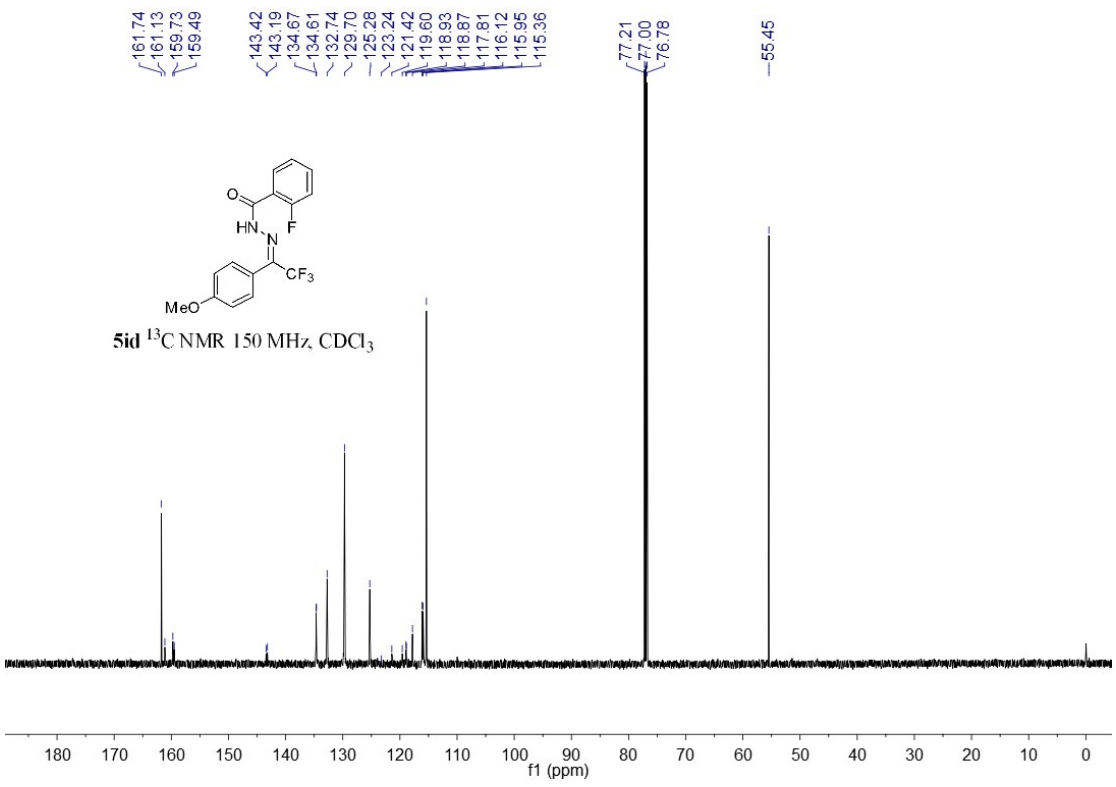


5ic ^{19}F NMR 376 MHz, $CDCl_3$

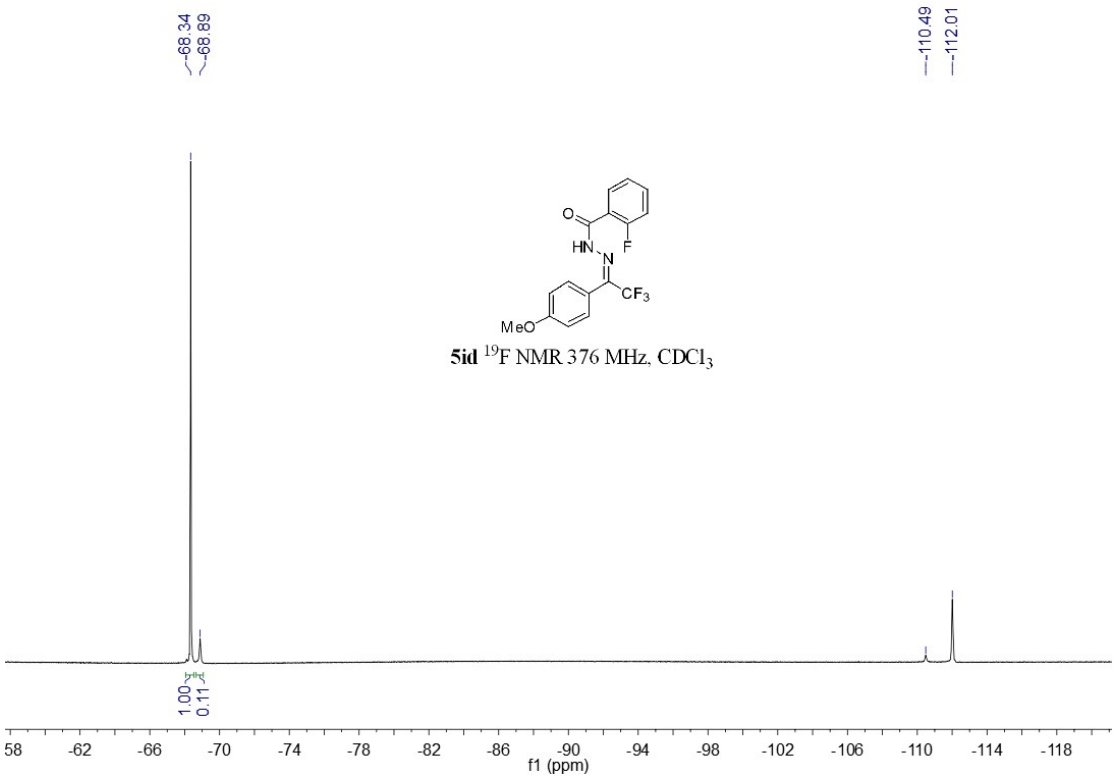
NMR copies of major product of compound **5id**:



5id ^{1}H NMR 400 MHz, $CDCl_3$

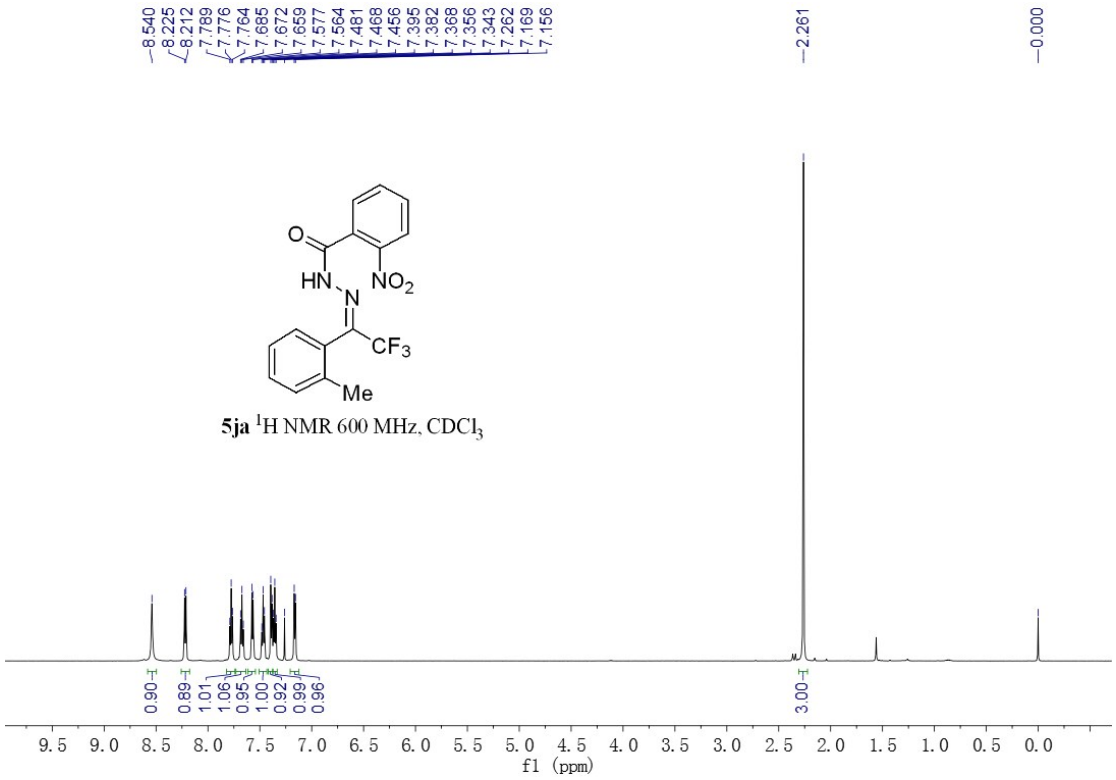


5id ^{13}C NMR 150 MHz, $CDCl_3$

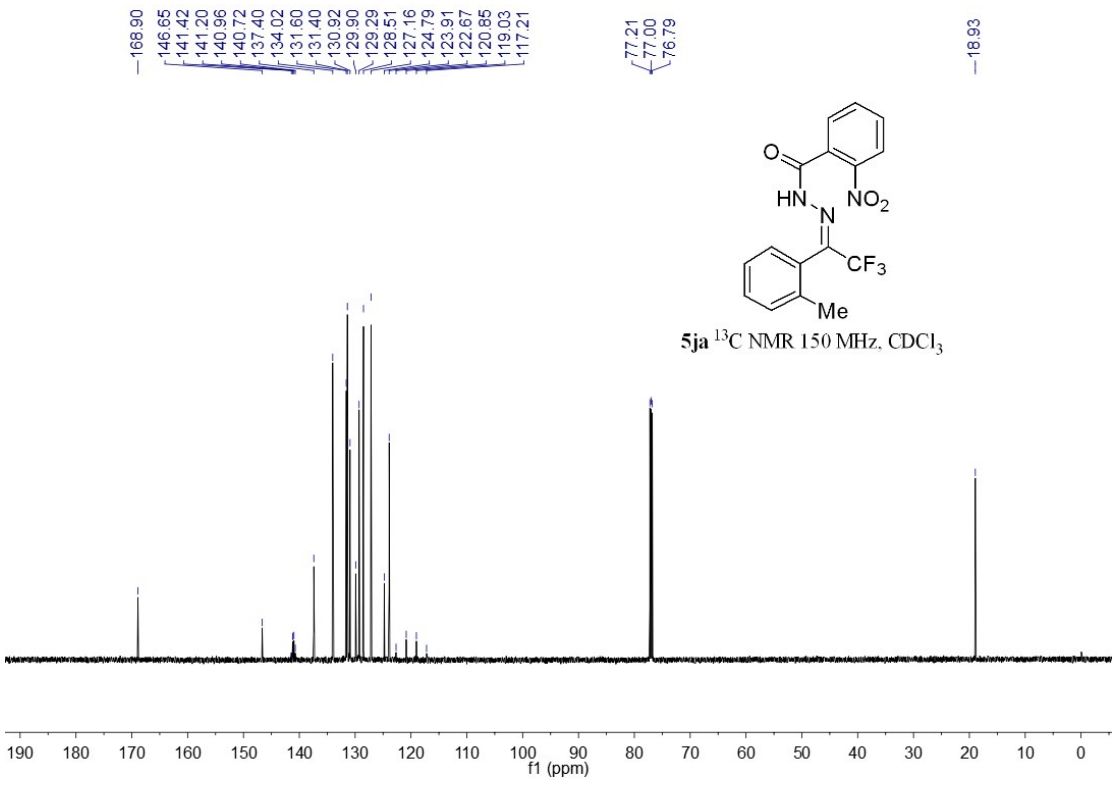


5id ^{19}F NMR 376 MHz, $CDCl_3$

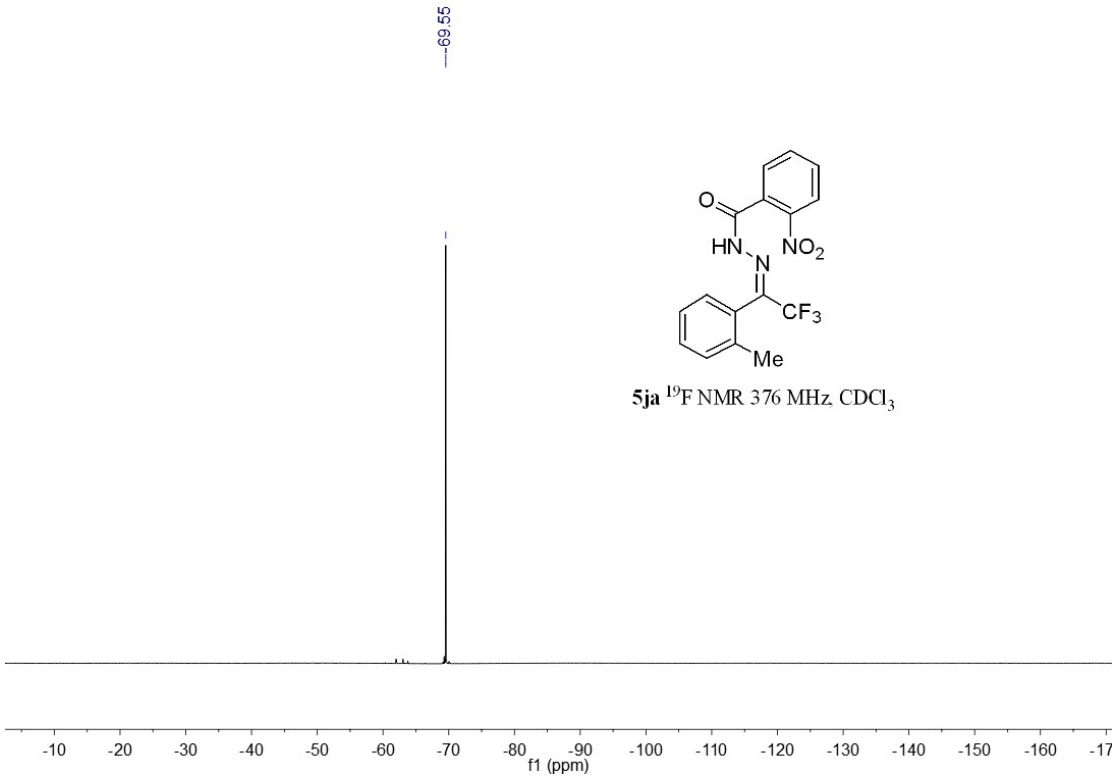
NMR copies of major product of compound **5ja**:



5ja ^{1}H NMR 600 MHz, $CDCl_3$

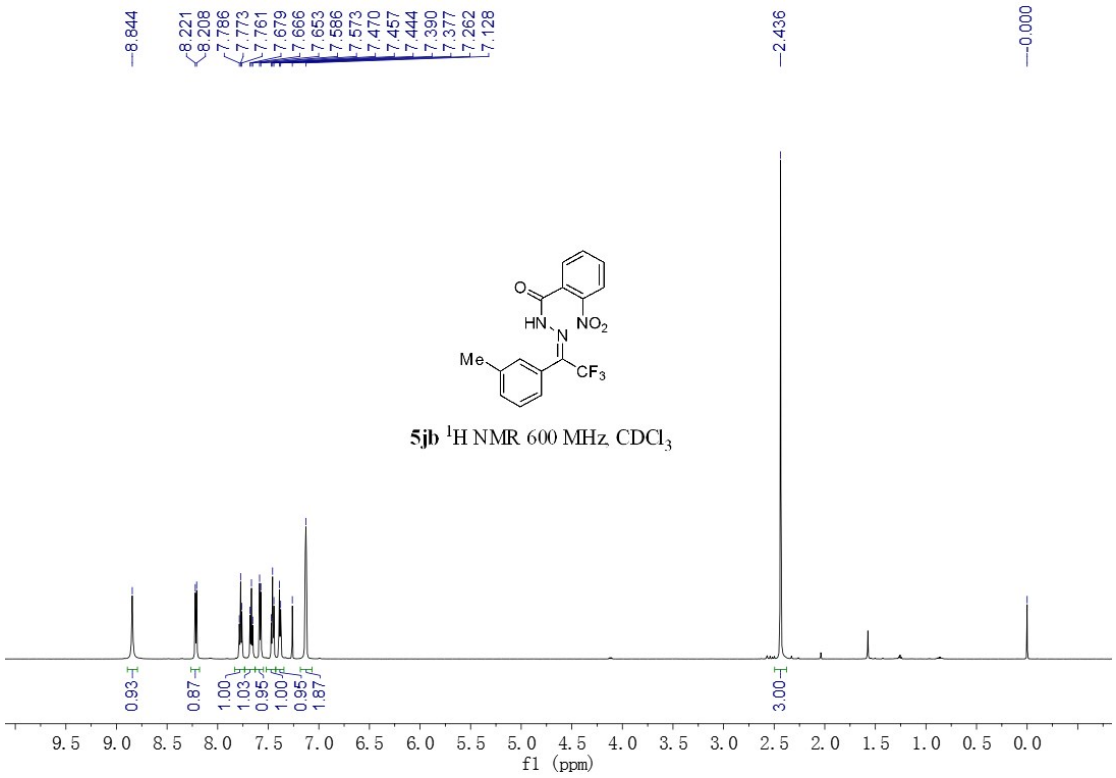


5ja ^{13}C NMR 150 MHz, $CDCl_3$

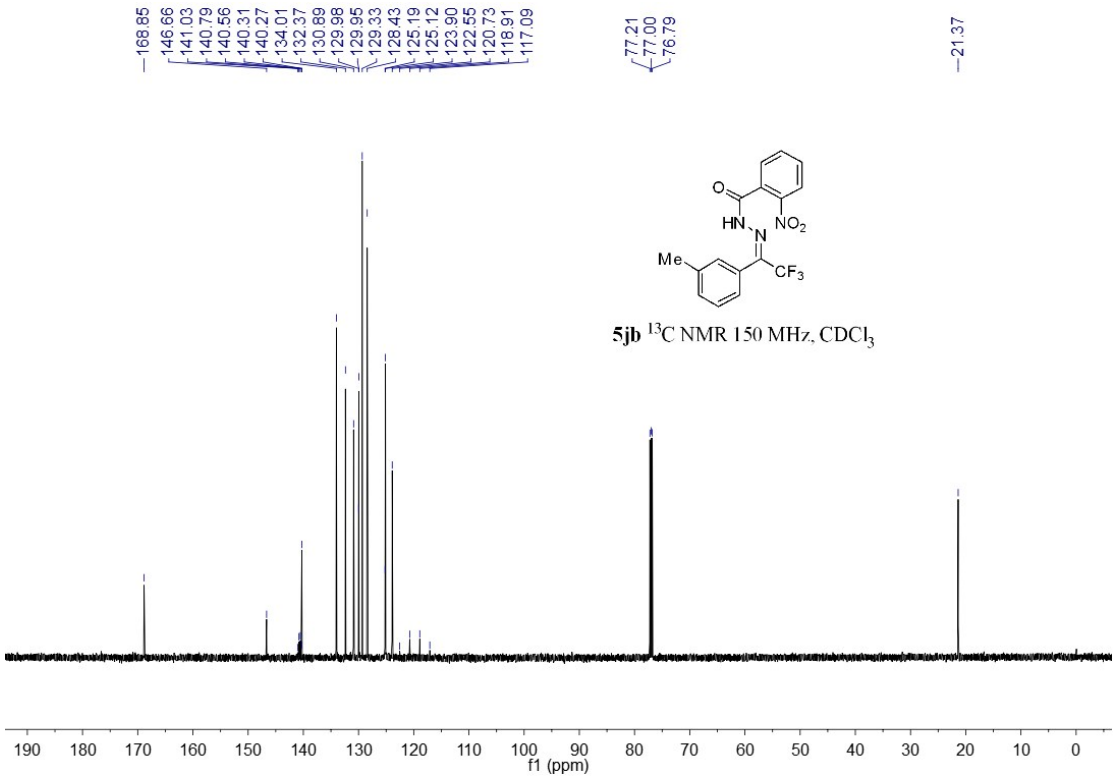


5ja ^{19}F NMR 376 MHz, $CDCl_3$

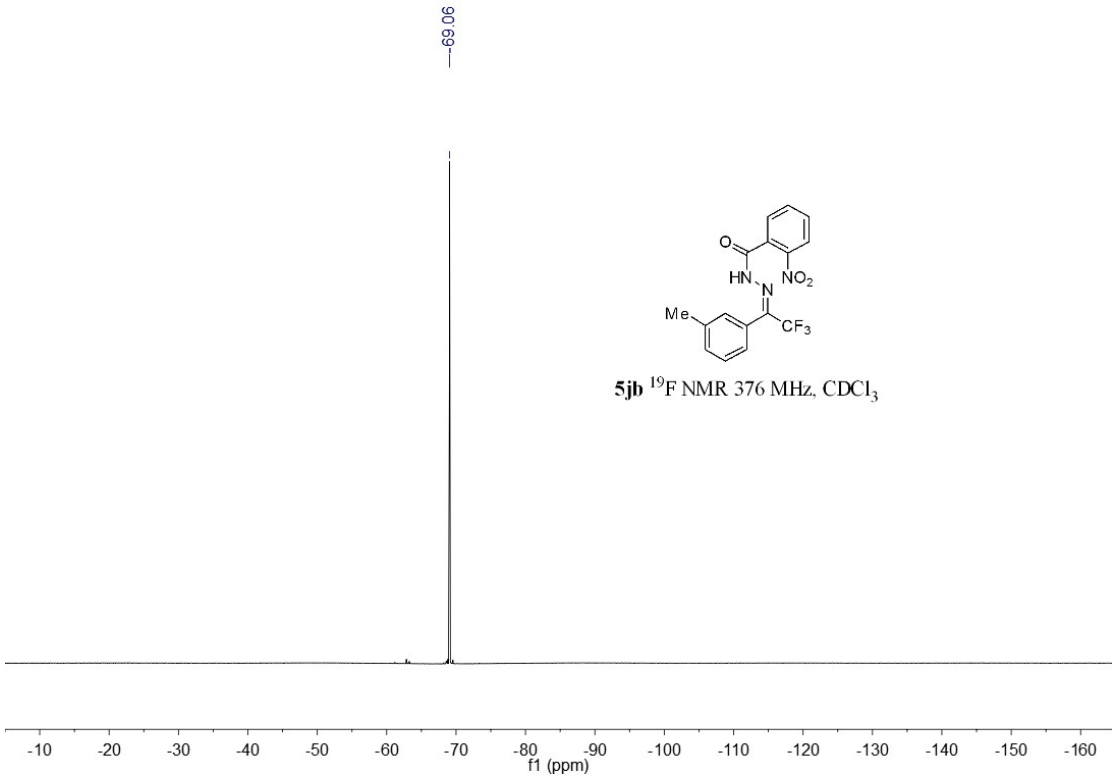
NMR copies of major product of compound **5jb**:



5jb ^{1}H NMR 600 MHz, $CDCl_3$

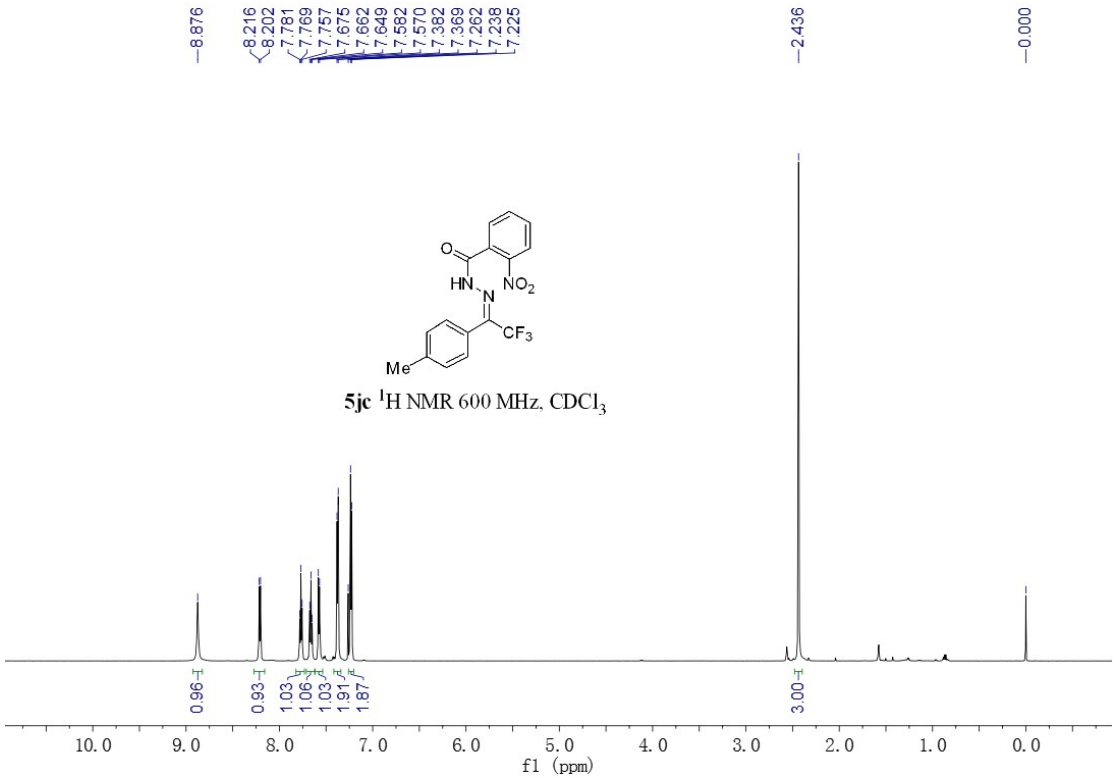


5jb ^{13}C NMR 150 MHz, $CDCl_3$

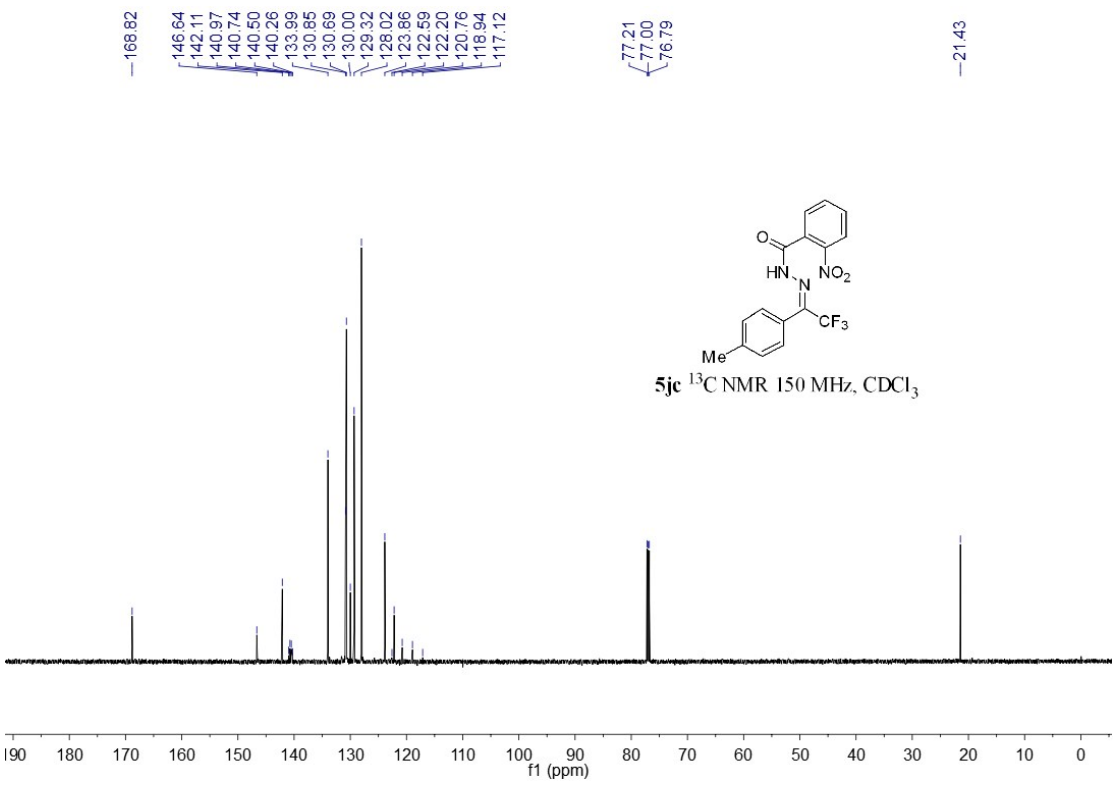


5jb ^{19}F NMR 376 MHz, $CDCl_3$

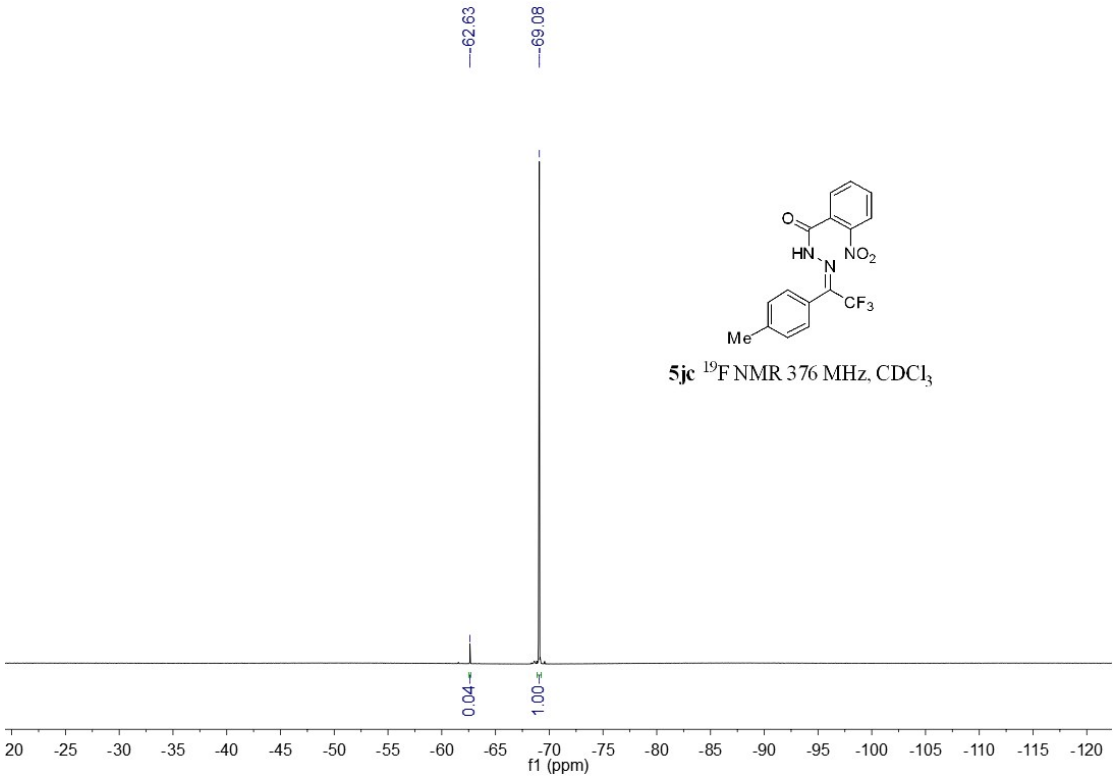
NMR copies of major product of compound **5jc**:



5jc ^{1}H NMR 600 MHz, $CDCl_3$

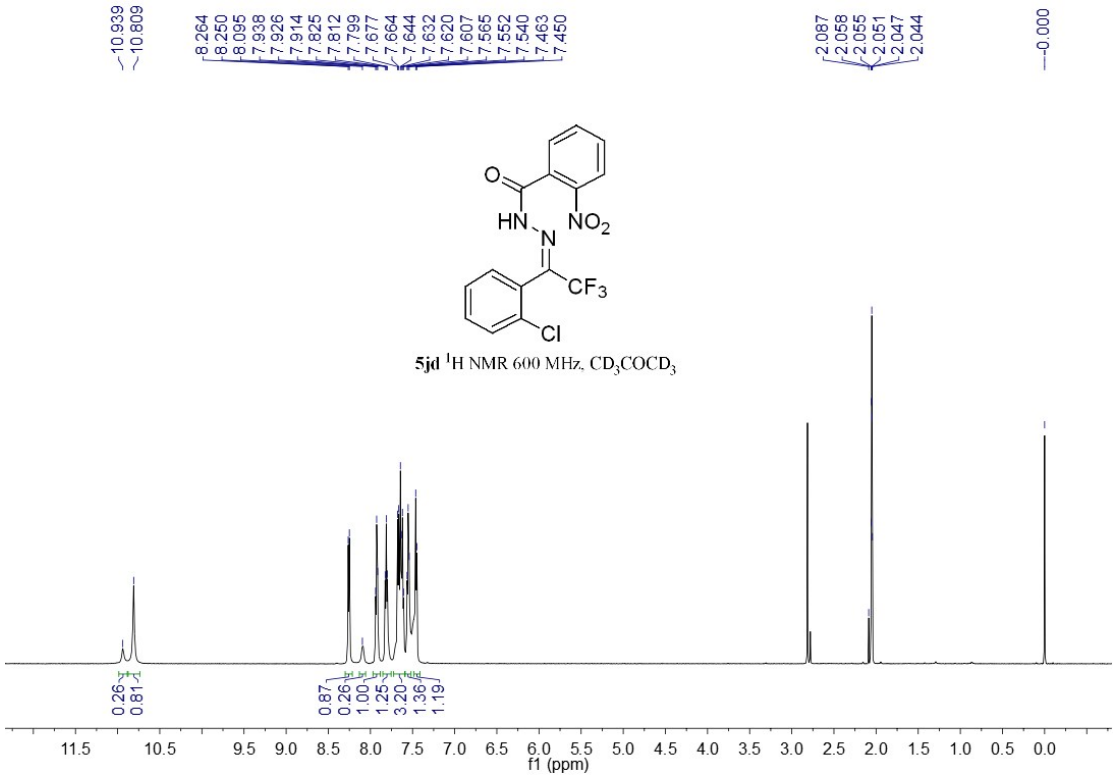


5jc ^{13}C NMR 150 MHz, $CDCl_3$

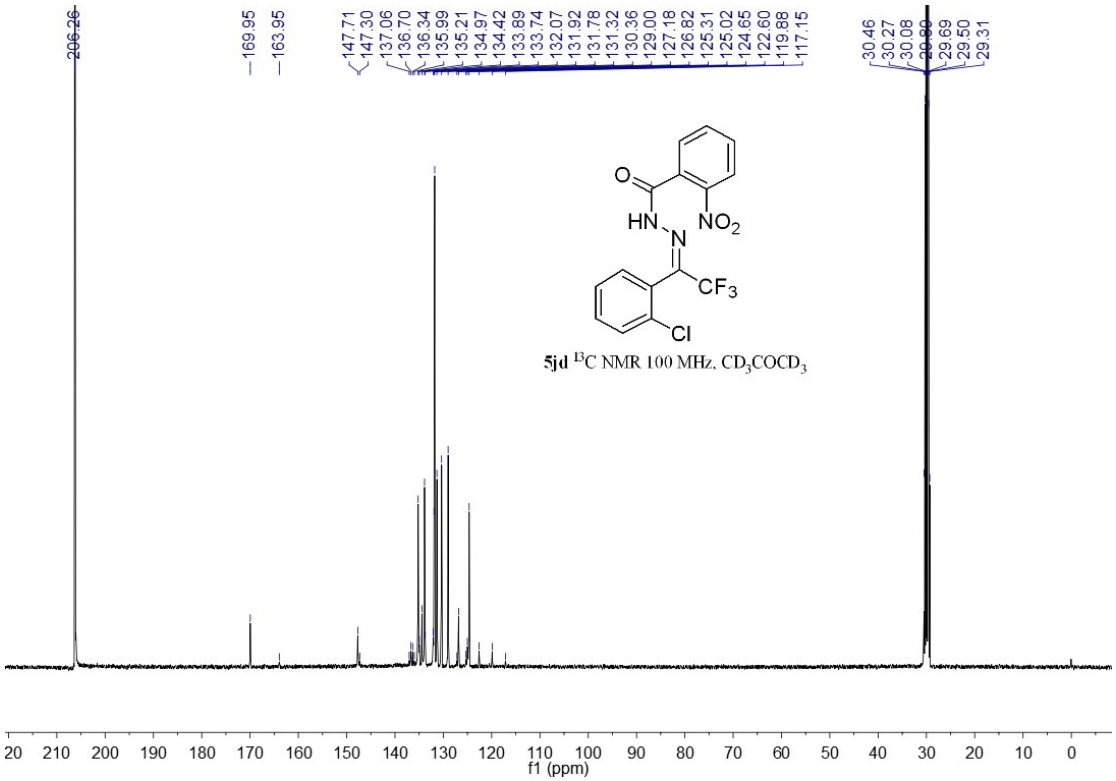


5jc ^{19}F NMR 376 MHz, $CDCl_3$

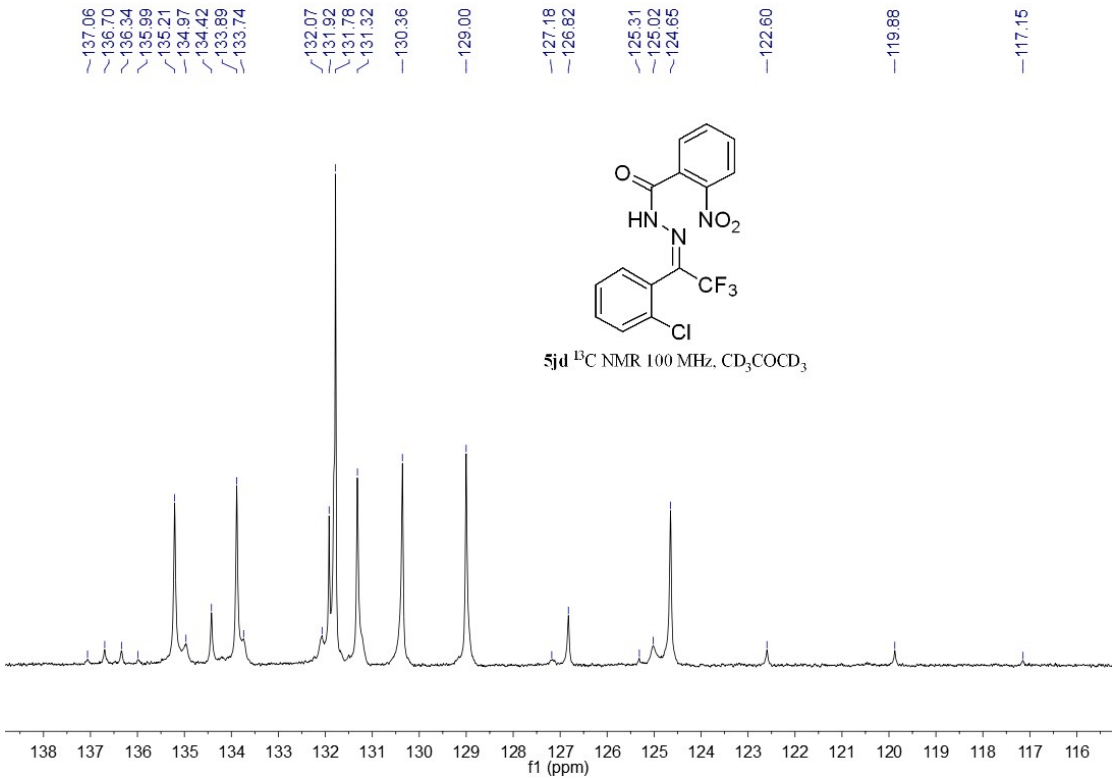
NMR copies of major product of compound **5jd**:



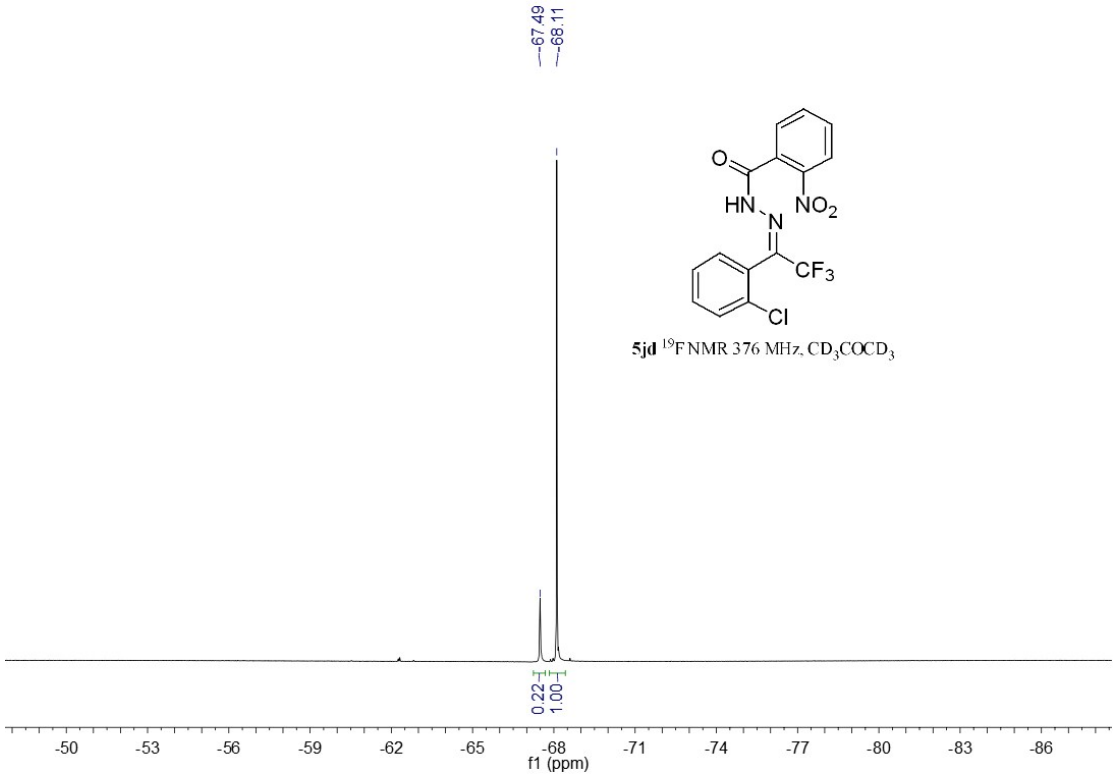
5jd ^{1}H NMR 600 MHz, CD_3COCD_3



5jd ^{13}C NMR 100 MHz, CD_3COCD_3

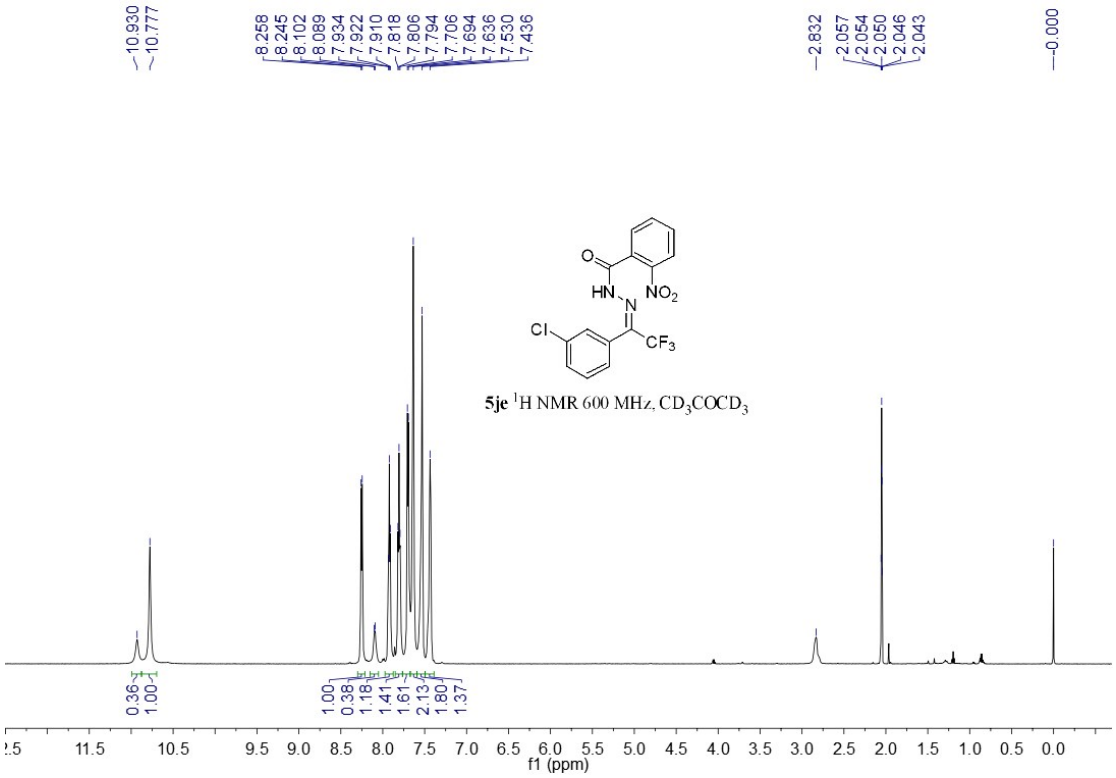


5jd ^{13}C NMR 100 MHz, CD_3COCD_3

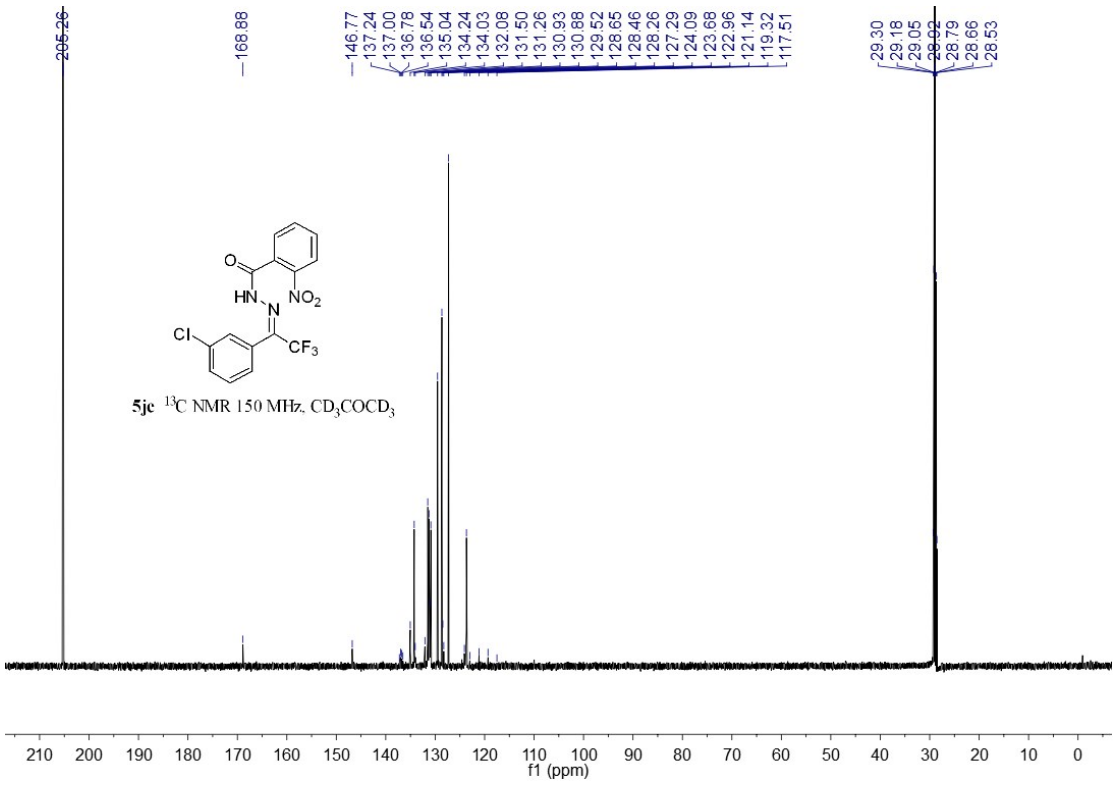


5jd ^{19}F NMR 376 MHz, CD_3COCD_3

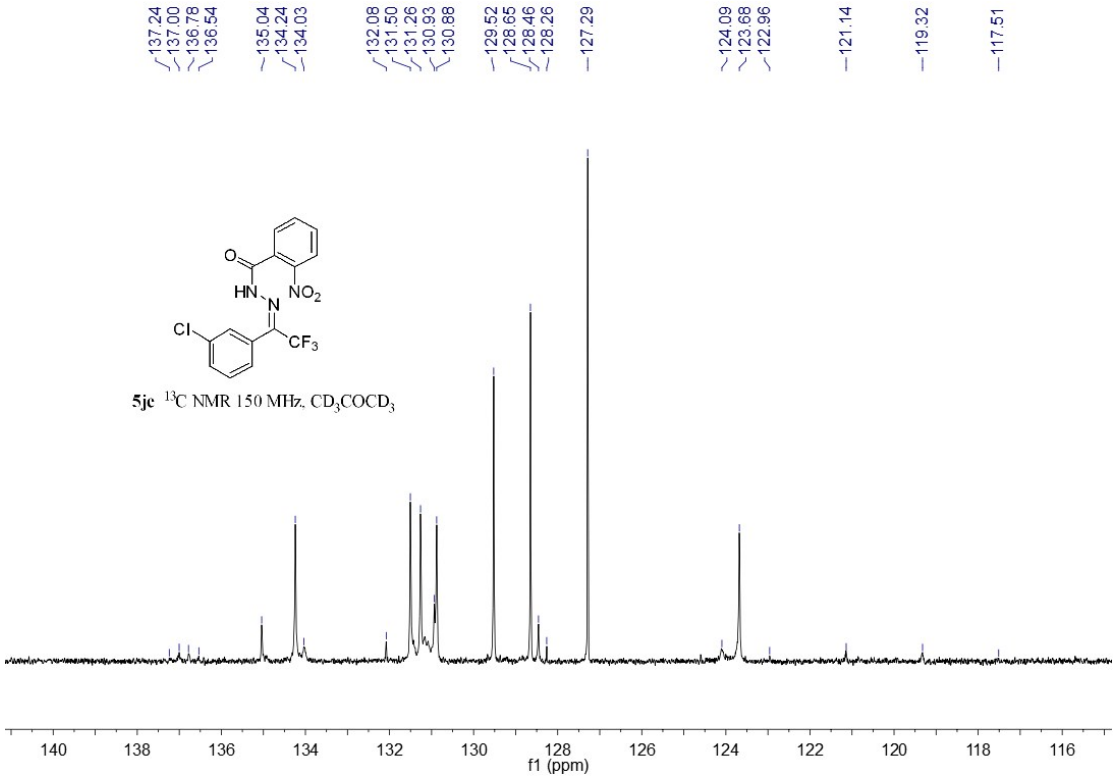
NMR copies of major product of compound **5je**:



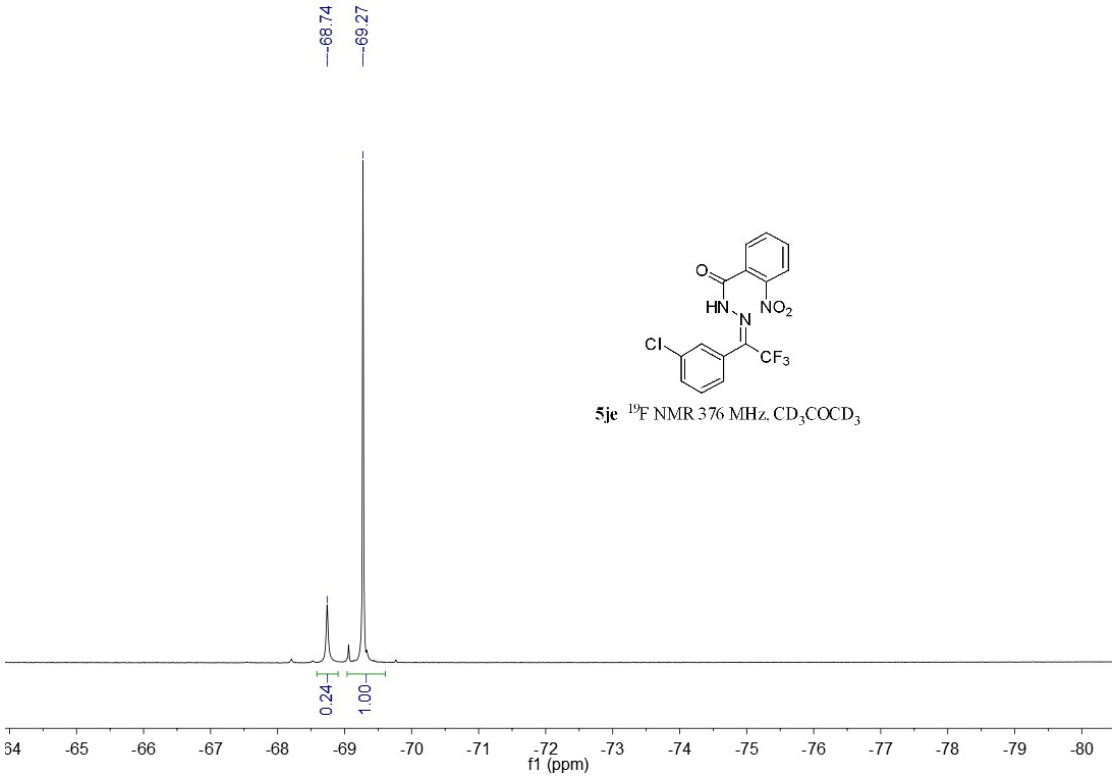
5je ^{1}H NMR 600 MHz, CD_3COCD_3



5je ^{13}C NMR 150 MHz, CD_3COCD_3

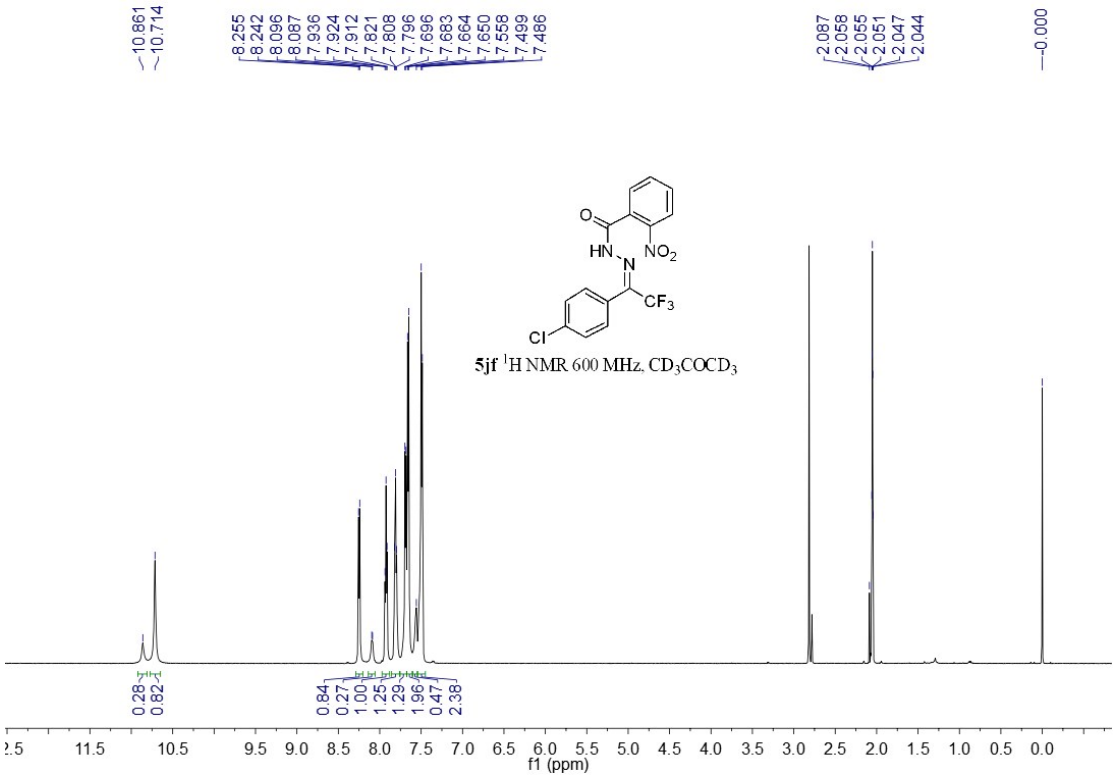


5je ^{13}C NMR 150 MHz, CD_3COCD_3

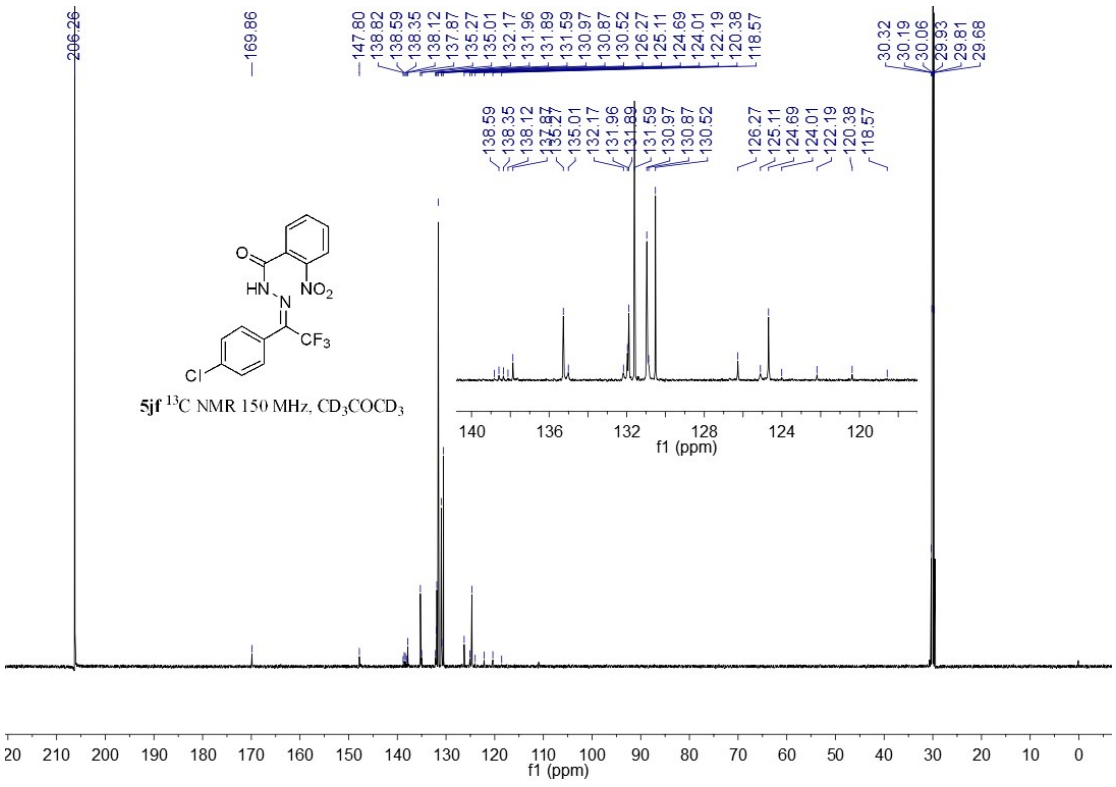


5je ^{19}F NMR 376 MHz, CD_3COCD_3

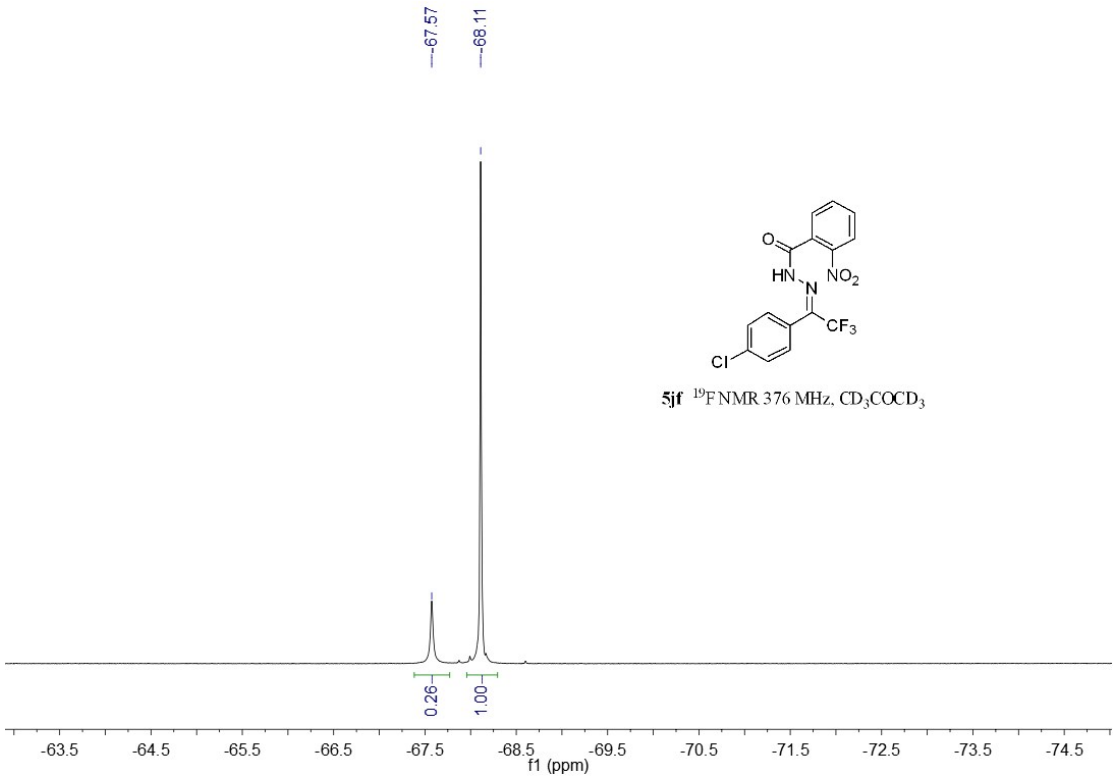
NMR copies of major product of compound **5jf**:



5jf ^{1}H NMR 600 MHz, CD_3COCD_3

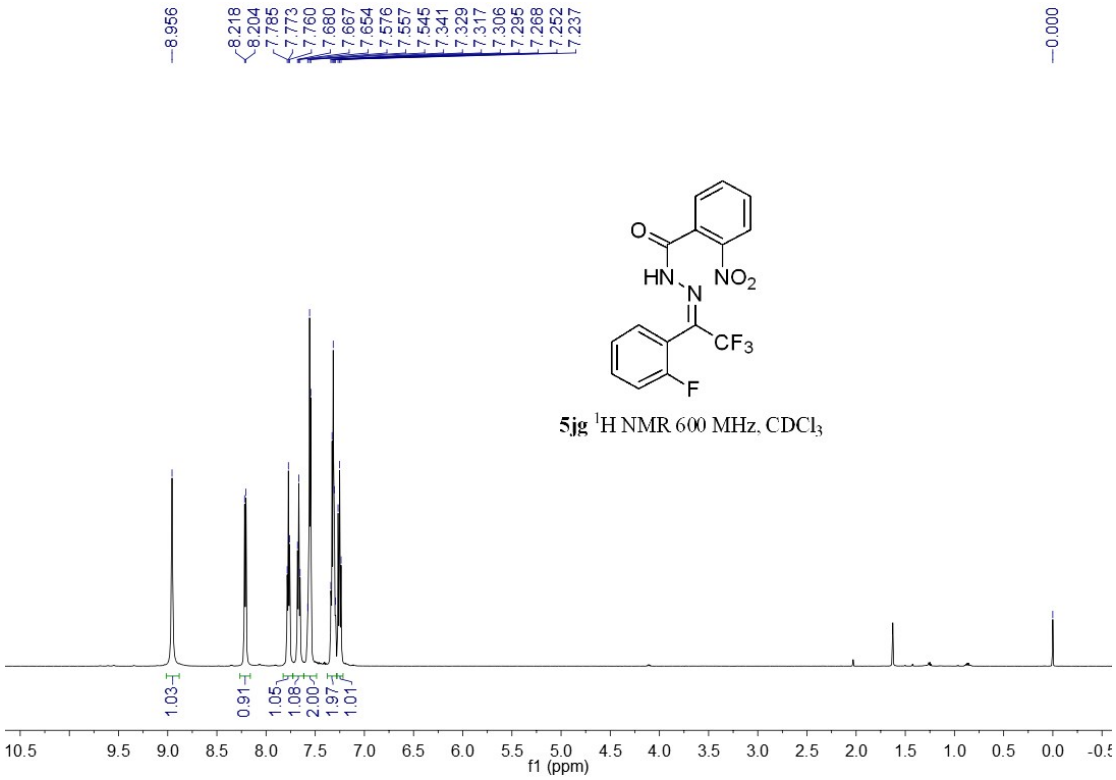


5jf ^{13}C NMR 150 MHz, CD_3COCD_3

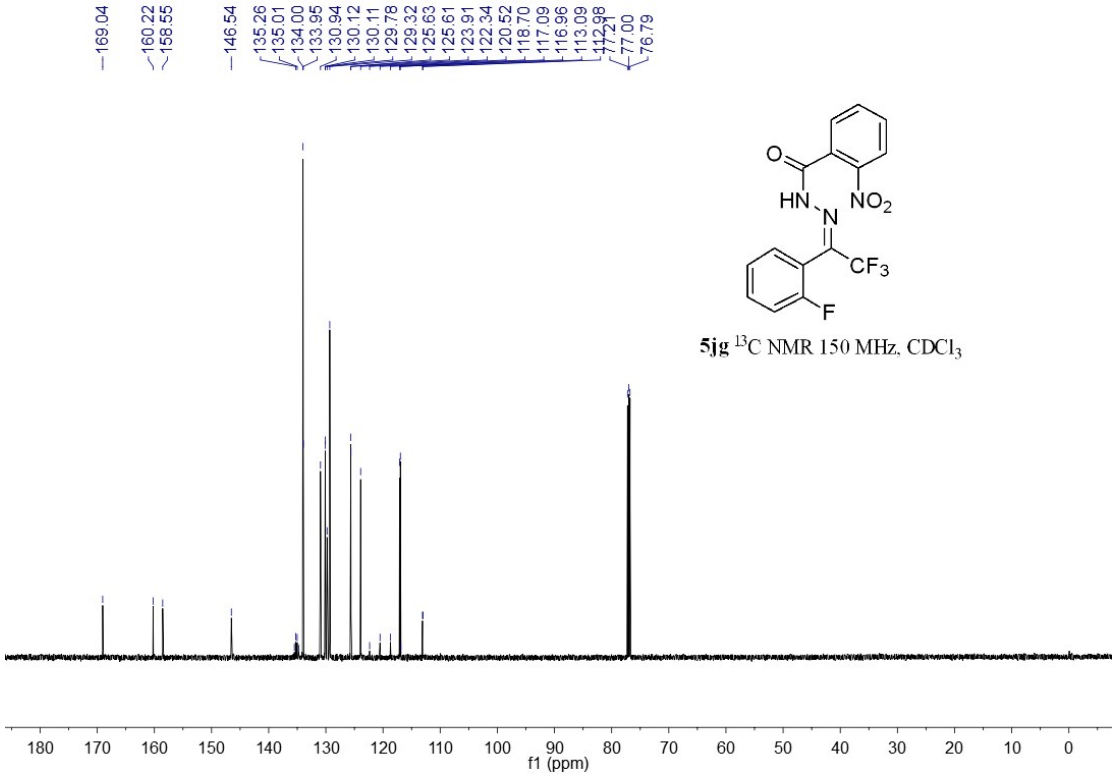


5jf ^{19}F NMR 376 MHz, CD_3COCD_3

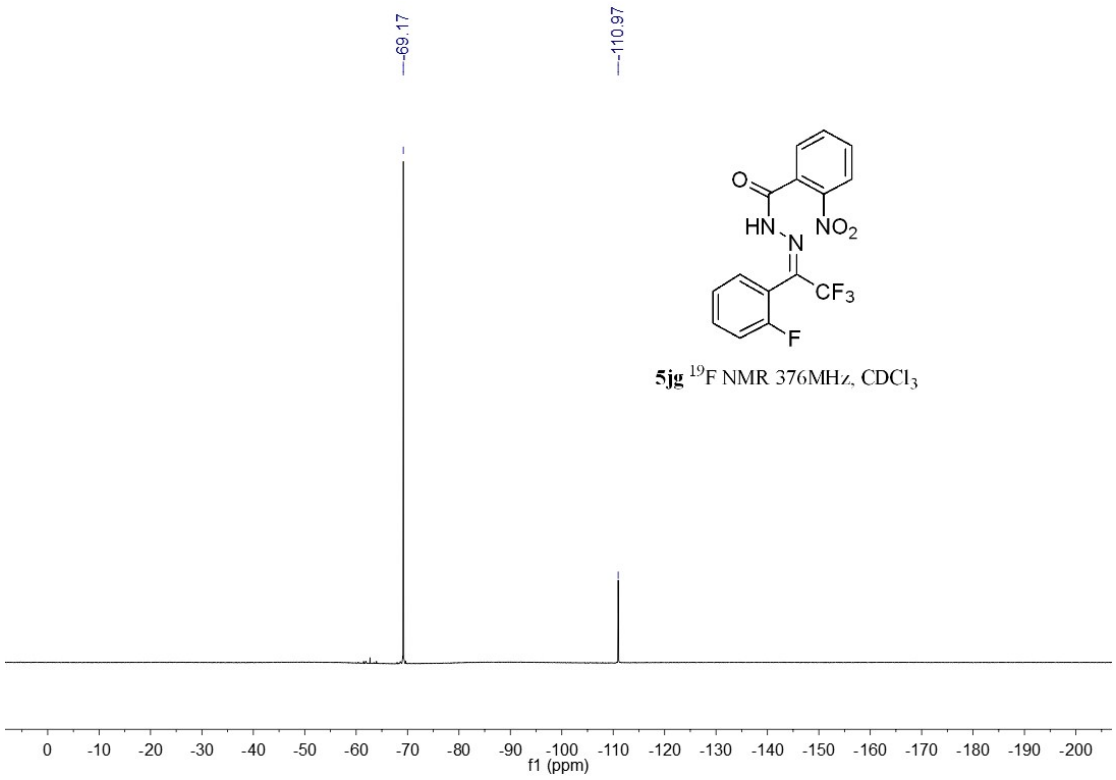
NMR copies of major product of compound **5jg**:



5jg ^{1}H NMR 600 MHz, $CDCl_3$

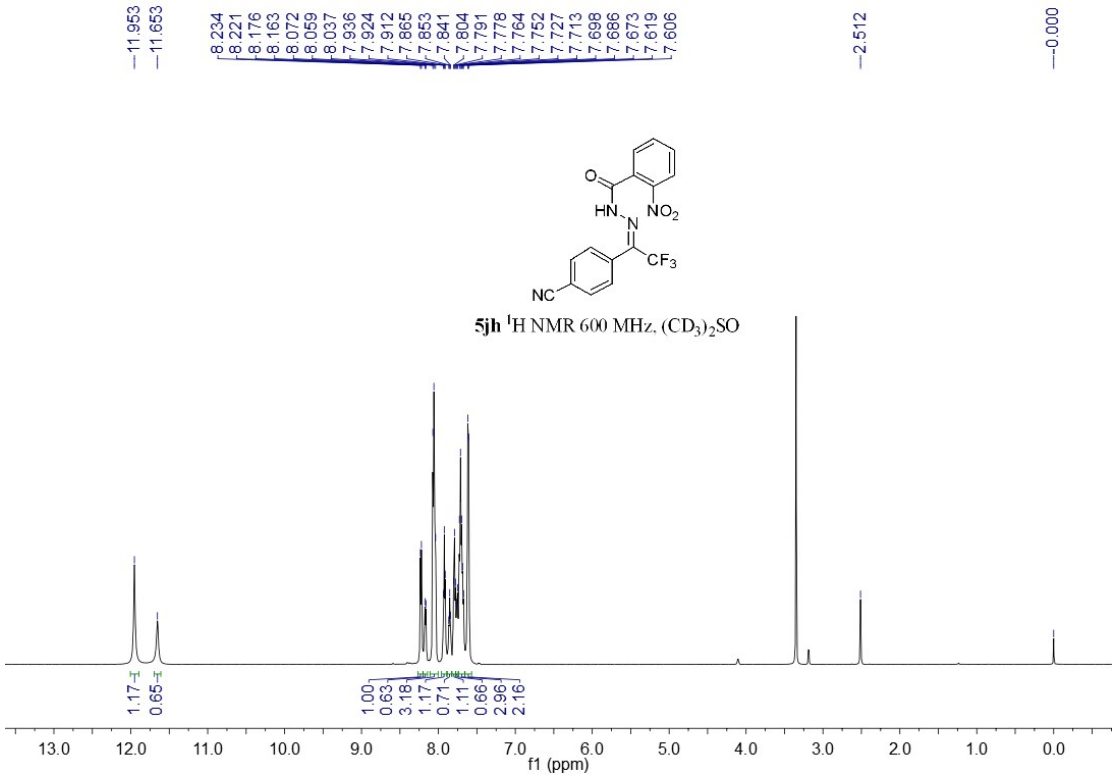


5jg ^{13}C NMR 150 MHz, $CDCl_3$

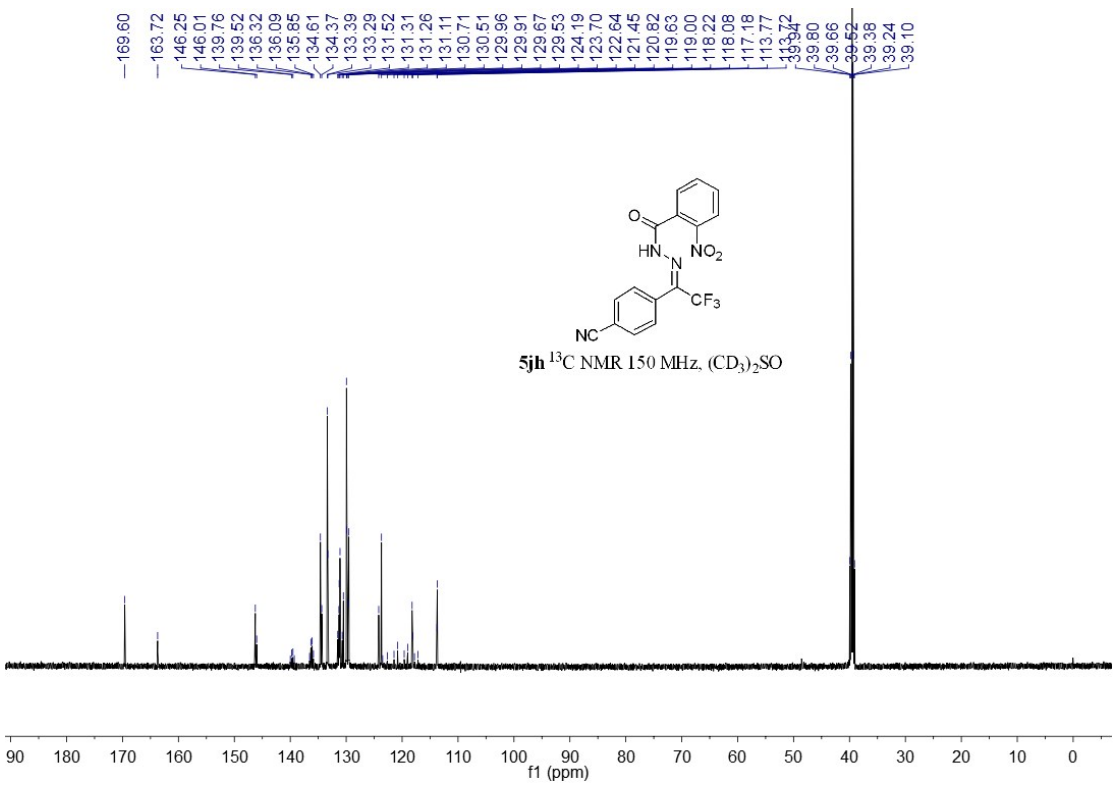


5jg ^{19}F NMR 376MHz, $CDCl_3$

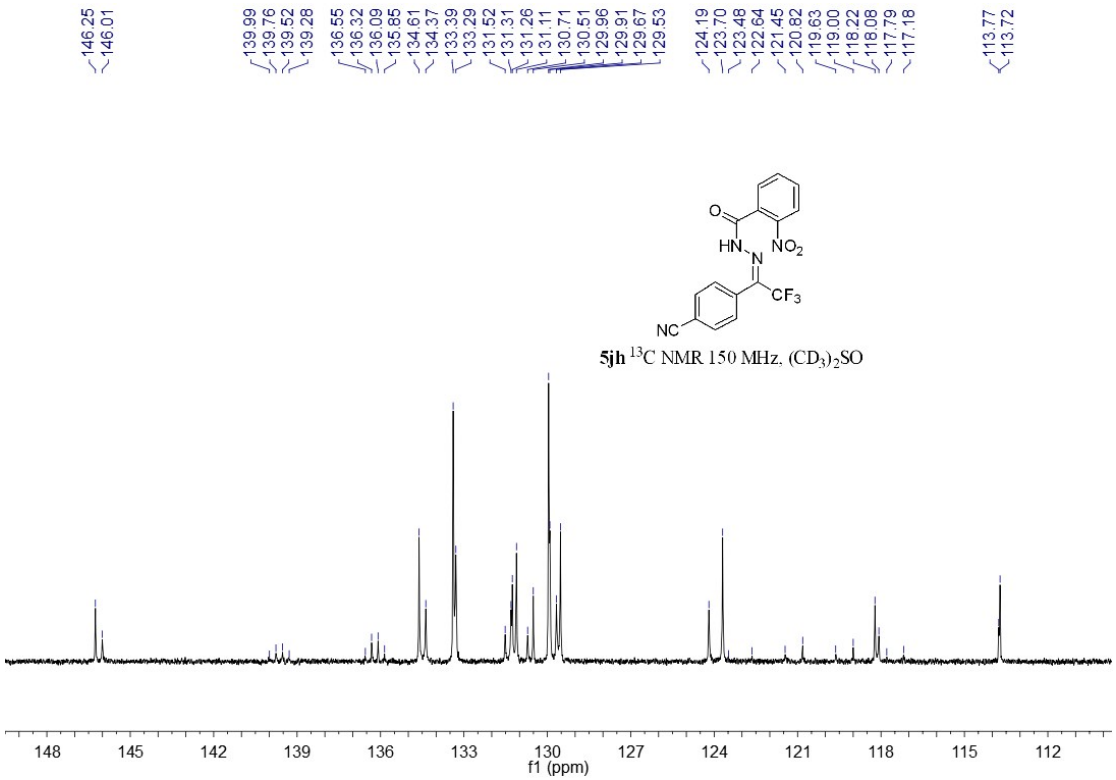
NMR copies of major product of compound **5jh**:



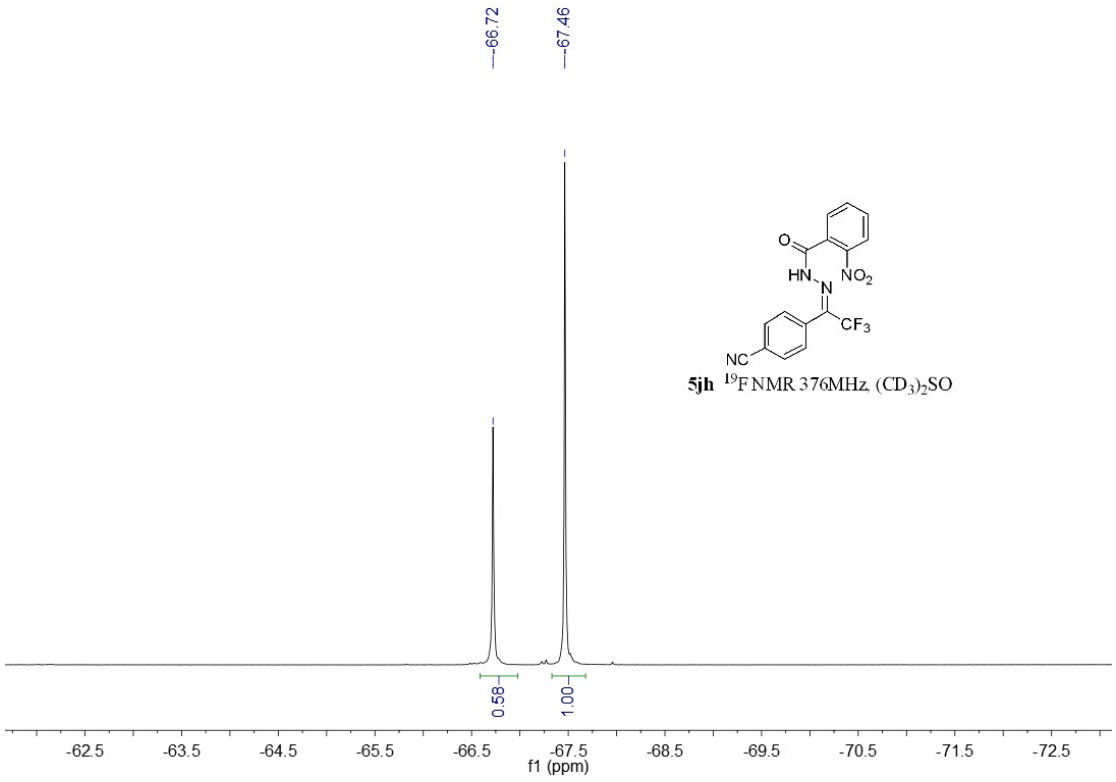
5jh ^{1}H NMR 600 MHz, $(CD_3)_2SO$



5jh ^{13}C NMR 150 MHz, $(CD_3)_2SO$

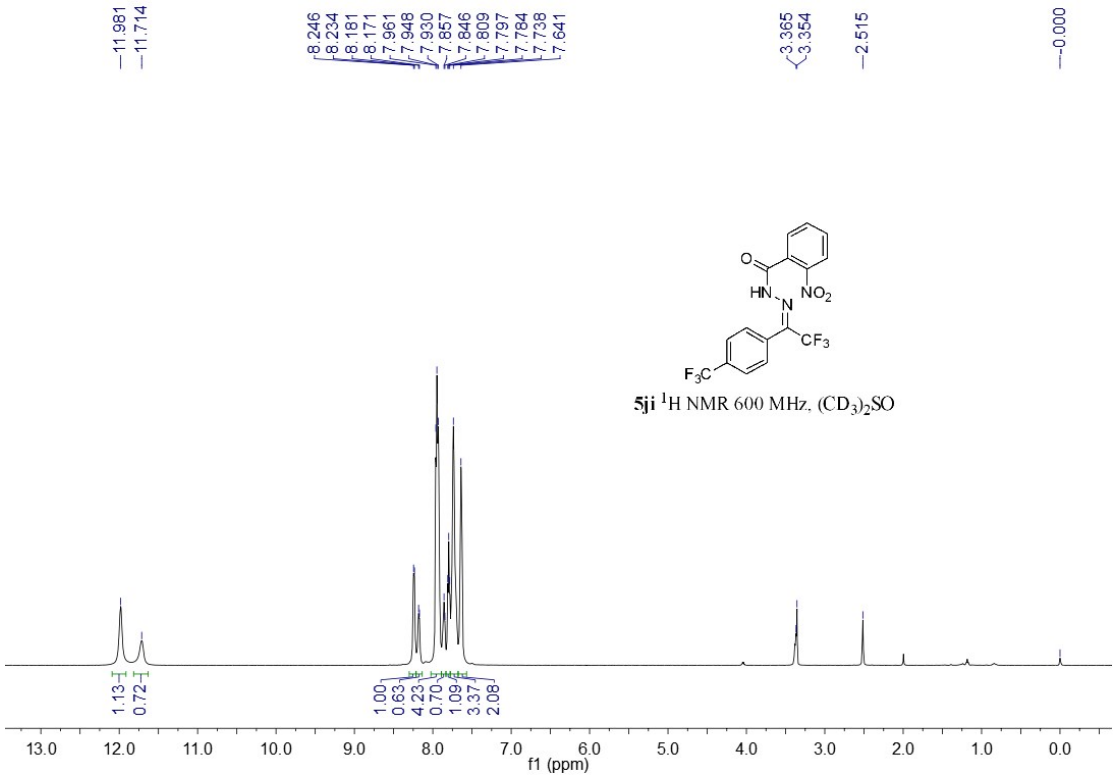


5jh ^{13}C NMR 150 MHz, $(CD_3)_2SO$

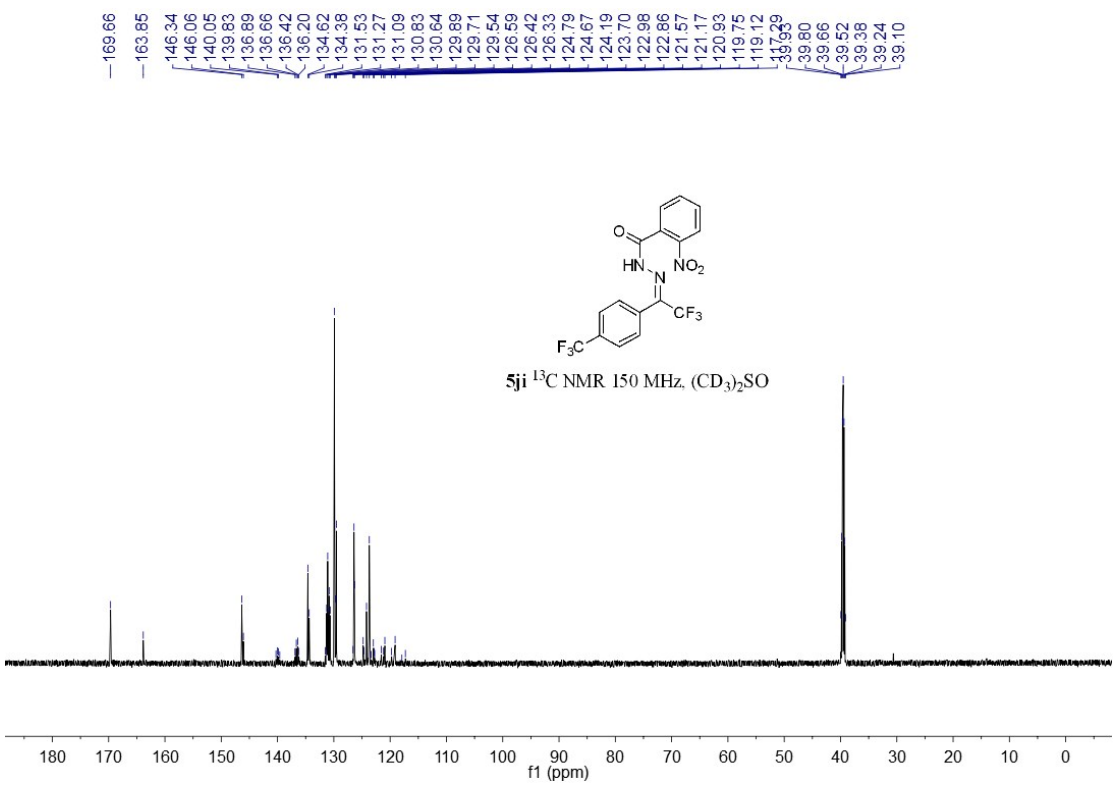


5jh ^{19}F NMR 376MHz, $(CD_3)_2SO$

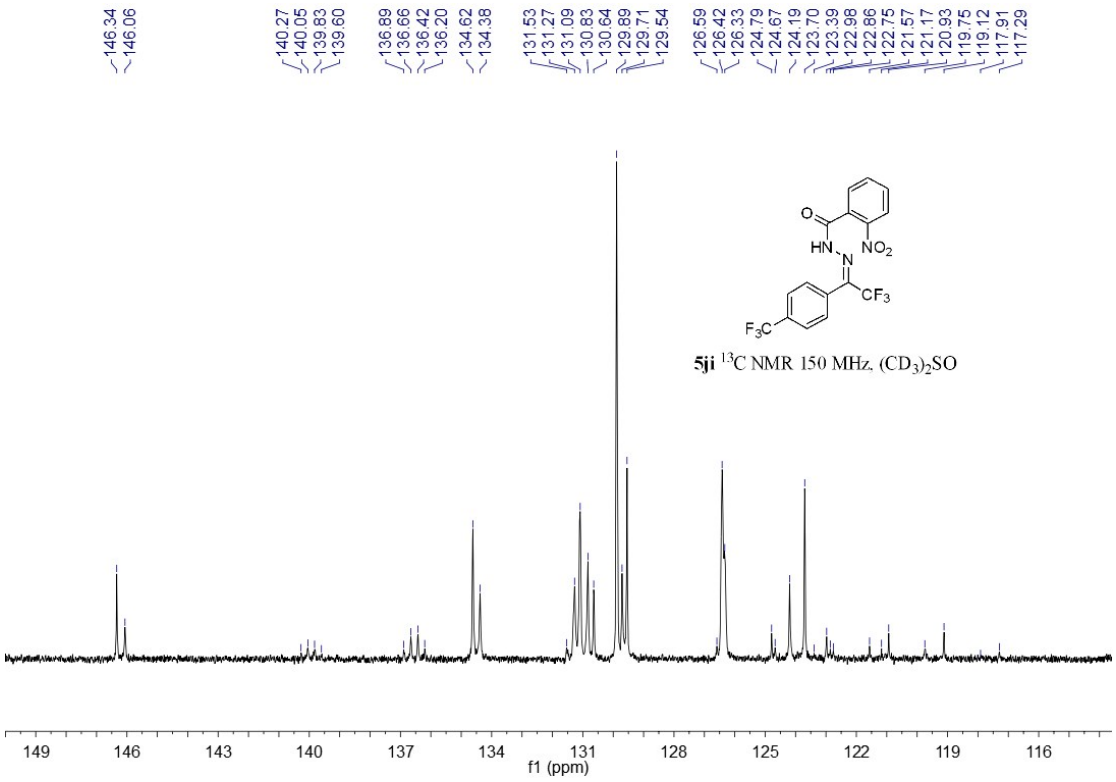
NMR copies of major product of compound **5ji**:



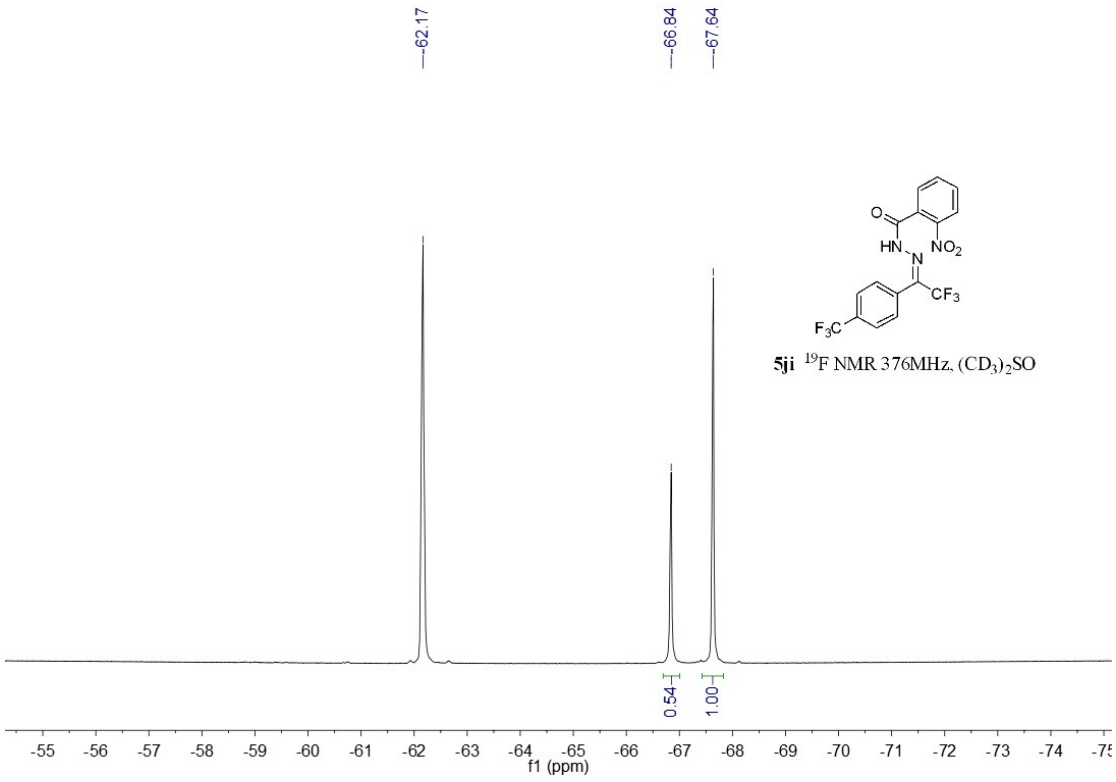
5ji ^{1}H NMR 600 MHz, $(CD_3)_2SO$



5ji ^{13}C NMR 150 MHz, $(CD_3)_2SO$

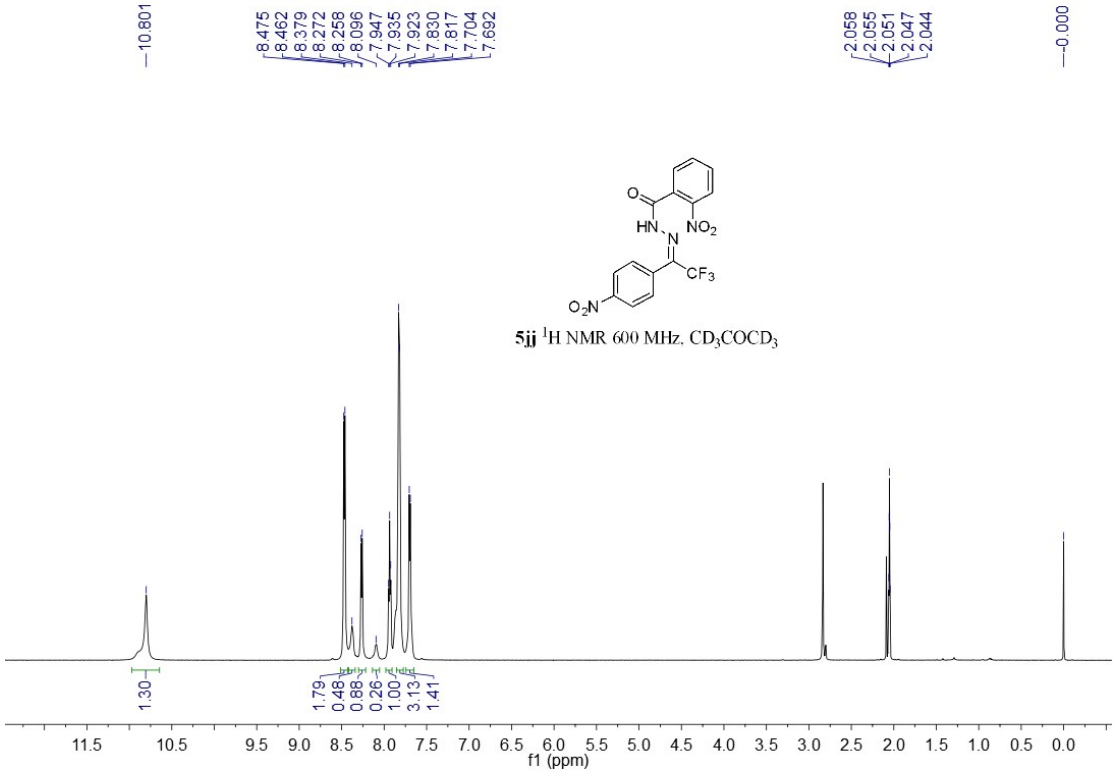


5ji ^{13}C NMR 150 MHz, $(CD_3)_2SO$

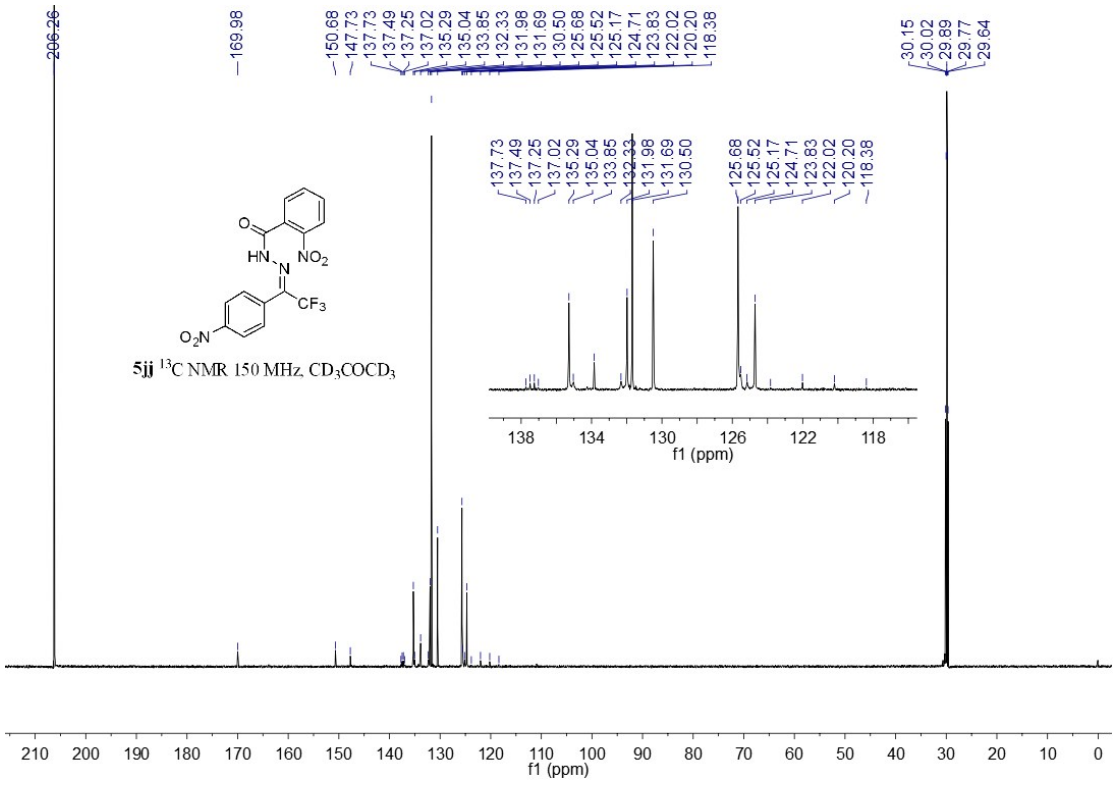


5ji ^{19}F NMR 376MHz, $(CD_3)_2SO$

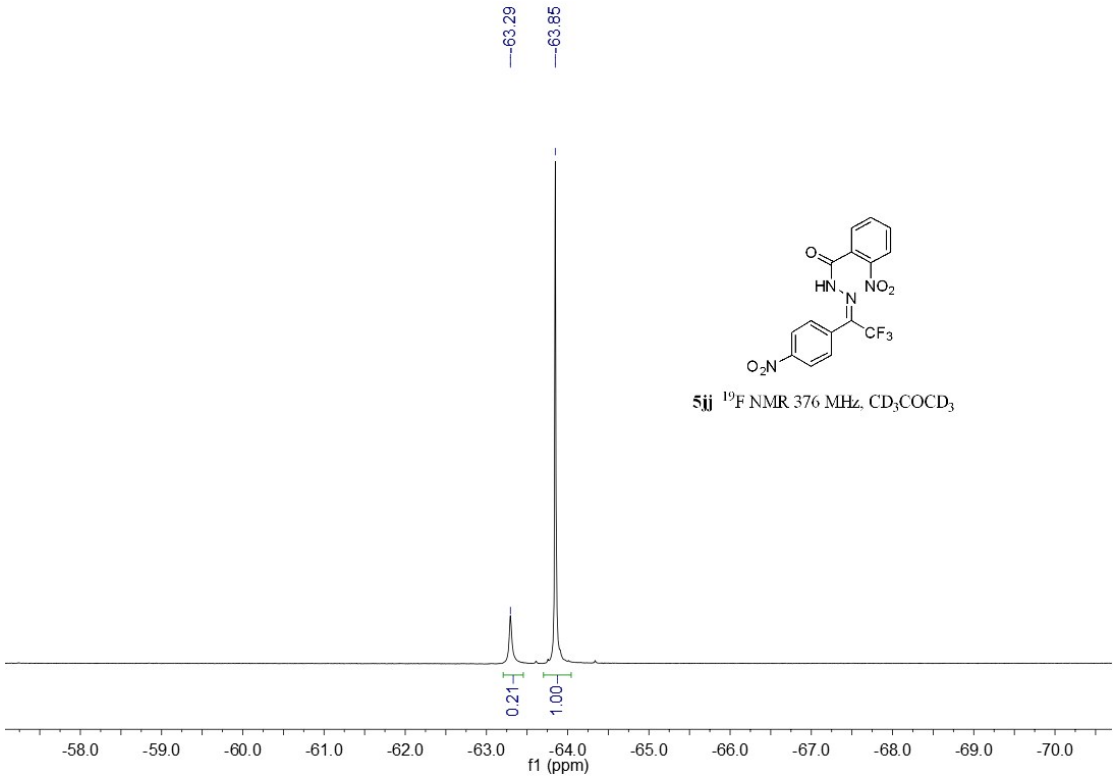
NMR copies of major product of compound **5jj**:



5jj ^{1}H NMR 600 MHz, CD_3COCD_3

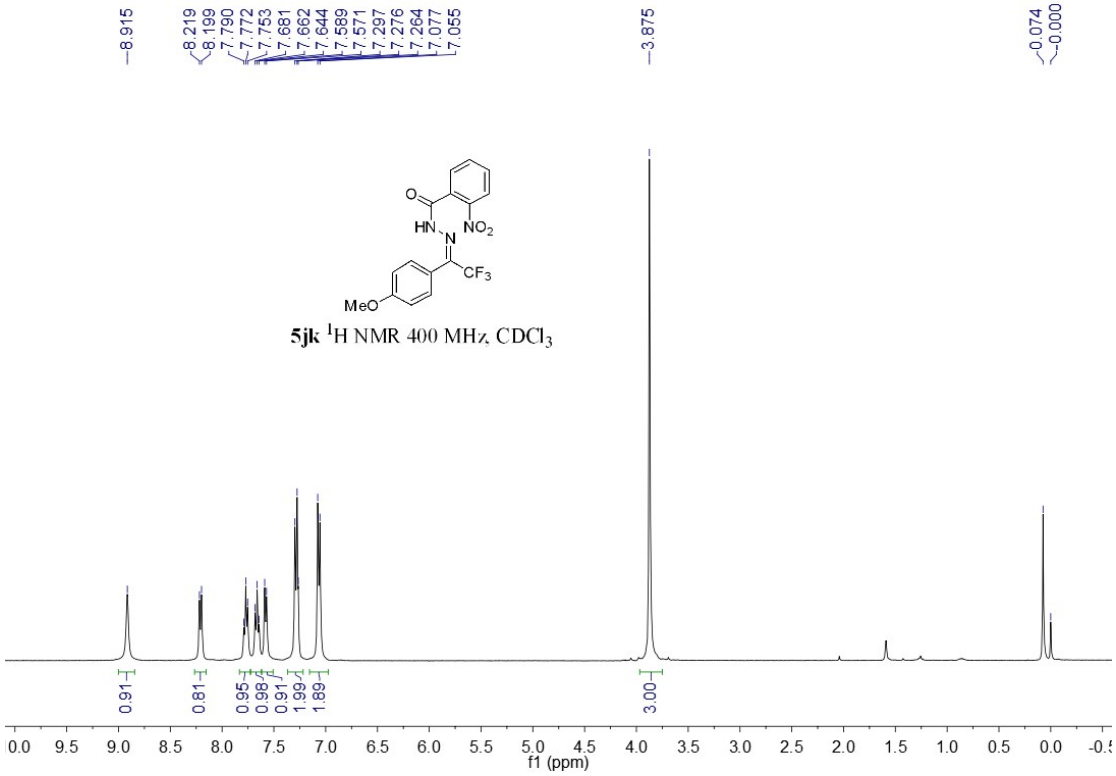


5jj ^{13}C NMR 150 MHz, CD_3COCD_3

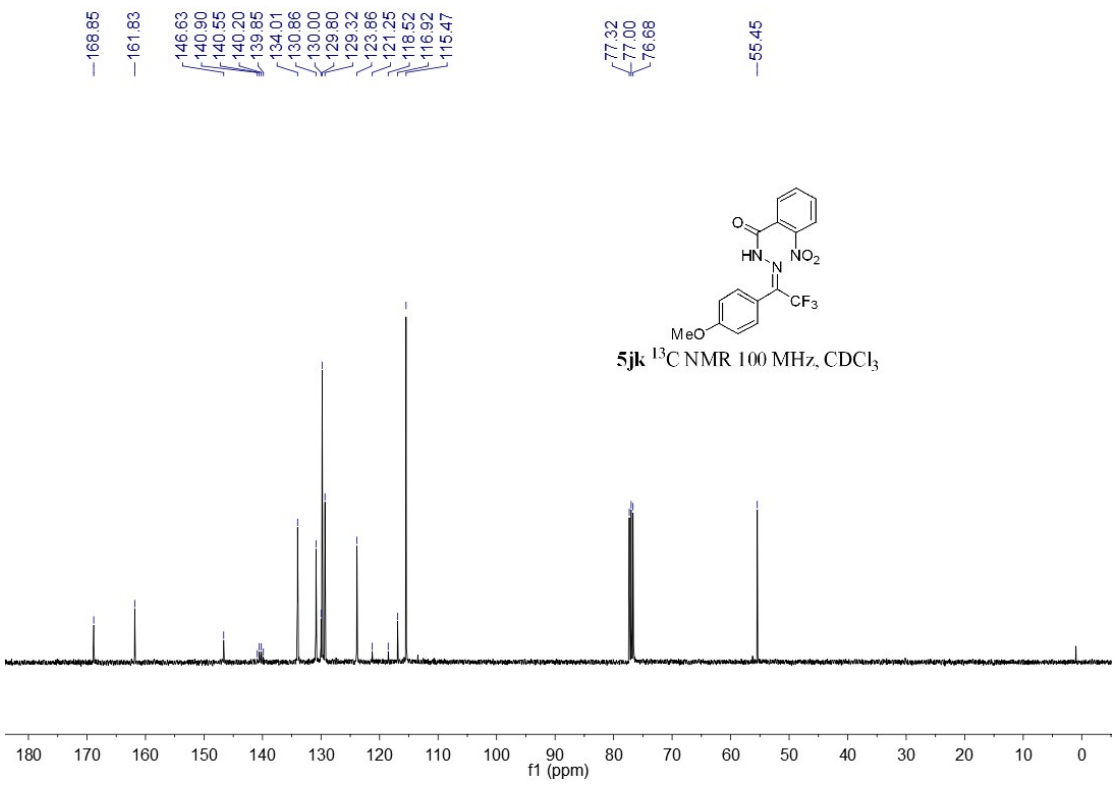


5ij ^{19}F NMR 376 MHz, CD_3COCD_3

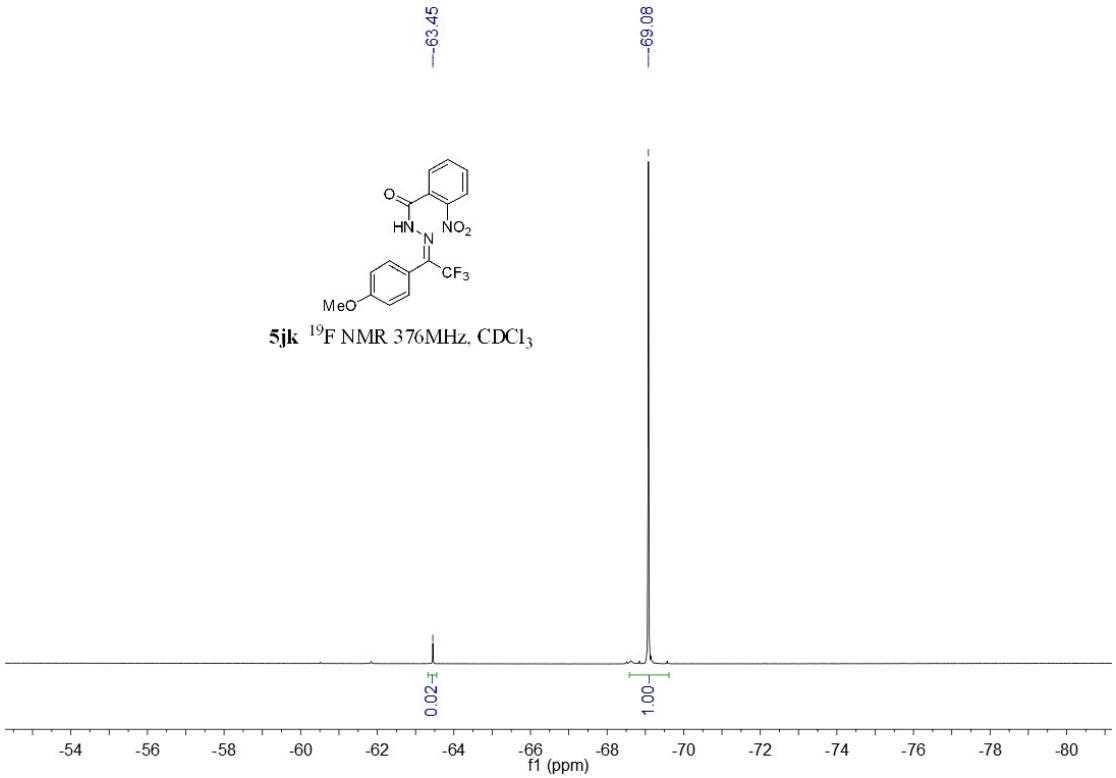
NMR copies of major product of compound **5jk**:



5jk ^{1}H NMR 400 MHz, $CDCl_3$

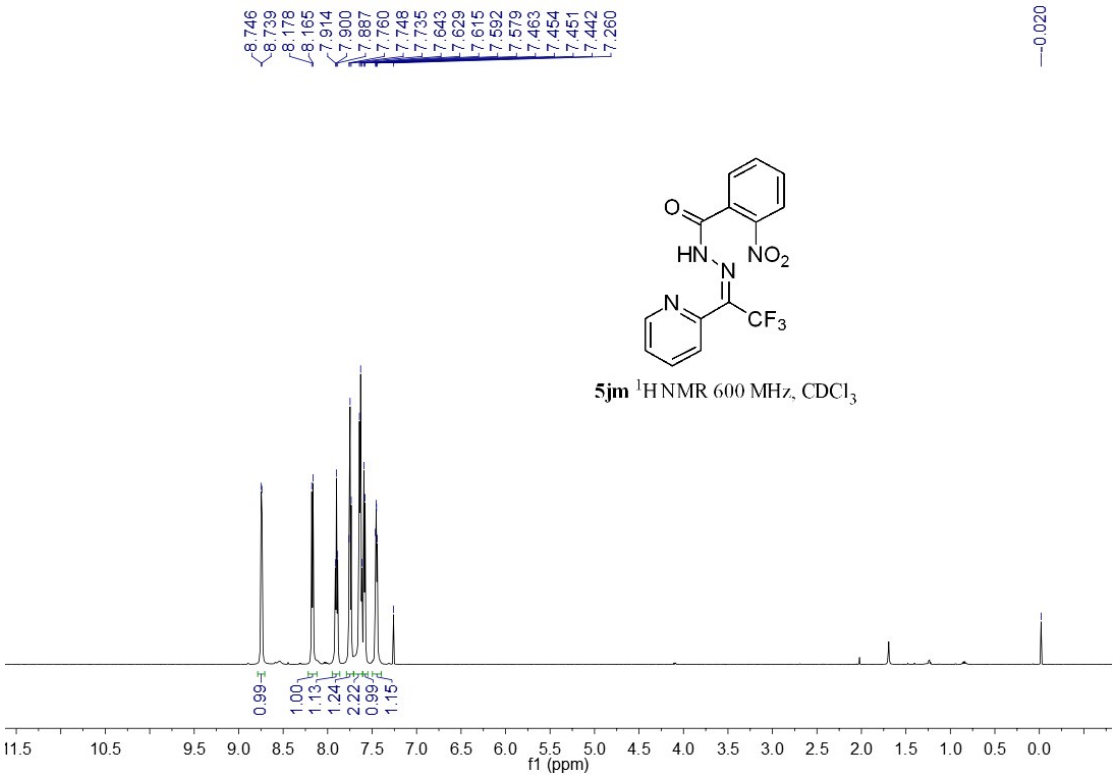


5jk ^{13}C NMR 100 MHz, $CDCl_3$

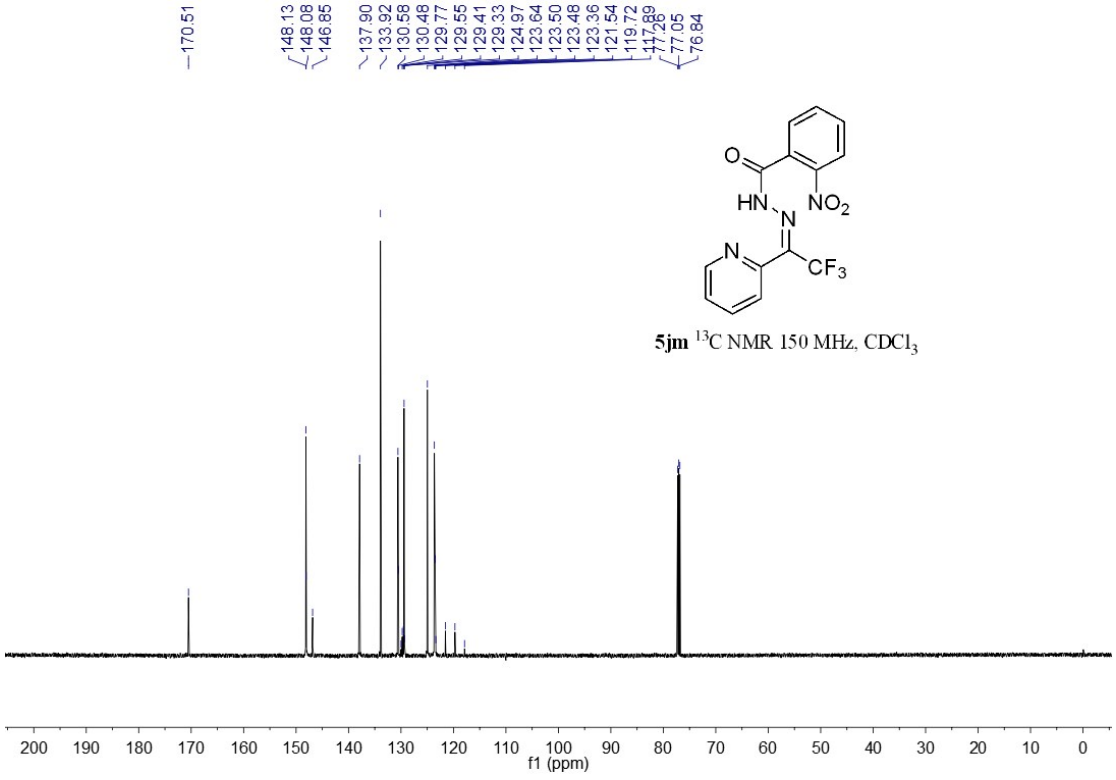


5jk ^{19}F NMR 376MHz, $CDCl_3$

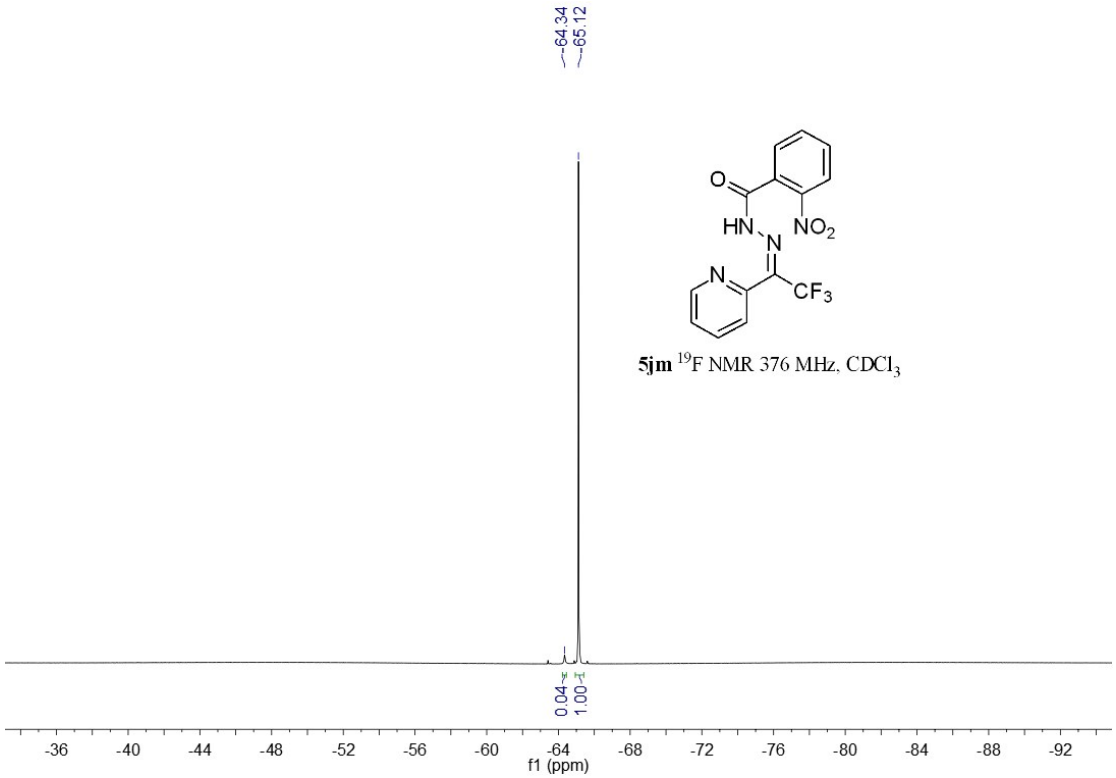
NMR copies of major product of compound **5jm**:



5jm ^{1}H NMR 600 MHz, $CDCl_3$

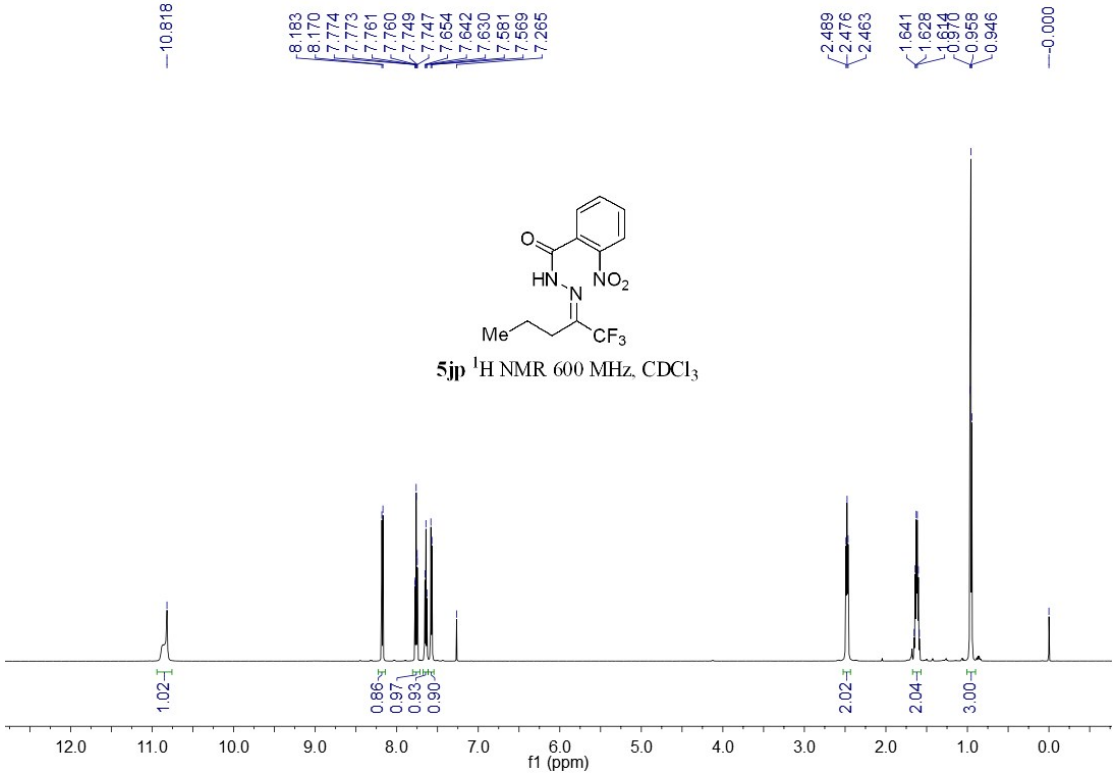


5jm ^{13}C NMR 150 MHz, $CDCl_3$

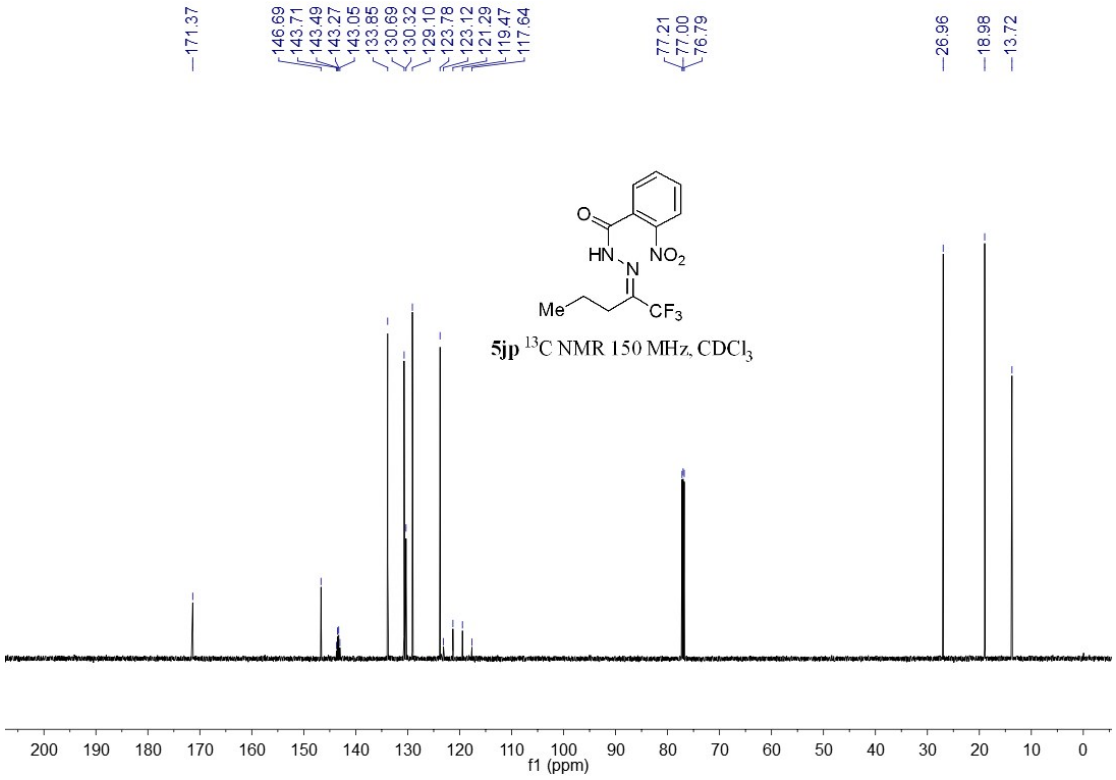


5jm ^{19}F NMR 376 MHz, $CDCl_3$

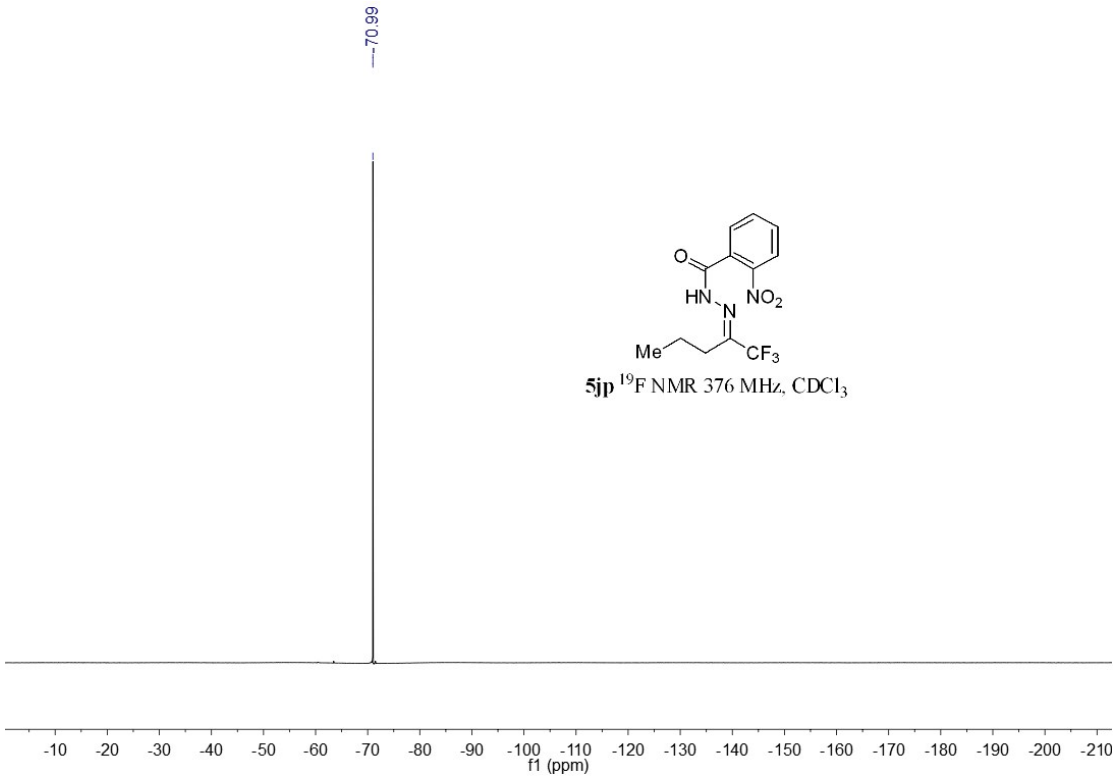
NMR copies of major product of compound **5jp**:



5jp ^{1}H NMR 600 MHz, $CDCl_3$

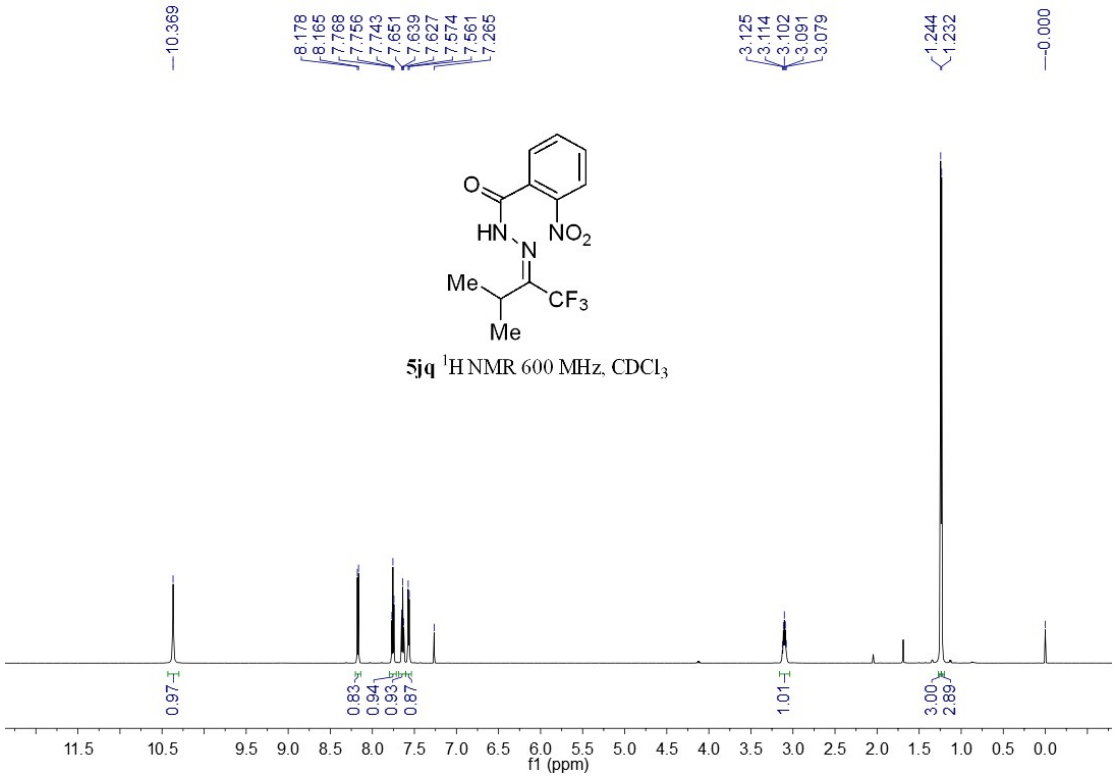


5jp ^{13}C NMR 150 MHz, $CDCl_3$

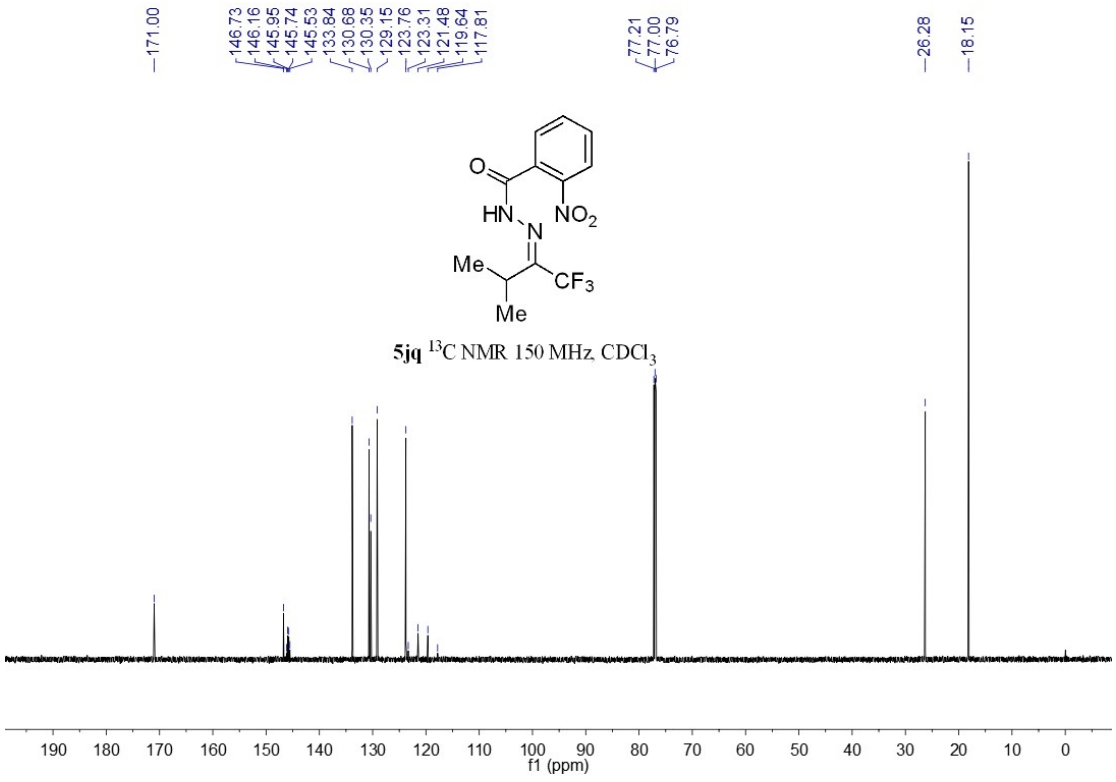


5jp ^{19}F NMR 376 MHz, $CDCl_3$

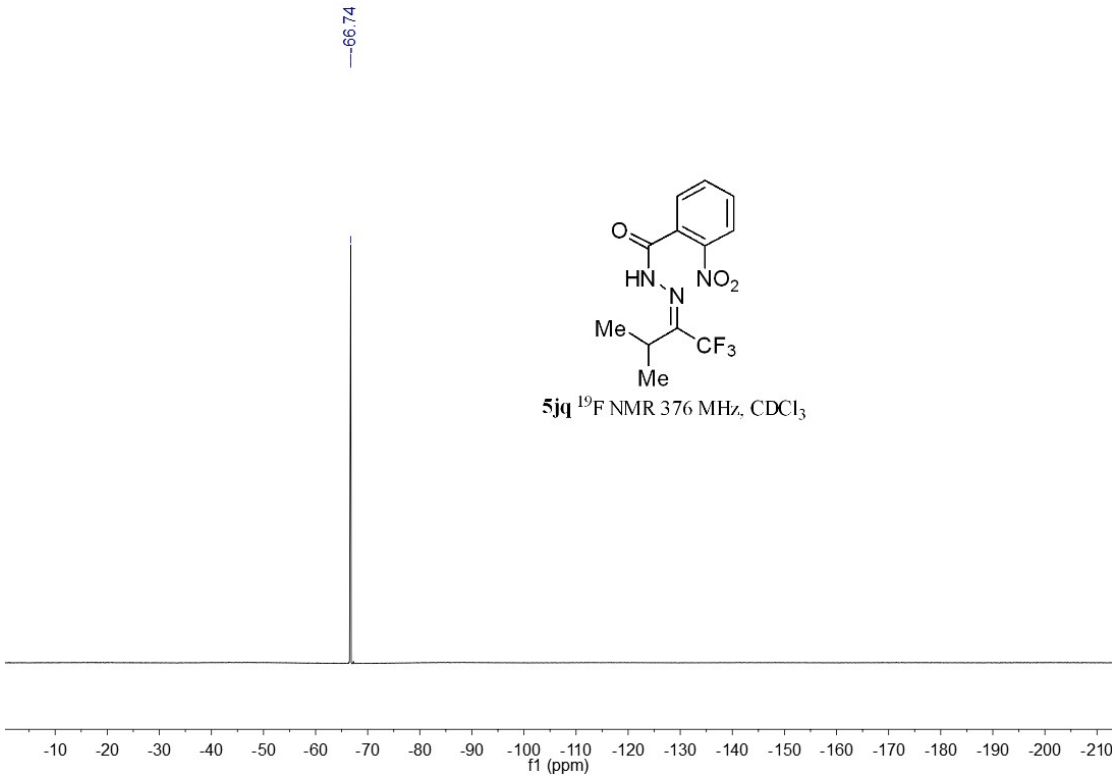
NMR copies of major product of compound **5jq**:



5jq ^{1}H NMR 600 MHz, $CDCl_3$

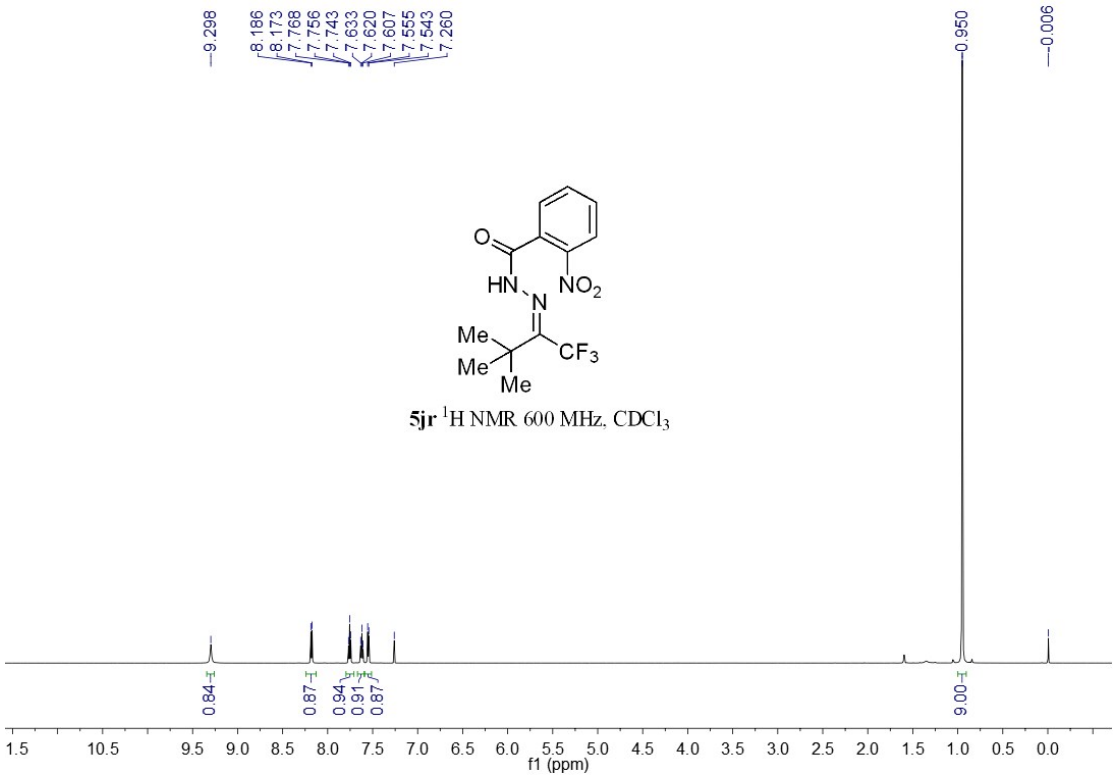


5jq ^{13}C NMR 150 MHz, $CDCl_3$

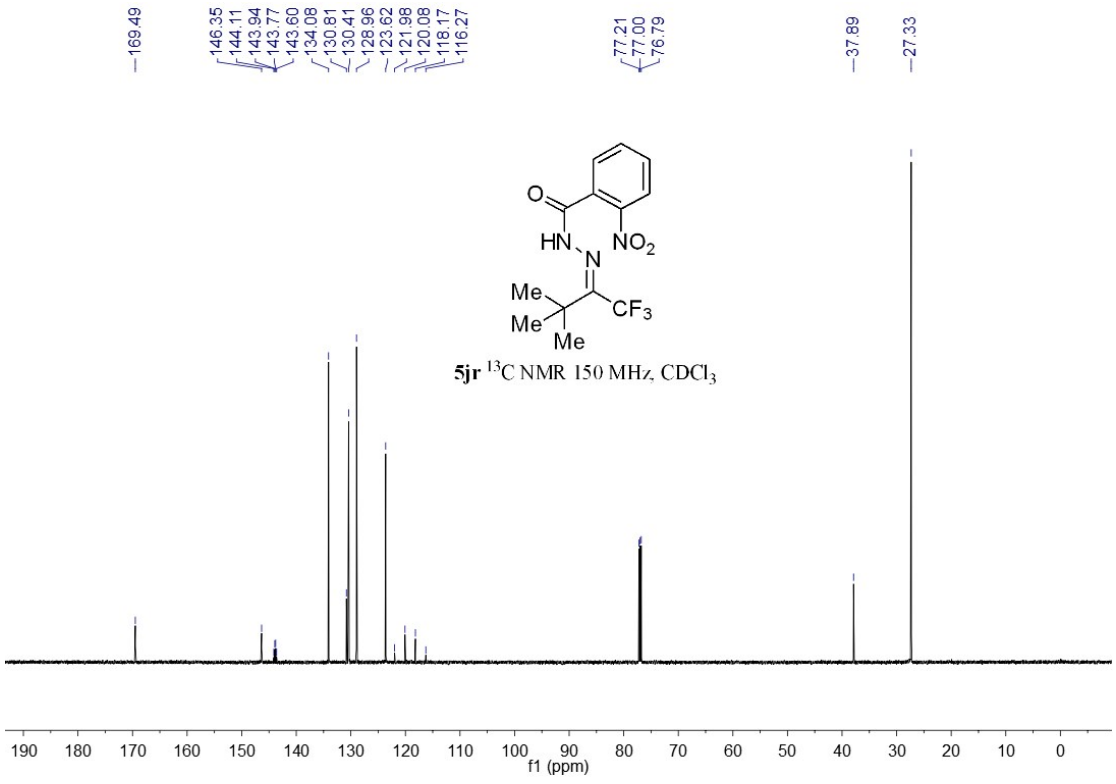


5jq ^{19}F NMR 376 MHz, $CDCl_3$

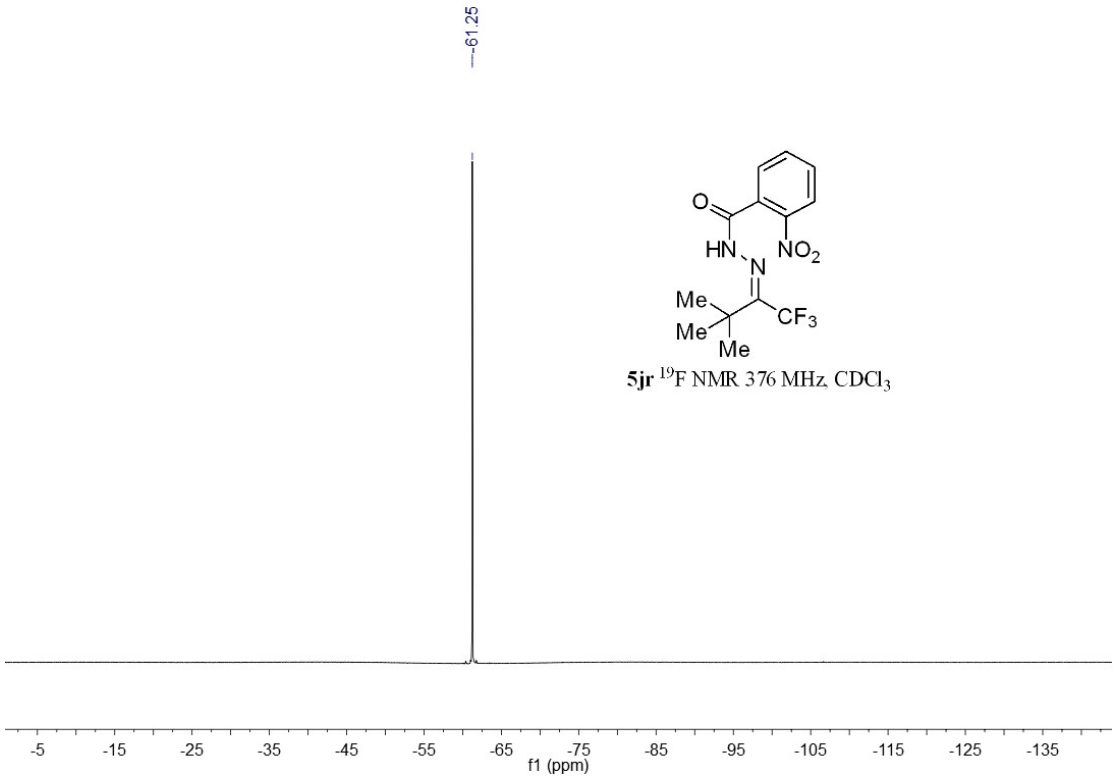
NMR copies of major product of compound **5jr**:



5jr ^{1}H NMR 600 MHz, $CDCl_3$

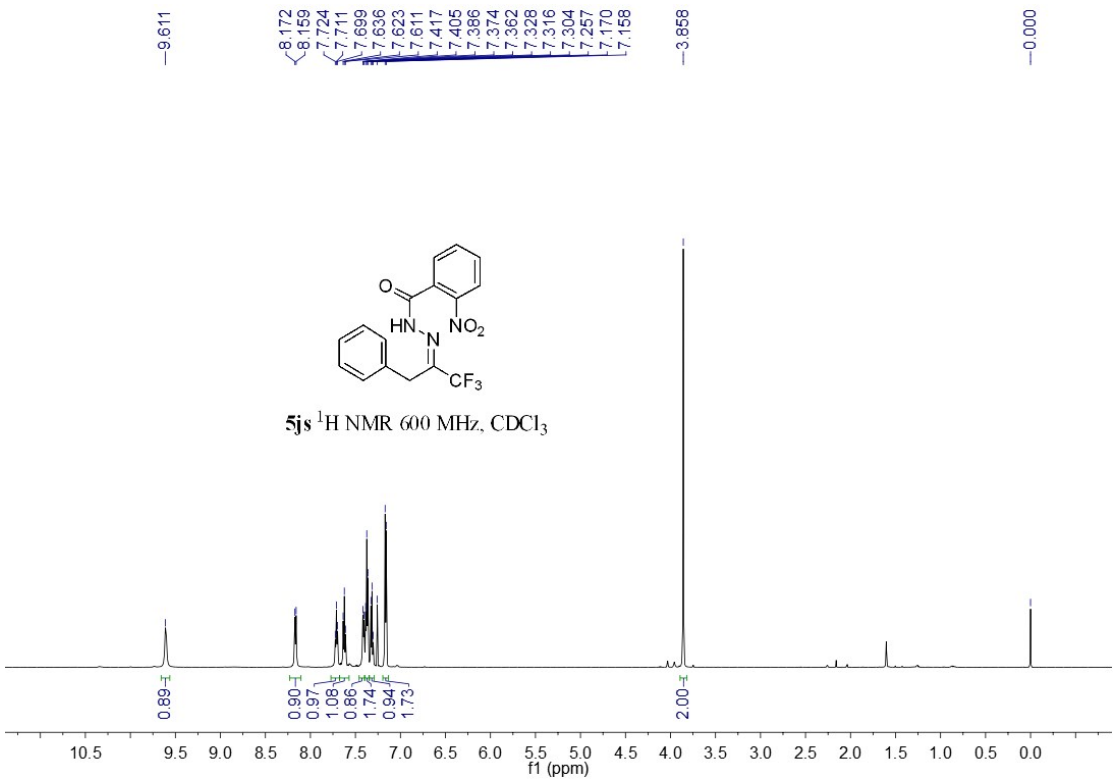


5jr ^{13}C NMR 150 MHz, $CDCl_3$

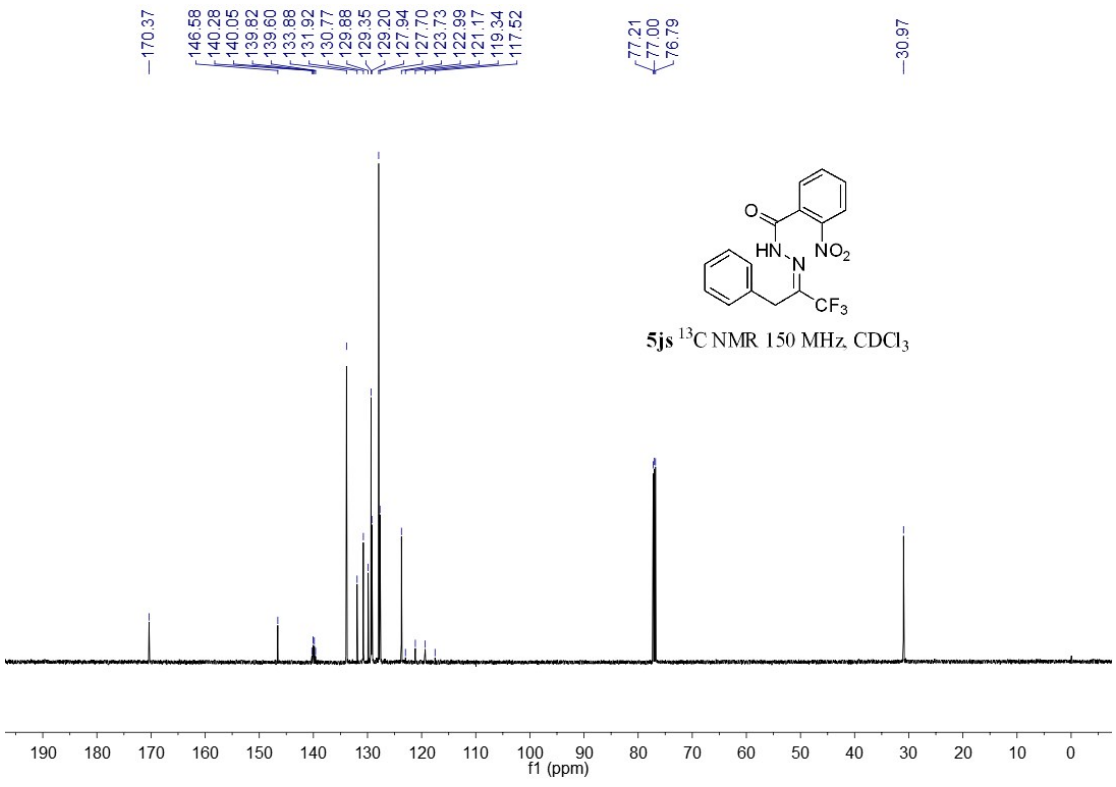


5jr ^{19}F NMR 376 MHz, $CDCl_3$

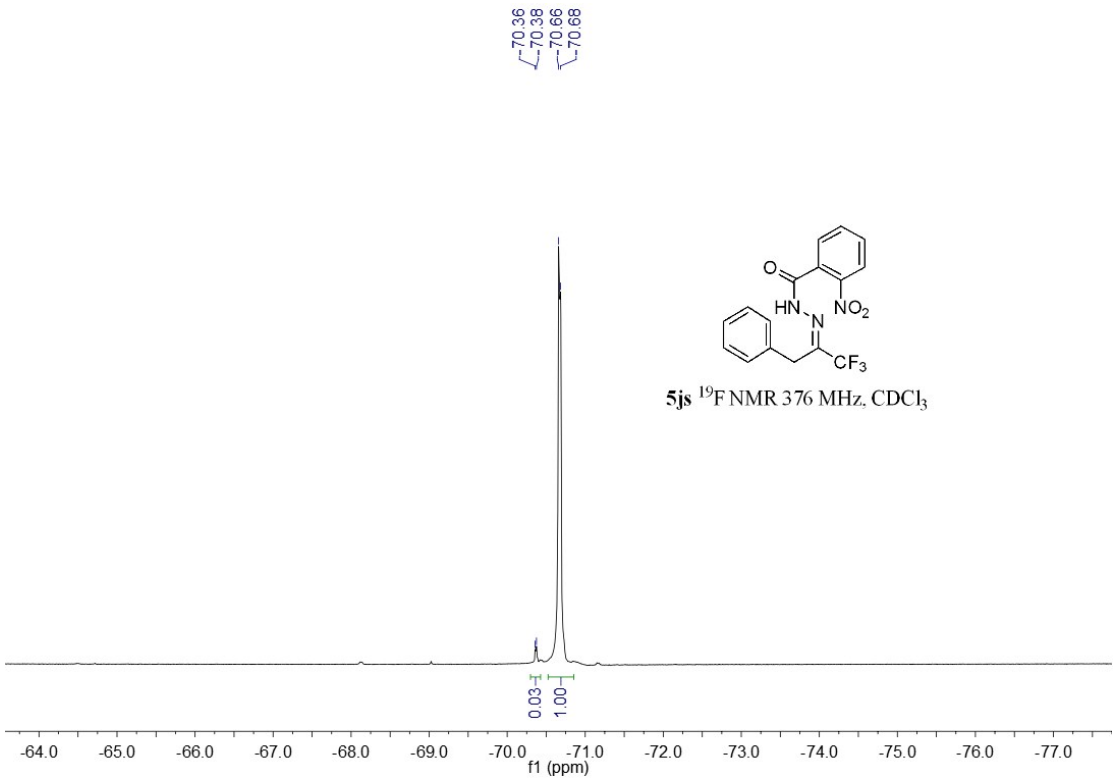
NMR copies of major product of compound **5js**:



5js ^{1}H NMR 600 MHz, $CDCl_3$

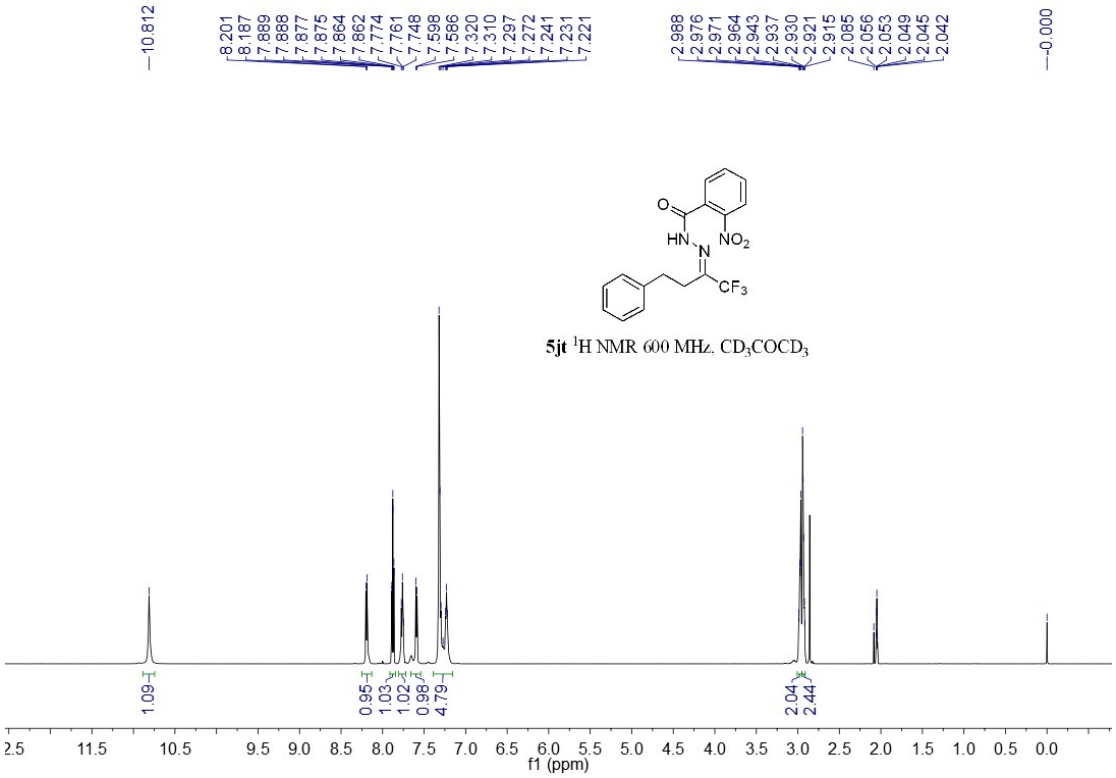


5js ^{13}C NMR 150 MHz, $CDCl_3$

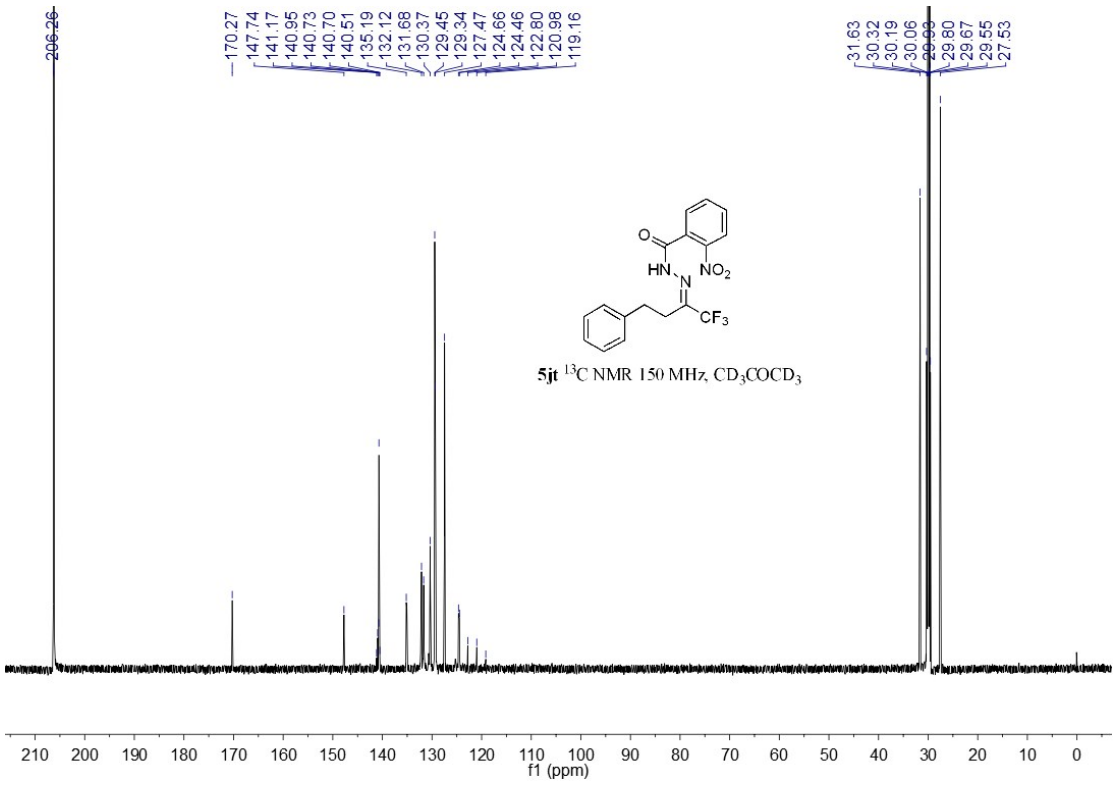


5js ^{19}F NMR 376 MHz, $CDCl_3$

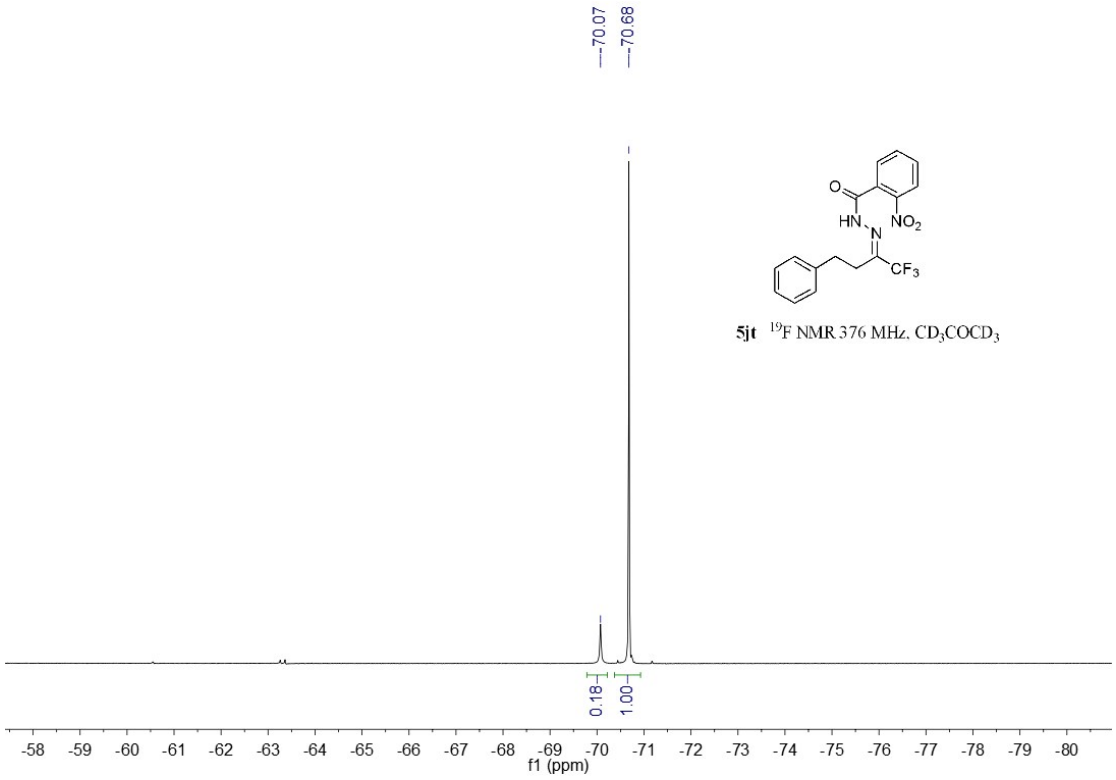
NMR copies of major product of compound **5jt**:



5jt ^{1}H NMR 600 MHz, CD_3COCD_3

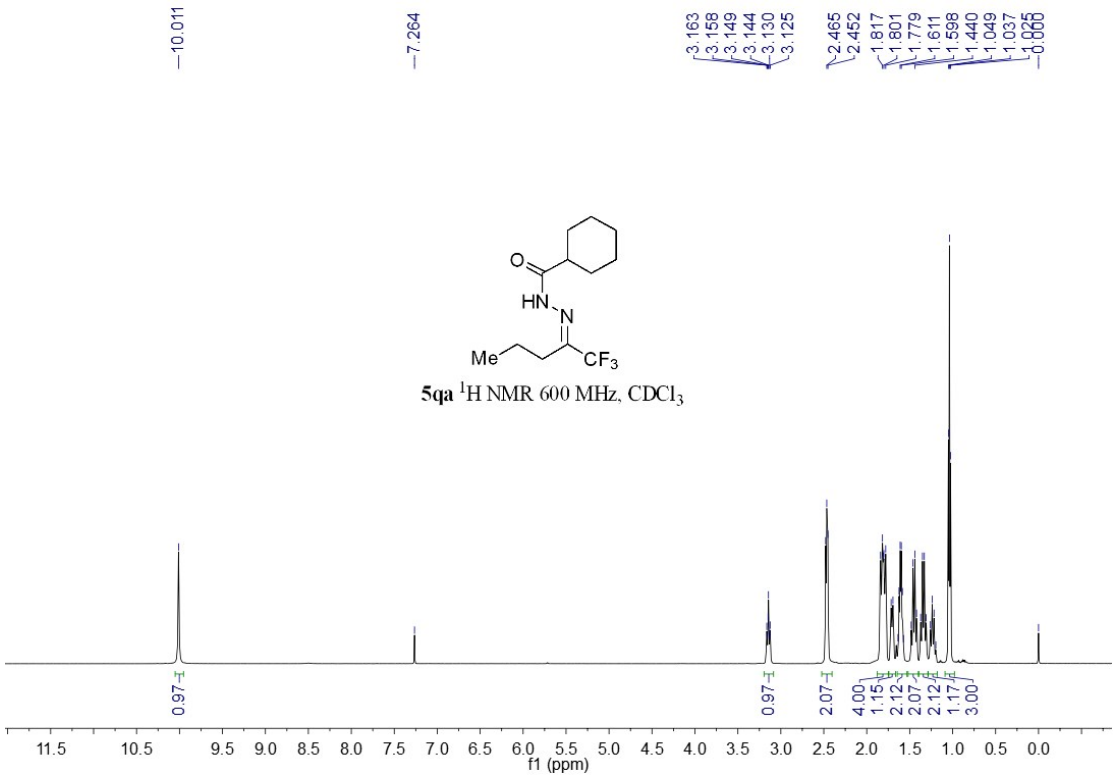


5jt ^{13}C NMR 150 MHz, CD_3COCD_3

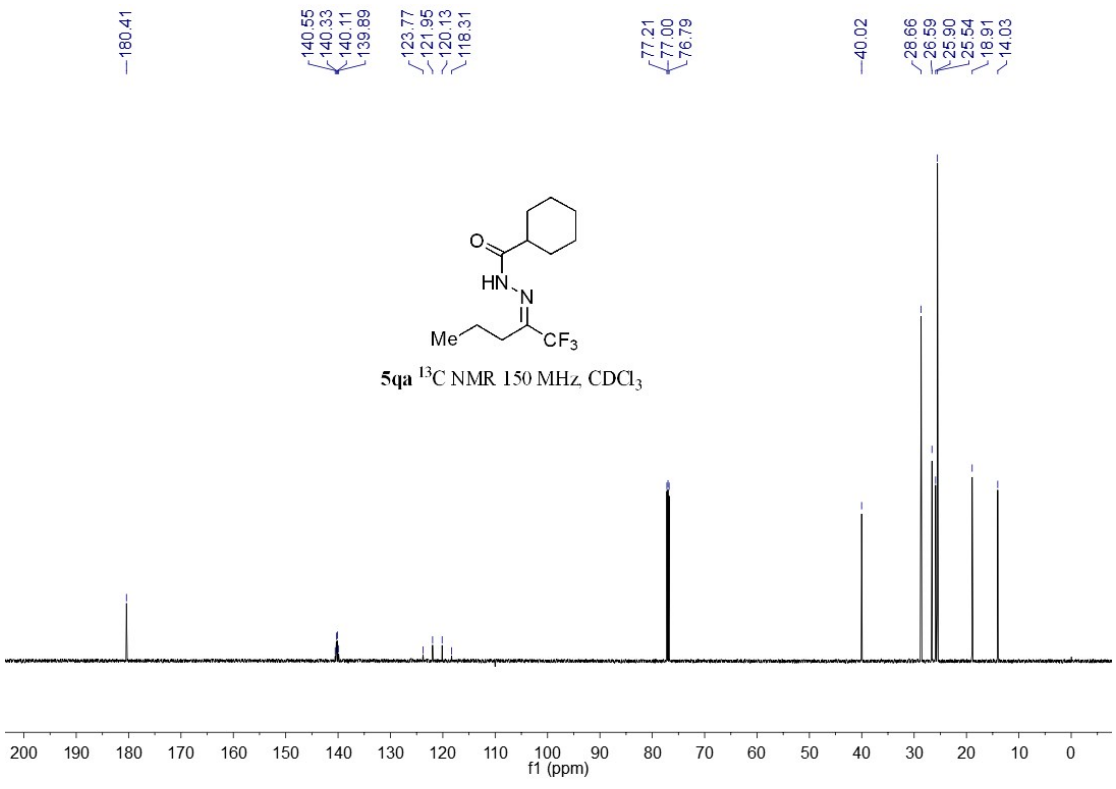


5jt ^{19}F NMR 376 MHz, CD_3COCD_3

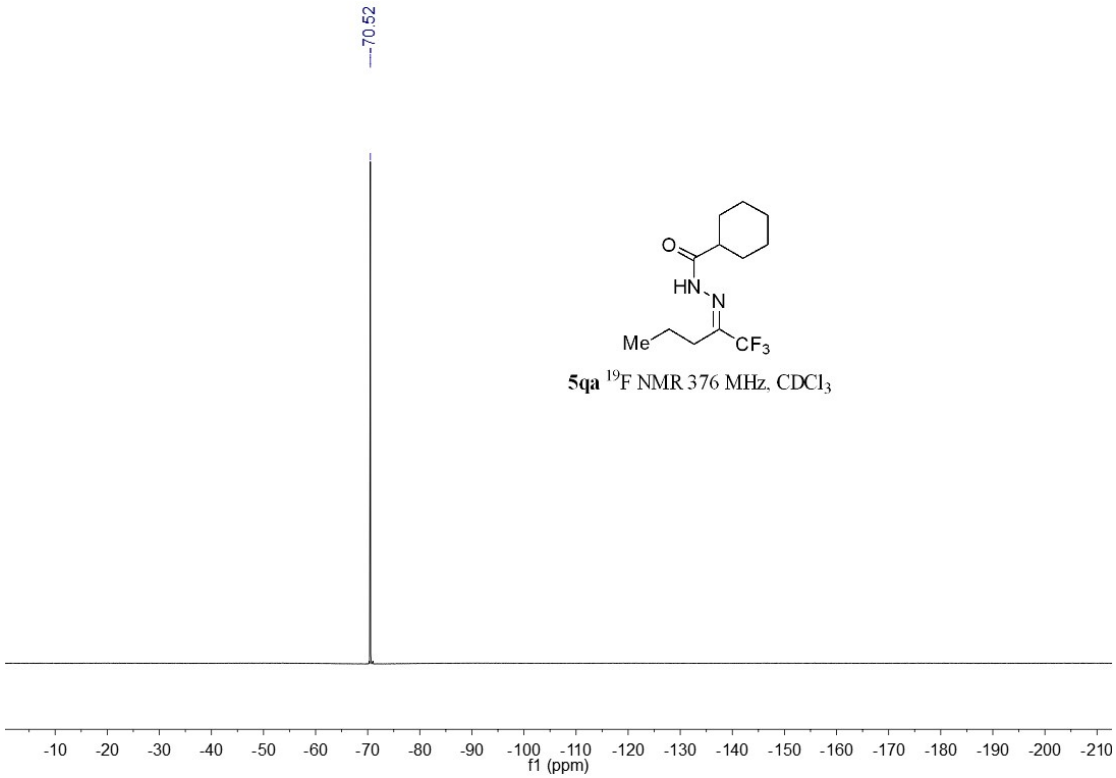
NMR copies of major product of compound **5qa**:



5qa ^{1}H NMR 600 MHz, $CDCl_3$

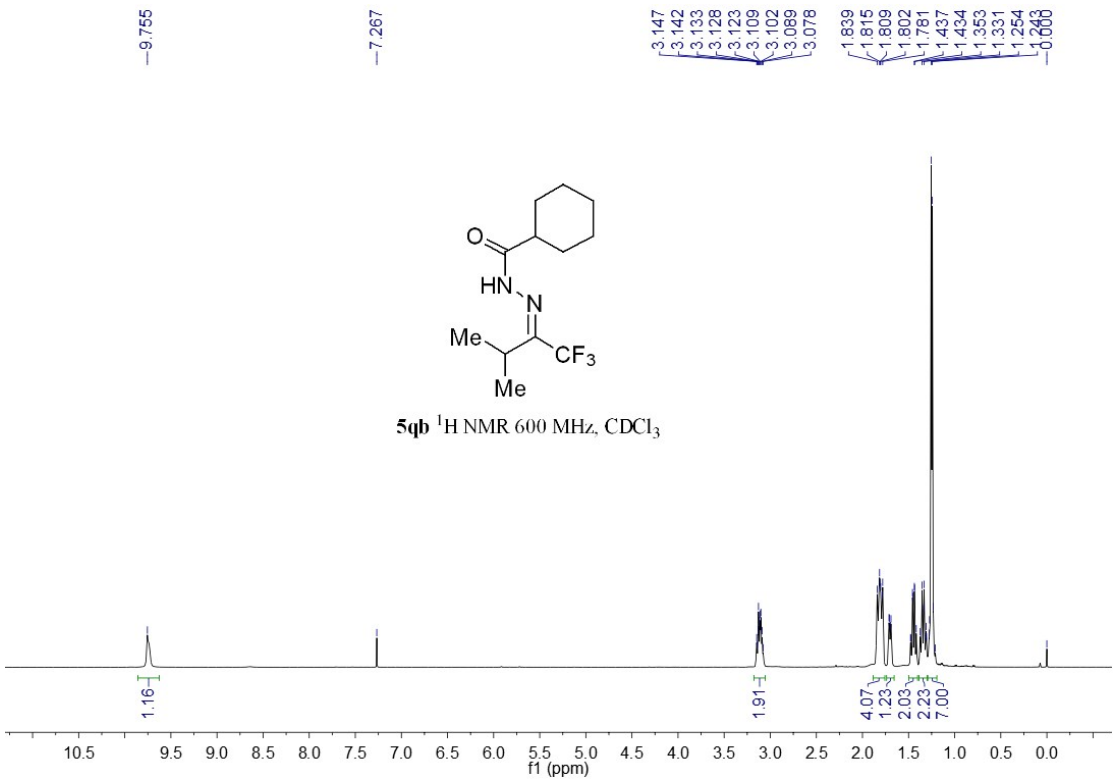


5qa ^{13}C NMR 150 MHz, $CDCl_3$

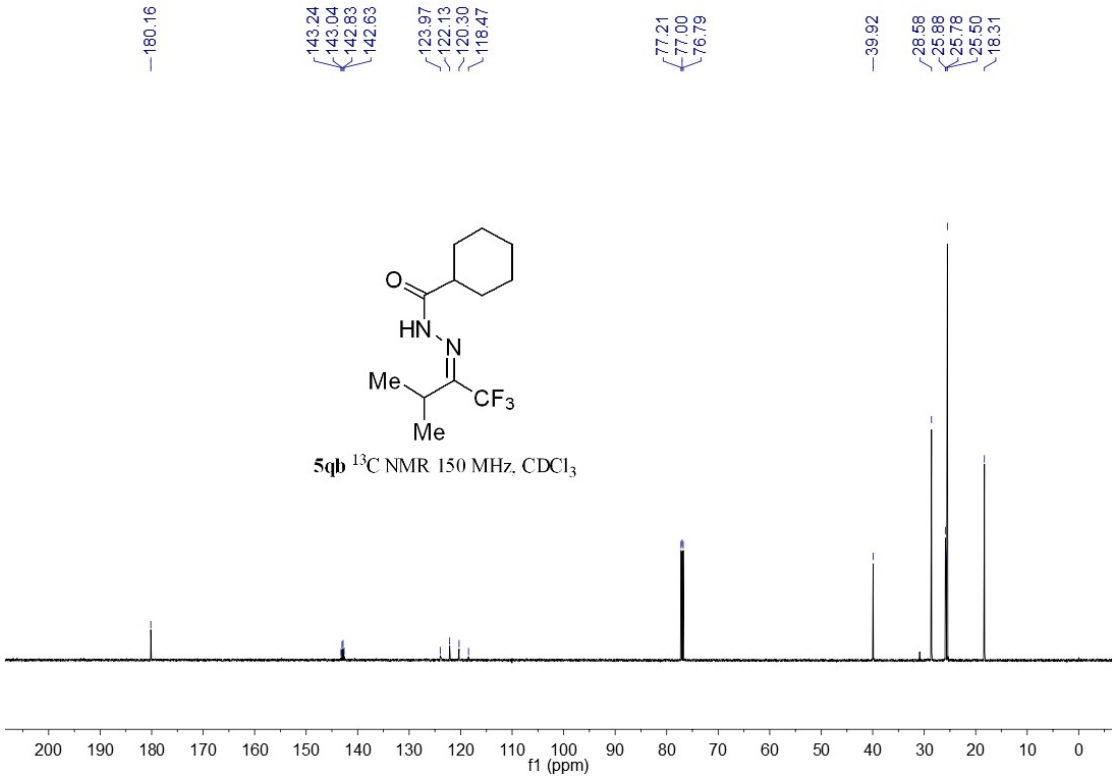


5qa ^{19}F NMR 376 MHz, $CDCl_3$

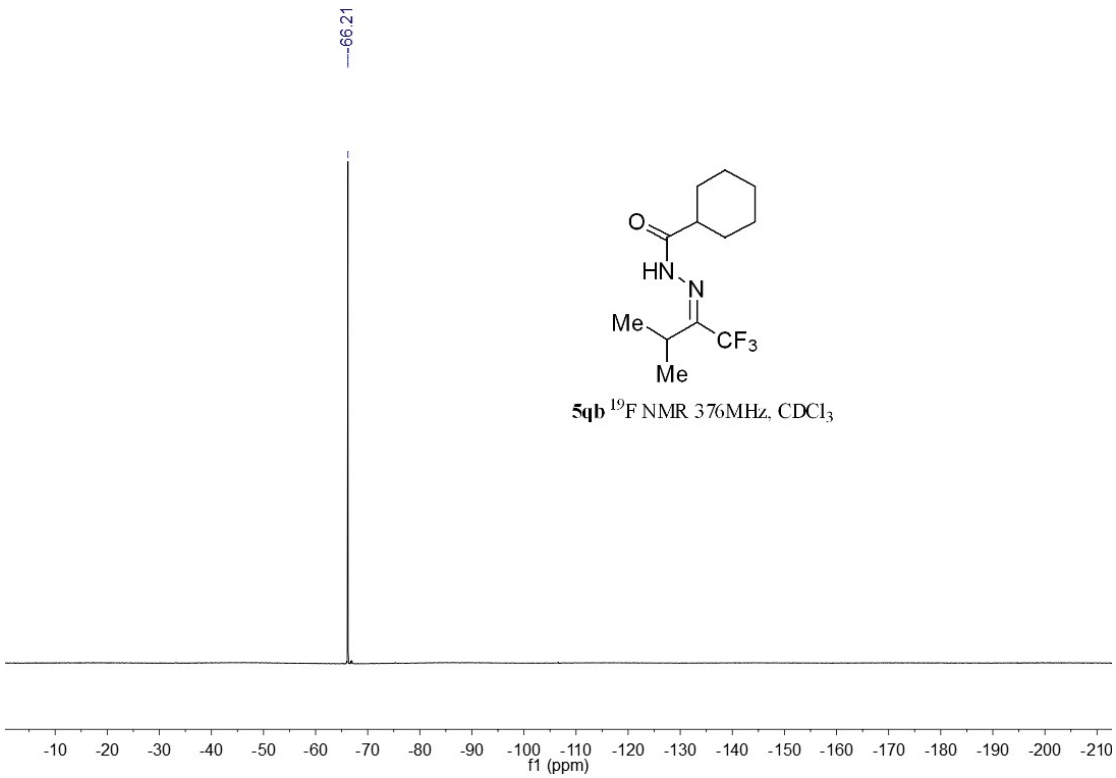
NMR copies of major product of compound **5qb**:



5qb ^{1}H NMR 600 MHz, $CDCl_3$

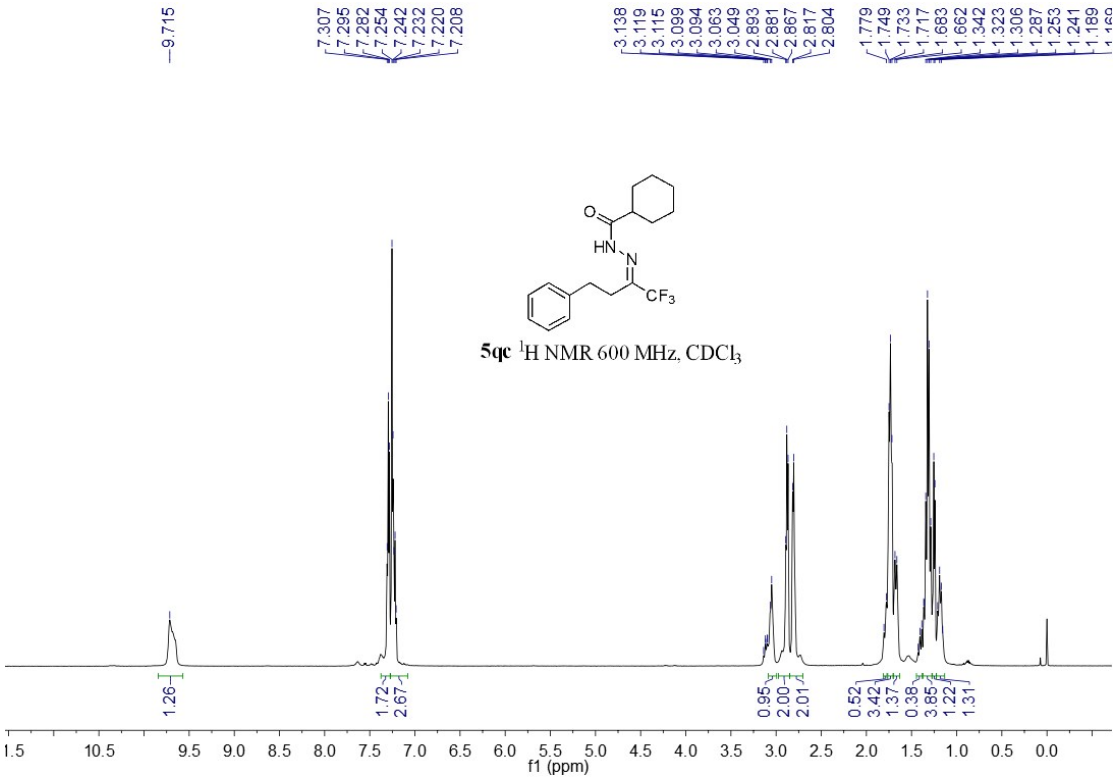


5qb ^{13}C NMR 150 MHz, $CDCl_3$

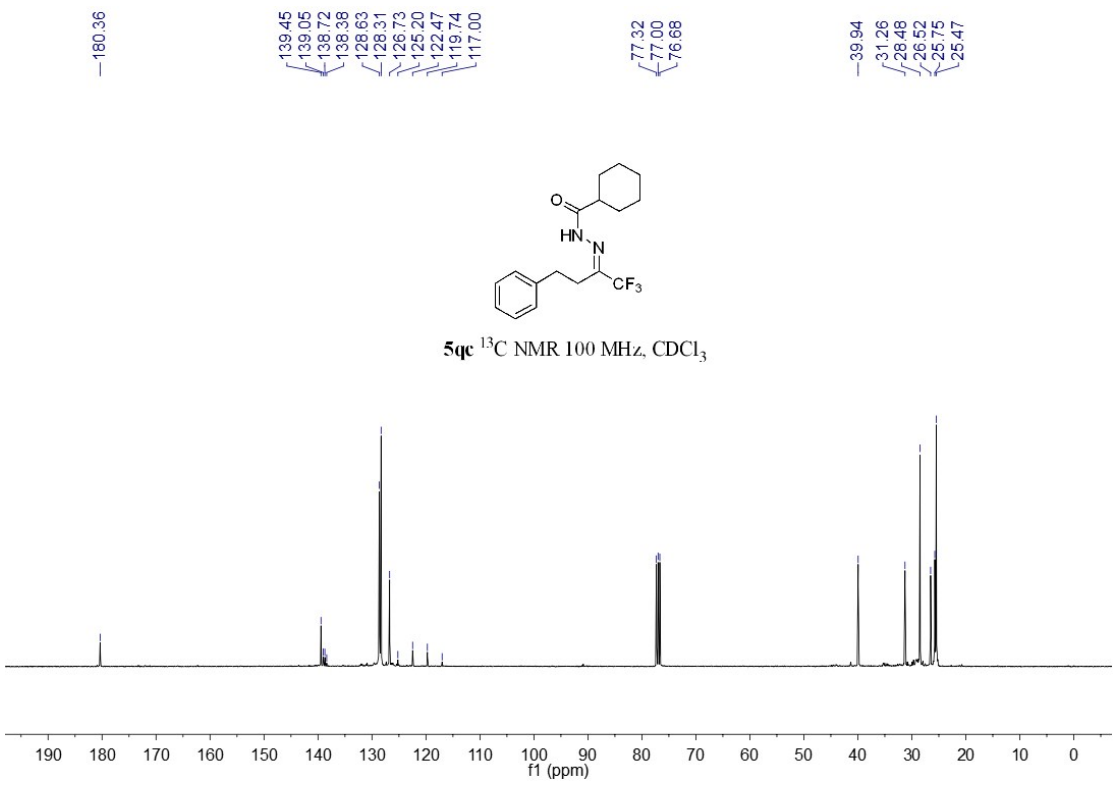


5qb ^{19}F NMR 376MHz, $CDCl_3$

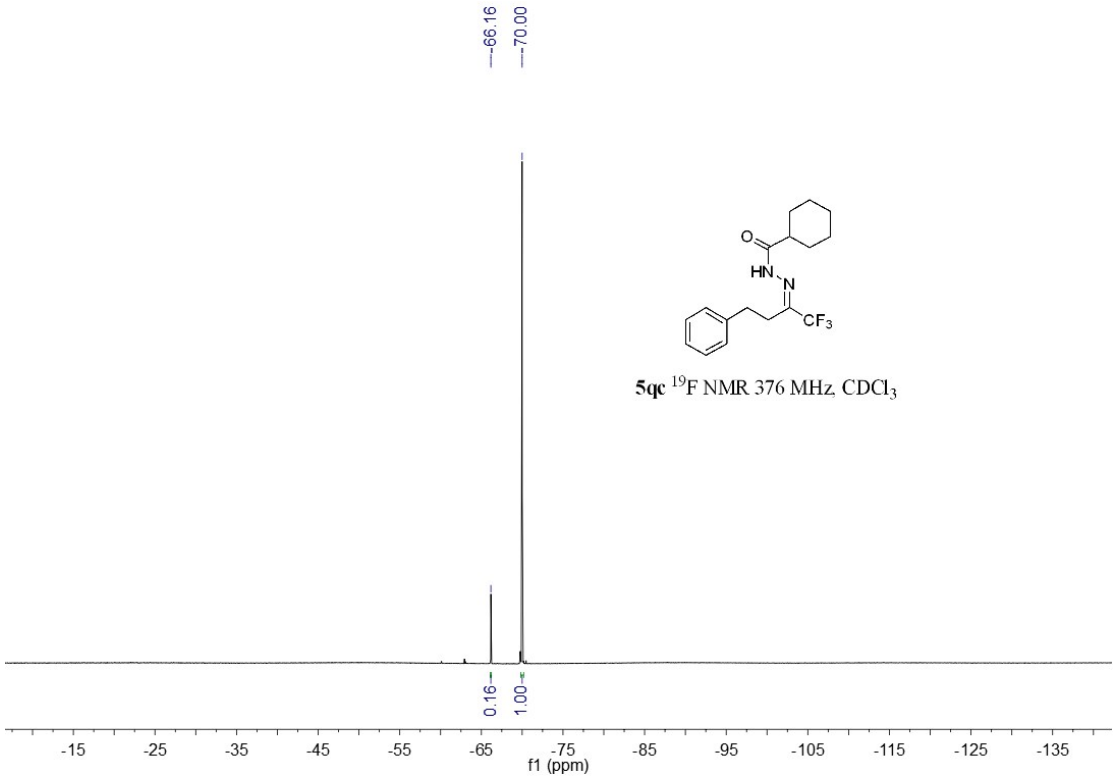
NMR copies of major product of compound **5qc**:



5qc ^{1}H NMR 600 MHz, $CDCl_3$

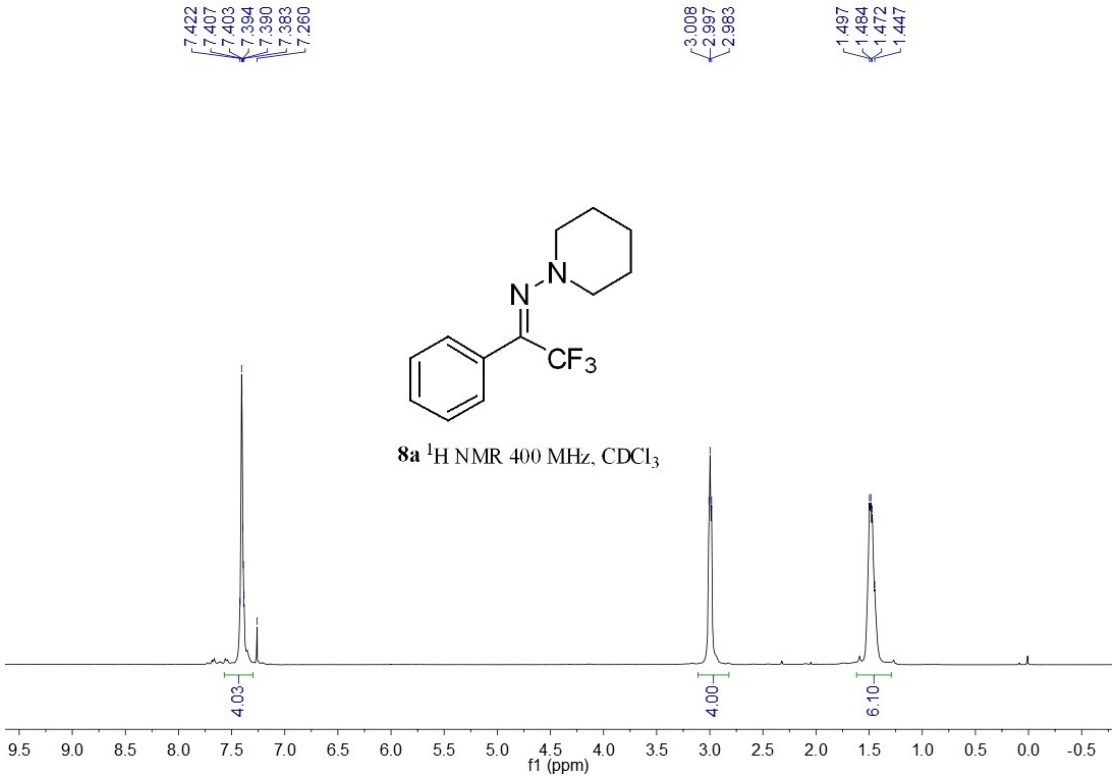


5qc ^{13}C NMR 100 MHz, $CDCl_3$

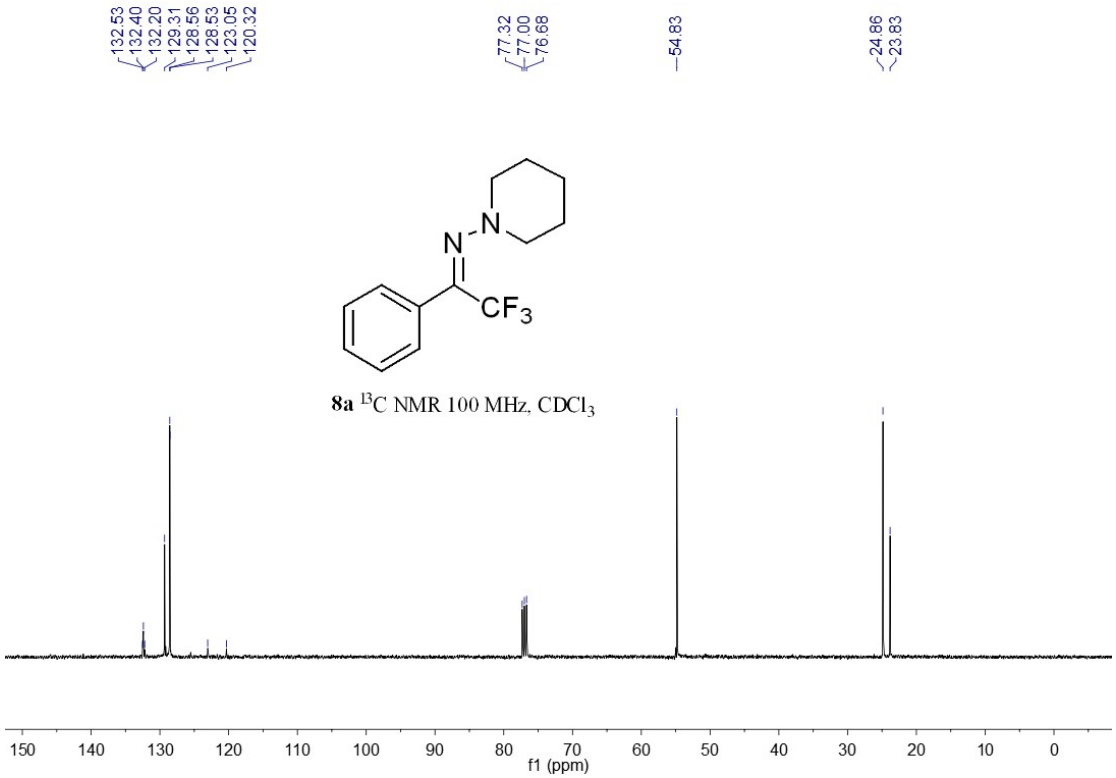


5qc ^{19}F NMR 376 MHz, $CDCl_3$

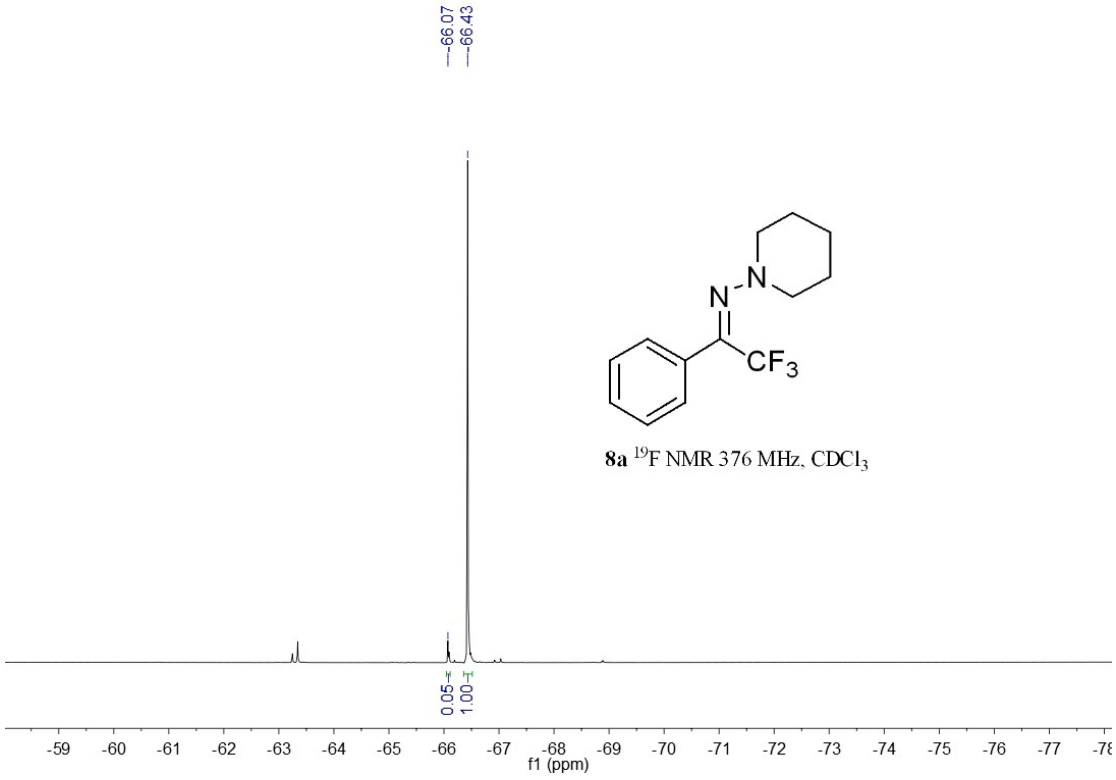
NMR copies of major product of compound **8a**:



8a ^{1}H NMR 400 MHz, $CDCl_3$

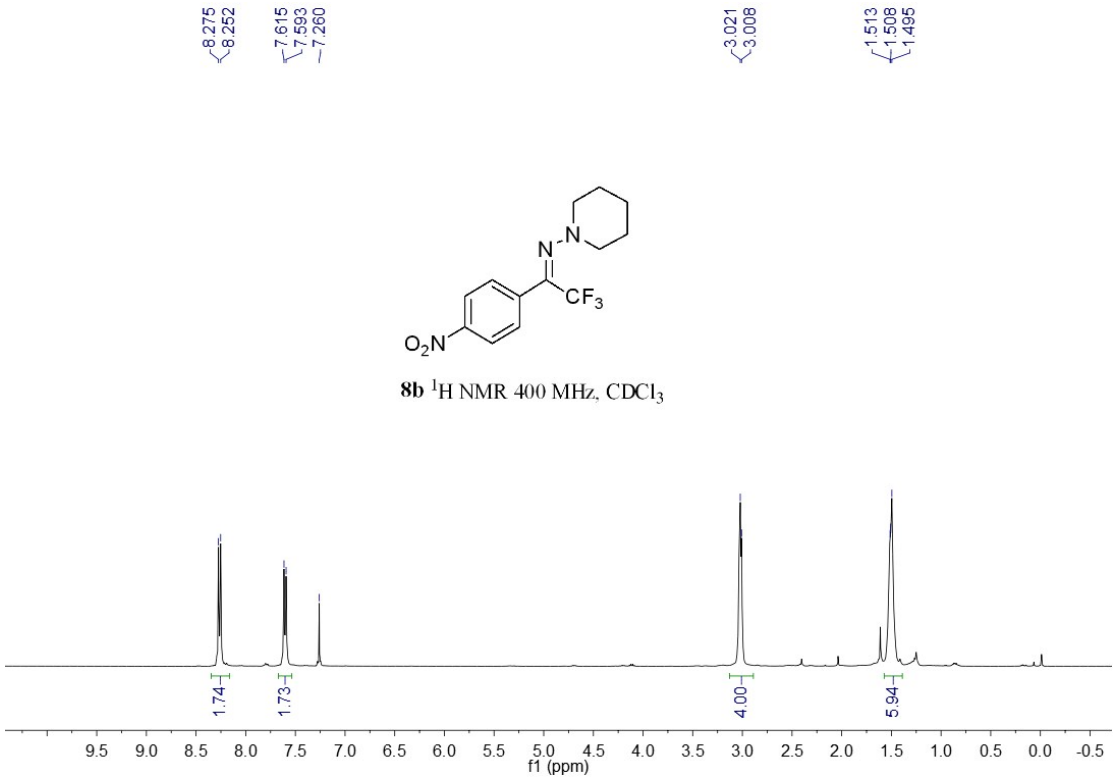


8a ^{13}C NMR 100 MHz, $CDCl_3$

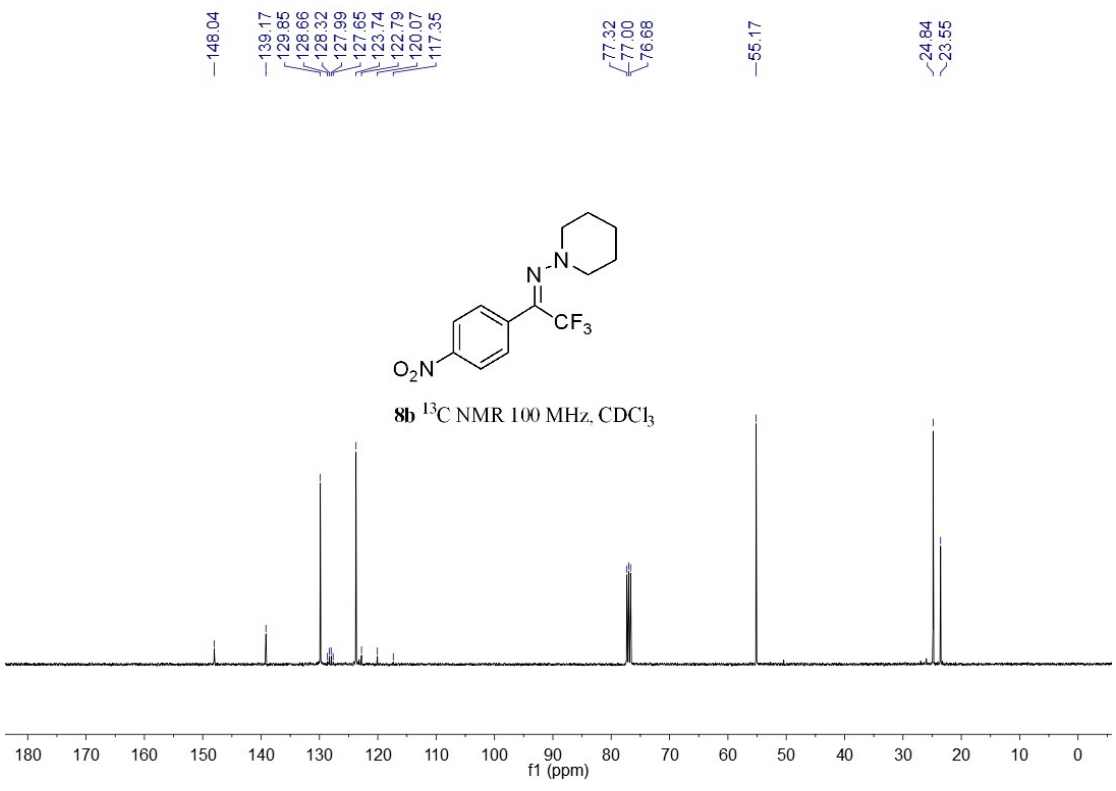


8a ^{19}F NMR 376 MHz, $CDCl_3$

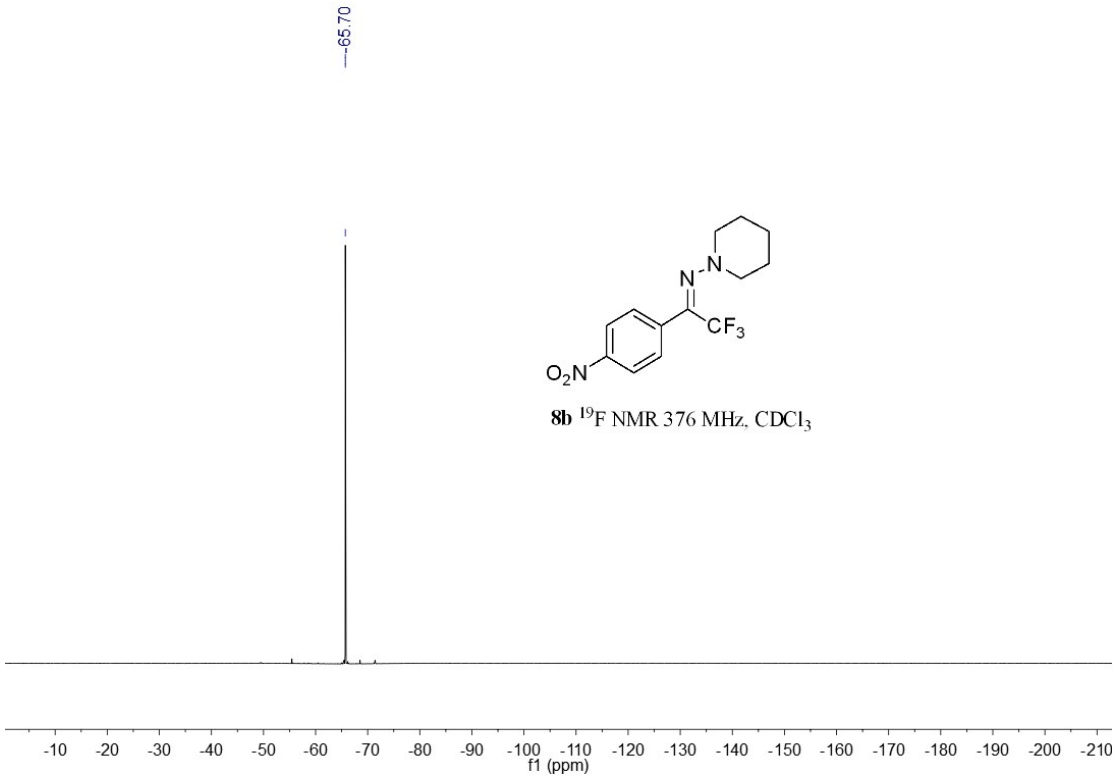
NMR copies of major product of compound **8b**:



8b ^{1}H NMR 400 MHz, $CDCl_3$

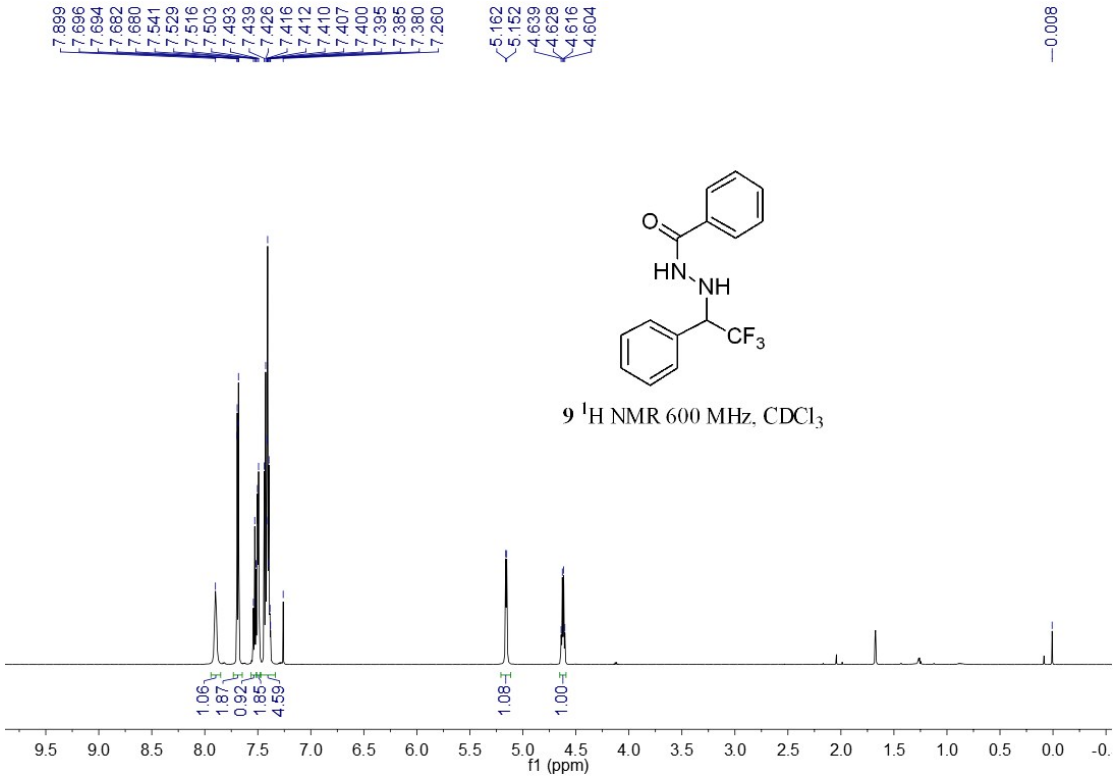


8b ^{13}C NMR 100 MHz, $CDCl_3$

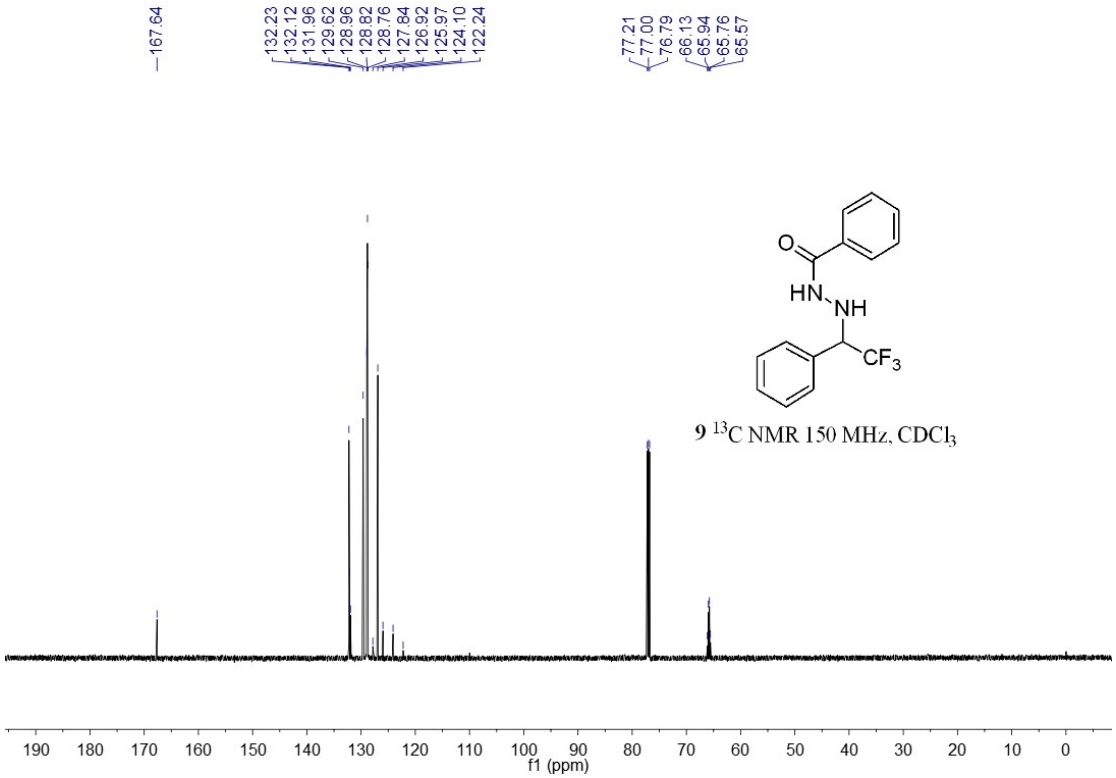


8b ^{19}F NMR 376 MHz, $CDCl_3$

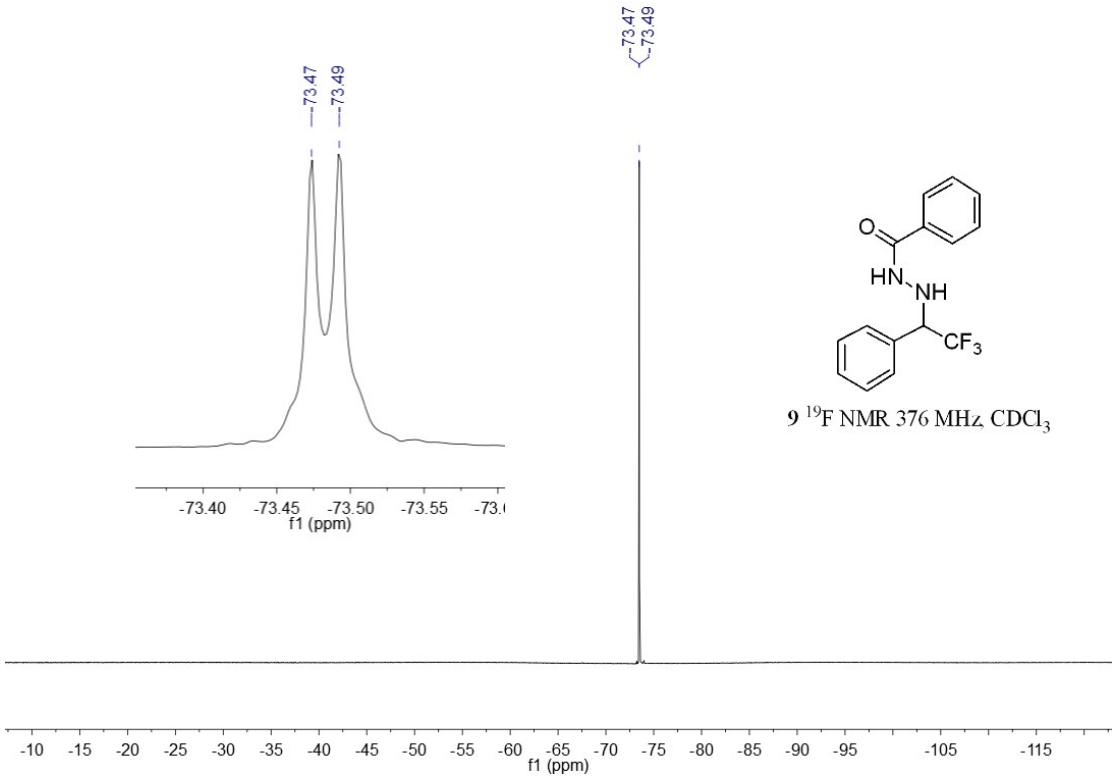
NMR copies of major product of compound **9**:



9 ^{1}H NMR 600 MHz, $CDCl_3$

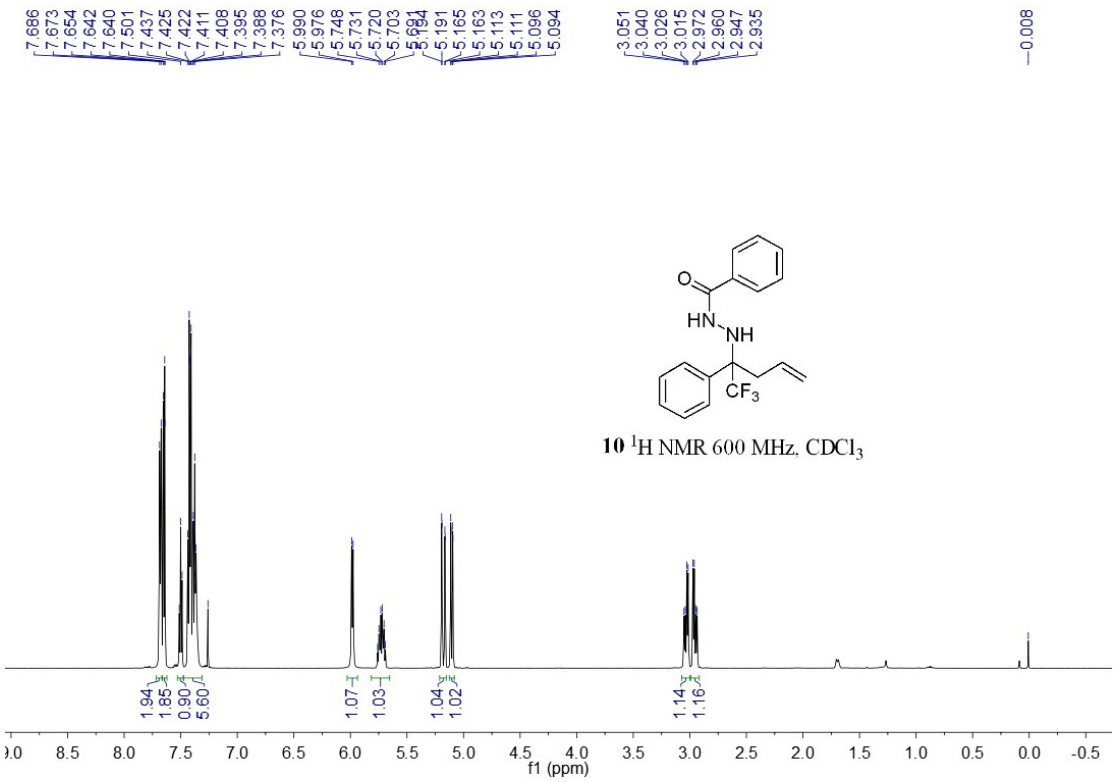


9 ^{13}C NMR 150 MHz, $CDCl_3$

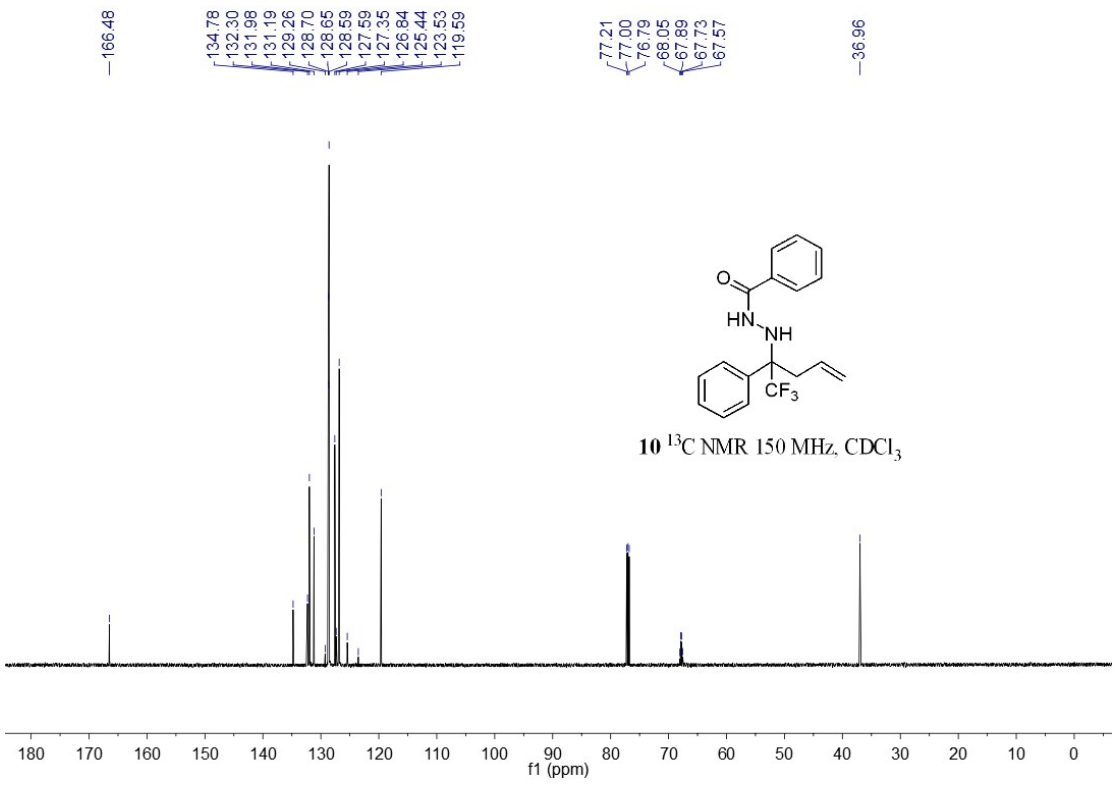


9 ^{19}F NMR 376 MHz, $CDCl_3$

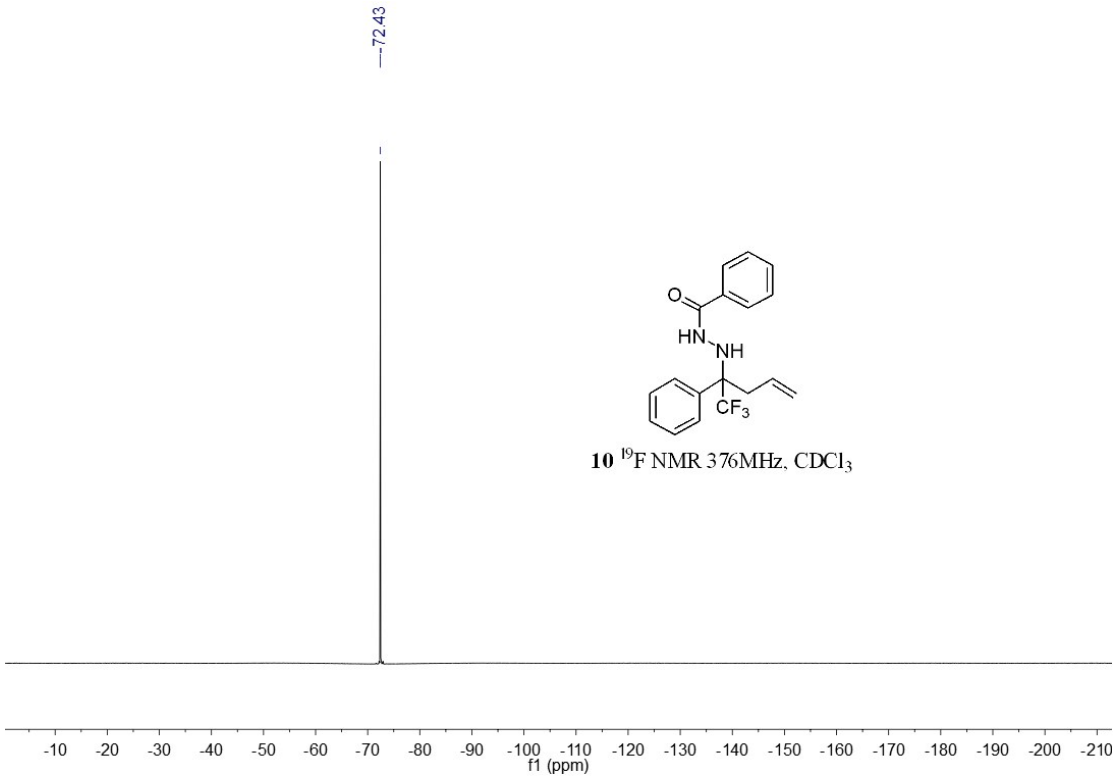
NMR copies of major product of compound **10**:



10 ^{1}H NMR 600 MHz, $CDCl_3$

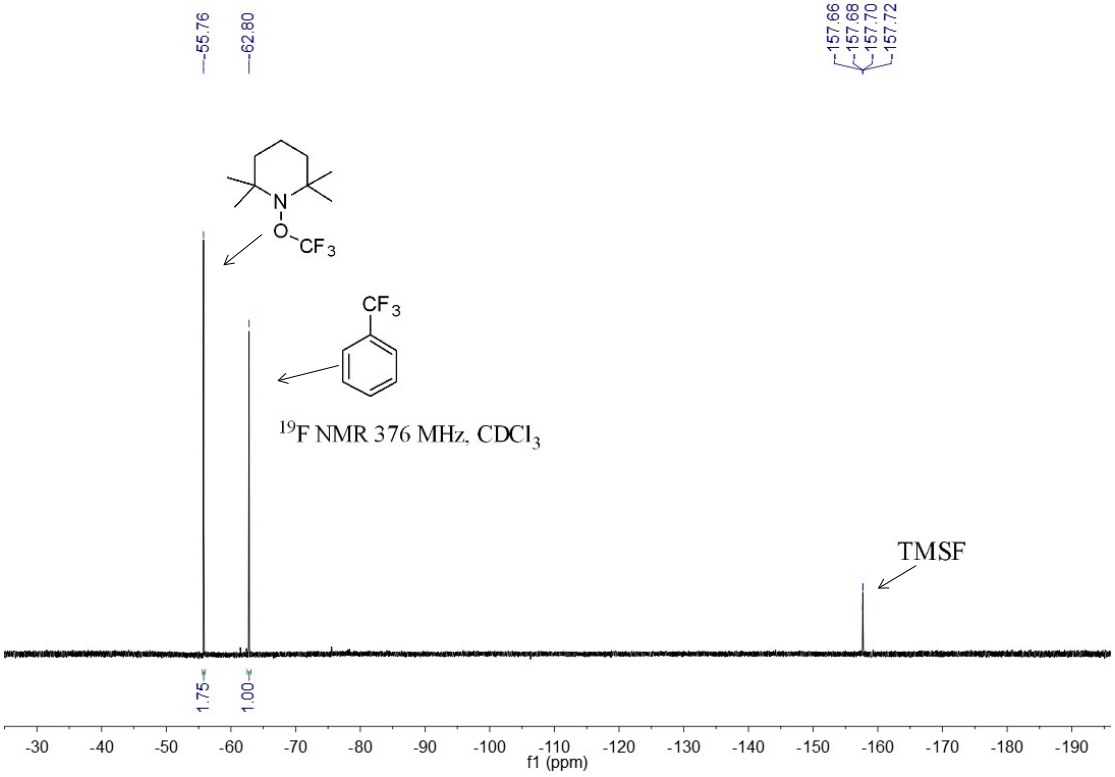


10 ^{13}C NMR 150 MHz, $CDCl_3$



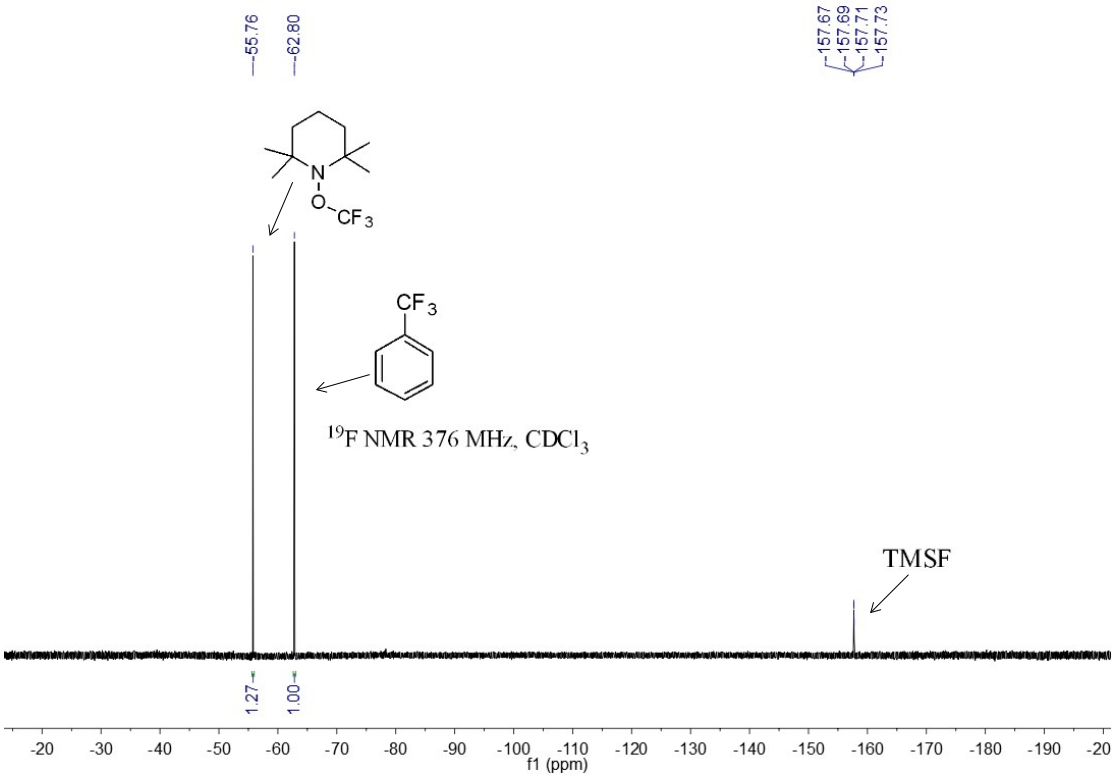
10 ^{19}F NMR 376MHz, $CDCl_3$

NMR copie of control experiment of **1jh** under standard reaction condition with TEMPO:

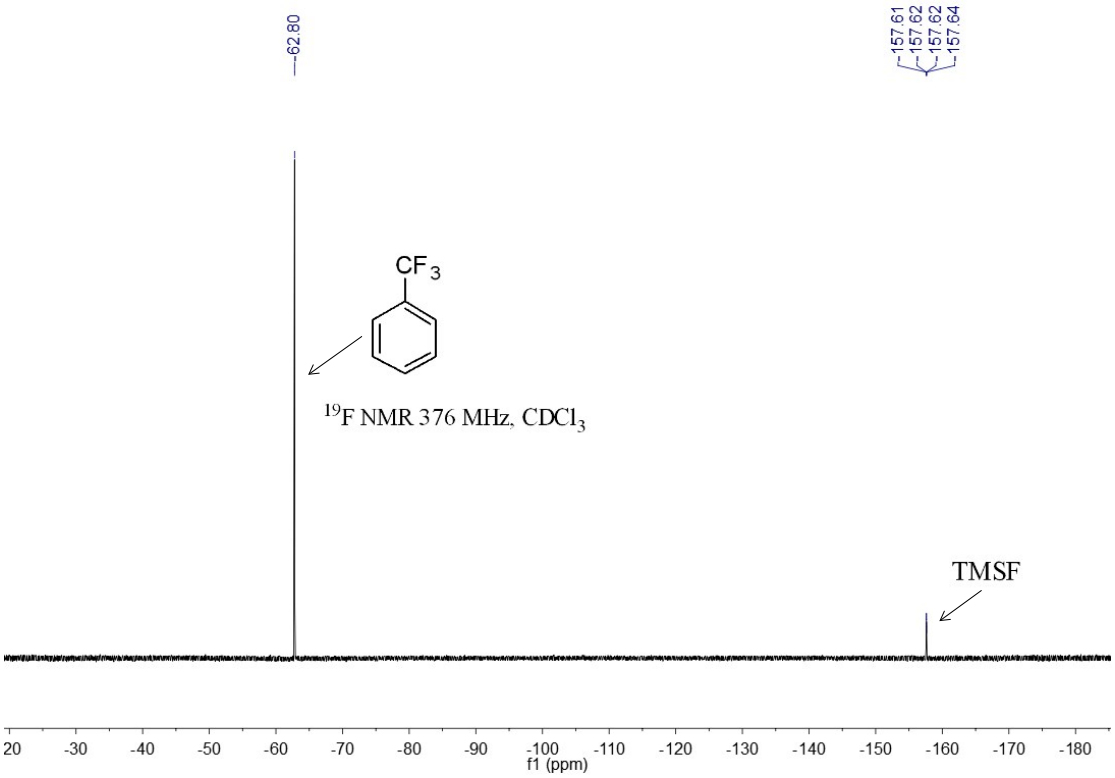


^{19}F NMR 376 MHz, $CDCl_3$

NMR copie of control experiment to proof the existence of the CF_3 radical **18**:



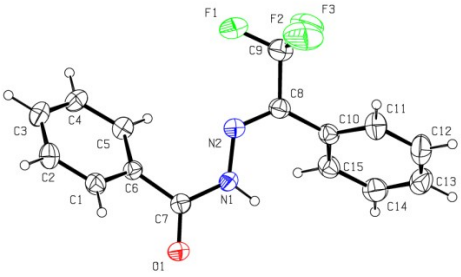
NMR copie of control experiment to proof the existence of the intermediate TMSF **13**:



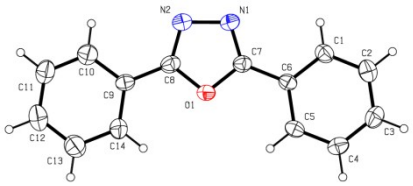
2. X-ray crystallographic data of 5a and 6a

The structure of **5a** and **6a** were determined by the X-ray diffraction analysis.

CCDC 1482106 (**5a)**, CCDC 1481842 (**6a')** contain the structure and supplementary crystallographic data. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.



5a



6a

Table 1 Crystal data and structure refinement for Compounds **5a** and **6a**

Identification code	**5a**	**6a**
CCDC Deposit number	1482106	1481842
Empirical formula	$C_{15}H_{11}F_3N_2O$	$C_{14}H_{10}N_2O$
Formula weight	292.26	222.24
Temperature (K)	294(16)	293(10)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
space group	$P2_1/c$	$P2_1/c$
Unit cell dimensions (Å)	a = 5.7064(5) b = 17.0772(13) c = 14.1572(9)	a = 5.1901(6) b = 18.063(3) c = 12.1410(13)
(°)	α = 90.00 β = 94.665(7) γ = 90.00	α = 90 β = 93.142(11) γ = 90
Volume	1375.04(18) $Å^3$	1136.5(3) $Å^3$
Z	4	4

Calcd. density (Mg/m^3)	1.412	1.299
F(000)	600.0	464
Limiting indices	-7≤ h ≤6 -21≤ k ≤ 20 -17≤ l ≤ 17	-6≤ h ≤6 -22≤ k ≤ 19 -14≤ l ≤ 9