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Photocatalytic α-arylation of cyclic ketones by a thermally activated delayed fluorescence molecule

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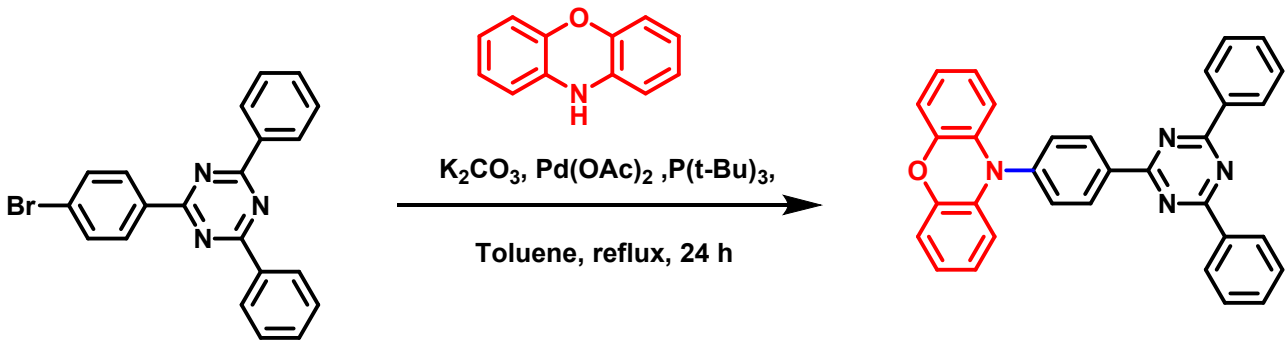
1. General information:

Unless otherwise noted, all chemicals were purchased from commercial suppliers (Sigma Aldrich, TCI, BLD pharma) and used without further purification. When required, solvents were dried according to general purification methods. Two blue light source (30 W) were used as the light source. The product mixtures were analyzed by thin layer chromatography using TLC silica gel plates (Merck) with UV indicator (λ = 254 nm). The purification of the products was performed by column chromatography using SD Fine silica gel (60-120 μm) using a gradient of ethyl acetate and hexane as mobile phase. ^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded on a 400 MHz Bruker Biospin Advance III FT-NMR spectrometer. Chemical shifts were calibrated using residual undeuterated solvent as an internal reference ($CDCl_3$: 7.26 ppm for ^{1}H NMR, 77.16 ppm for ^{13}C NMR). Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), td (triplet of doublets), dt (doublet of triplets), ddd (doublet of doublets of doublets), brs (broad singlet). Absorption spectra were recorded using LAB-INDIA UV/Vis Spectrophotometer UV 3000 in an UV-cuvette of path length 10 mm fitted with cap. Emission spectra were collected by Fluoromax-4 (Horiba Jobin Yvon, NJ) Spectrofluorometer. The analyte solution was placed in quartz cuvettes equipped with screw cap having a path length of 10 mm. The photochemical reactions were conducted with two Eplite 30 W blue LED. The reaction tube was kept 7-8 cm away from the light source.

2. Synthesis of the photocatalyst:

The photocatalyst (PXZ-TRZ) was prepared according to the previously reported literature procedure[1]



Procedure: To a solution of 1.94 g of 2-Bromo-4,6-diphenyl-1,3,5-triazine (5.0 mmol), 1.01 gm of phenoxazine (5.5 mmol) and 2.07 g of potassium carbonate (15 mmol) in 40 mL of toluene was added. After that 33.6 mg of palladium (II) acetate (0.15 mmol) and 111.3 mg of tri-tert-butylphosphine (0.55 mmol) in 40 mL of toluene was added with continuous stirring. Subsequently, the mixture was refluxed for 24 h with continuous stirring. Upon completion of the reaction, it was cooled and the reaction mixture is extracted with chloroform and water. The organic layer was separated, and the aqueous layer was extracted with chloroform. The combined organic layers were washed with brine, dried over Mg_2SO_4, and concentrated in vacuo. Column chromatography of the residue solid (eluent: chloroform/hexane=1/4) afforded 1.42 gm of PXZ-TRZ in 58% yield.

The product was fully characterized by ^1H and ^{13}C NMR spectroscopies.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.99 (d, *J* = 8.3 Hz, 2H), 8.81 (d, *J* = 7.2 Hz, 4H), 7.68 – 7.54 (m, 8H), 6.73 (d, *J* = 7.4 Hz, 2H), 6.69 (d, *J* = 7.2 Hz, 2H), 6.63 (d, *J* = 6.8 Hz, 2H), 6.05 (d, *J* = 7.8 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.32, 144.44, 136.83, 136.48, 134.43, 133.20, 132.19, 131.55, 129.48, 129.19, 123.78, 122.09, 116.07, 113.79.

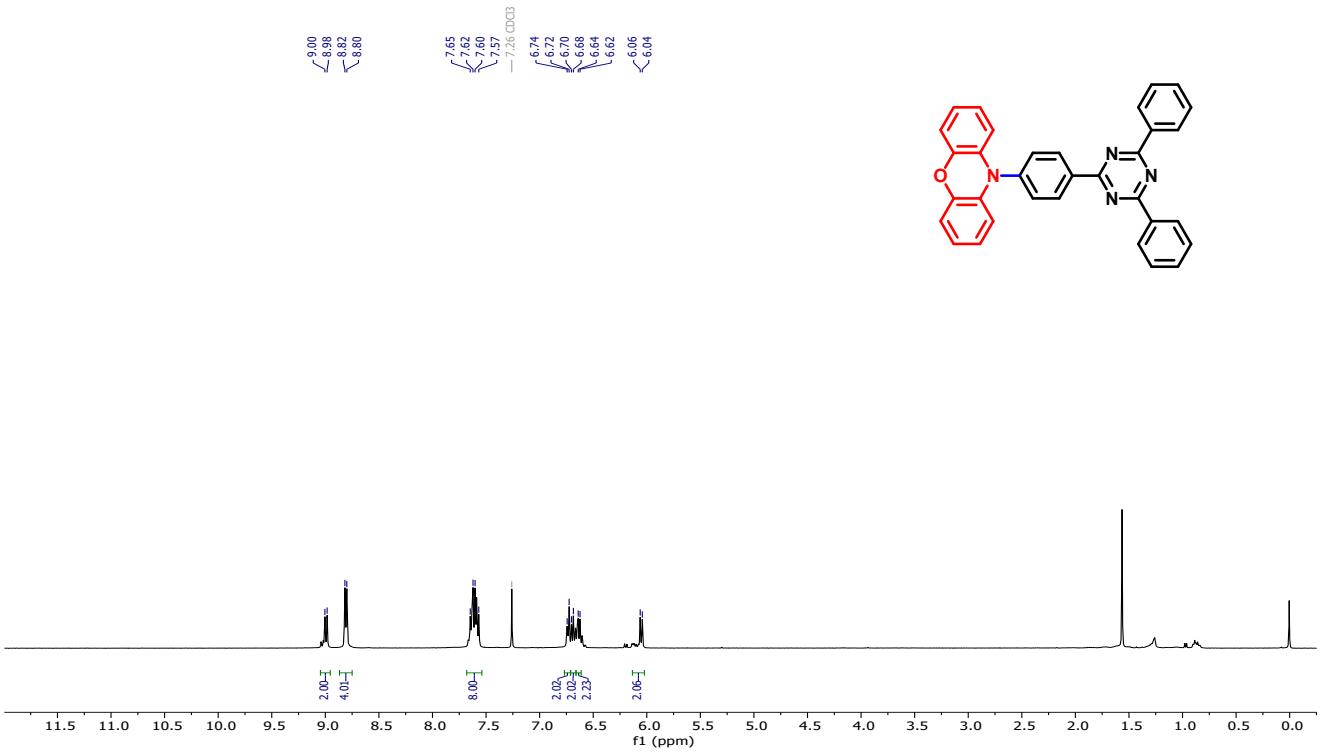


Figure S1. ^{1}H NMR spectrum (400 MHz) of **PXZ-TRZ** in $CDCl_3$.

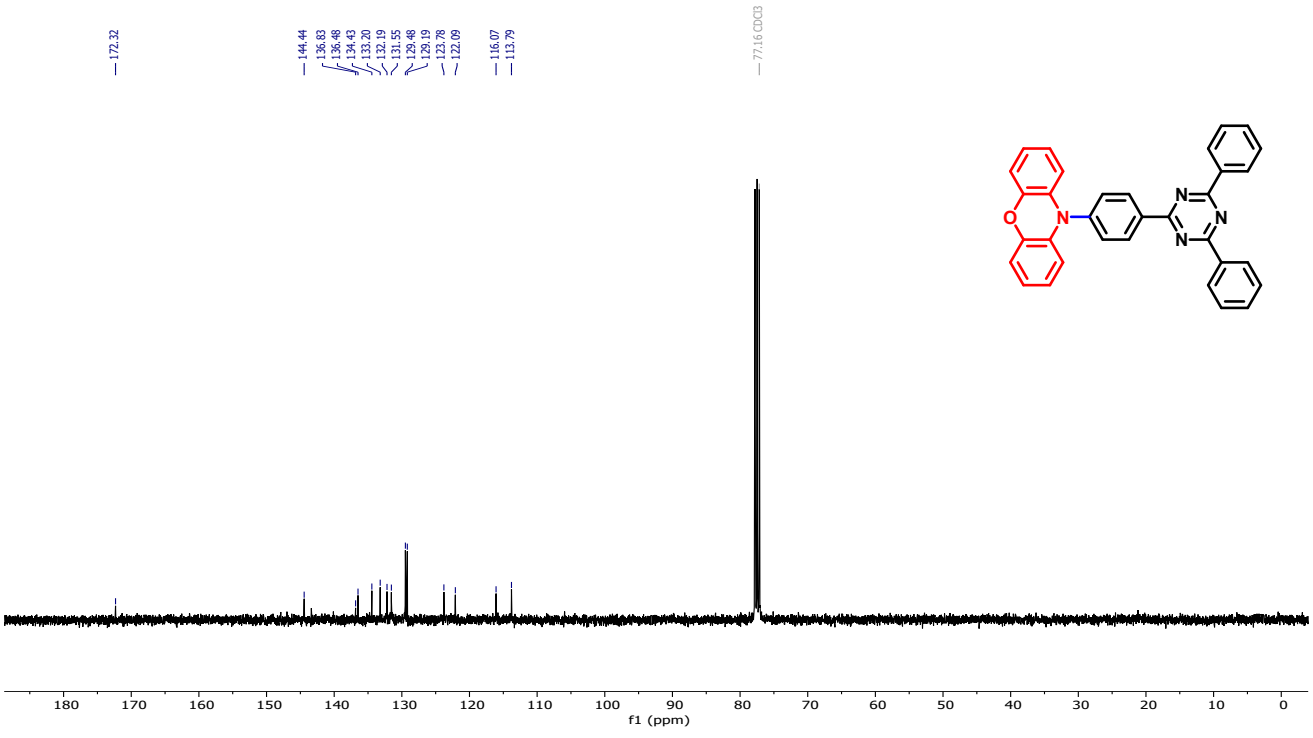
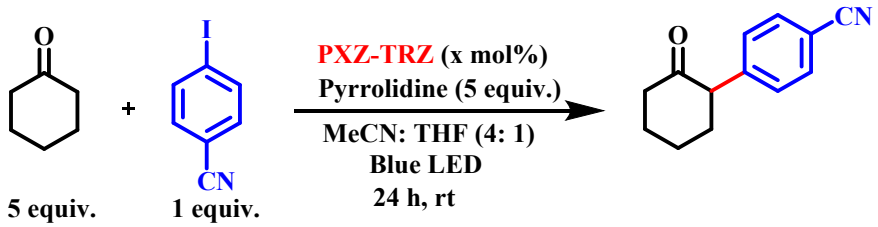


Figure S2. ^{13}C NMR spectrum (101 MHz) of **PXZ-TRZ** in $CDCl_3$

3. Optimization of reaction conditions:

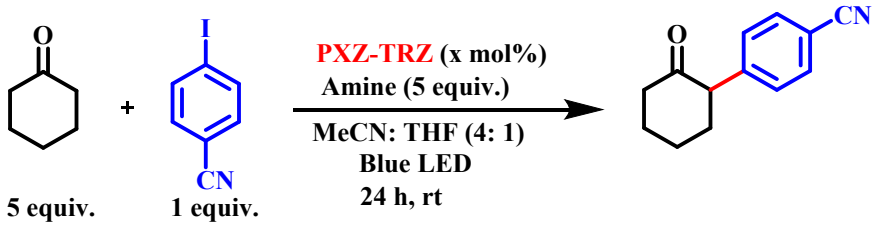
1. Screening of catalyst amount loading



Entry	Catalyst loading (mol%)	Product yield (%) *
1	0.2	68
2	0.3	72
3	0.4	79
4	0.5	94
5	0.7	87

*All yields are calculated from ^{1}H NMR spectra using ferrocene as an internal standard.

2. Screening of different amines

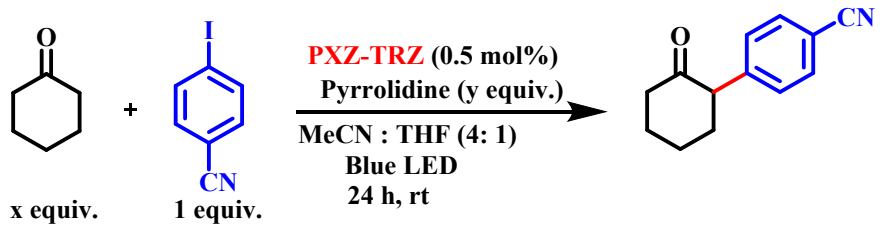


Entry	PXZ-TRZ (mol %)	Amine	Product yield (%)*
1	0.5	Pyrrolidine	94
2	0.5	Piperidine	14
3	0.5	Diphenyl amine	trace
4	0.5	Diethyl amine	trace
5	0.5	Morpholine	trace
6	0.5	Thiomorpholine	trace

7	0.5	N-methylpiperazine	trace
8	0.5	Diisopropyl amine	trace
9	0.5	Dicyclohexyl amine	trace
10	0.5	Dibenzyl amine	trace

*All yields are calculated from ^{1}H NMR spectrum using ferrocene as an internal standard.

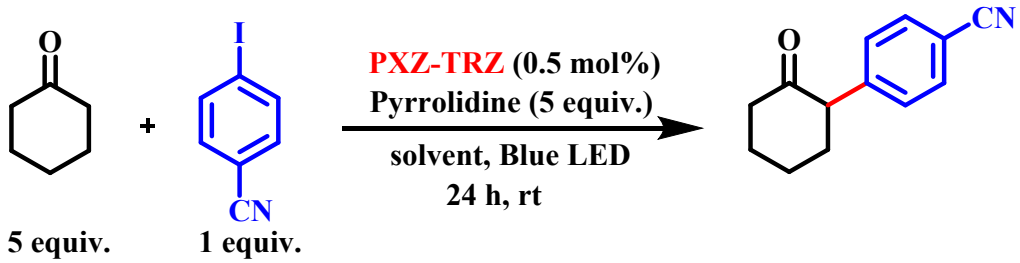
3. Screening of Ketone and Amine Equivalence:



Entry	PXZ-TRZ (mol%)	Cyclohexanone	Pyrrolidine	Product yield (%)*
1	0.5	5 equiv	5 equiv	94
2	0.5	4 equiv	4 equiv	73
3	0.5	3 equiv	3 equiv	64
4	0.5	2 equiv	2 equiv	45
5	0.5	8 equiv	8 equiv	48
6	0.5	10 equiv	10 equiv	36

*All yields are calculated from ^{1}H NMR spectra using ferrocene as an internal standard.

4. Screening of Solvents:



5 equiv. **1 equiv.**

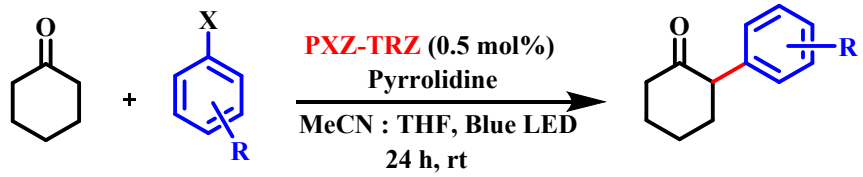
Entry	PXZ-TRZ (mol%)	Solvent	Product yield (%)*
1	0.5	MeCN	89
2	0.5	THF	84
3	0.5	MeCN : THF(4:1)	94
3	0.5	Toluene	81
4	0.5	Benzene	34
5	0.5	Dioxane	52
6	0.5	Hexane	12
7	0.5	MeOH	trace

*All yields are calculated from ^{1}H NMR spectra using ferrocene as an internal standard.

4. Experimental Procedures:

General procedure for the α-arylation of ketone

(a)

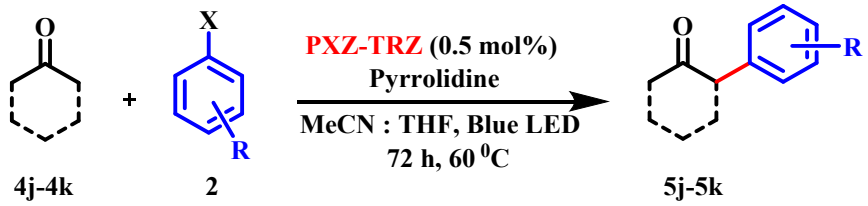


1,4 **2** **3a-3u, 5a-5i**

An oven-dried 10 mL screw capped pressure tube, equipped with a magnetic stir bar was charged with aryl halide (0.2 mmol), corresponding ketone (1 mmol), pyrrolidine (1 mmol) and PXZ-TRZ photocatalyst (0.5 mol%) in dry mixture solvent MeCN:THF (4:1, 2 mL) inside a nitrogen-filled glove box. The cap was tightly closed and taken out of the glove box. The reaction mixture was

irradiated with two blue LED (30 W each) for 24 hours with continuous stirring. After completion of the reaction the solvent was evaporated in rotary evaporator. The crude reaction mixture was neutralized by 1.0 N HCl and the aqueous layer was extracted with Et_2O. The organic layer was collected, washed with brine, dried in $MgSO_4$ and evaporated in vacuum. The residue was purified by column chromatography using hexane: ethyl acetate (10:1 to 5:1) as eluent to obtain the pure product.

(b) α-arylation of cycloheptanone and cyclooctanone



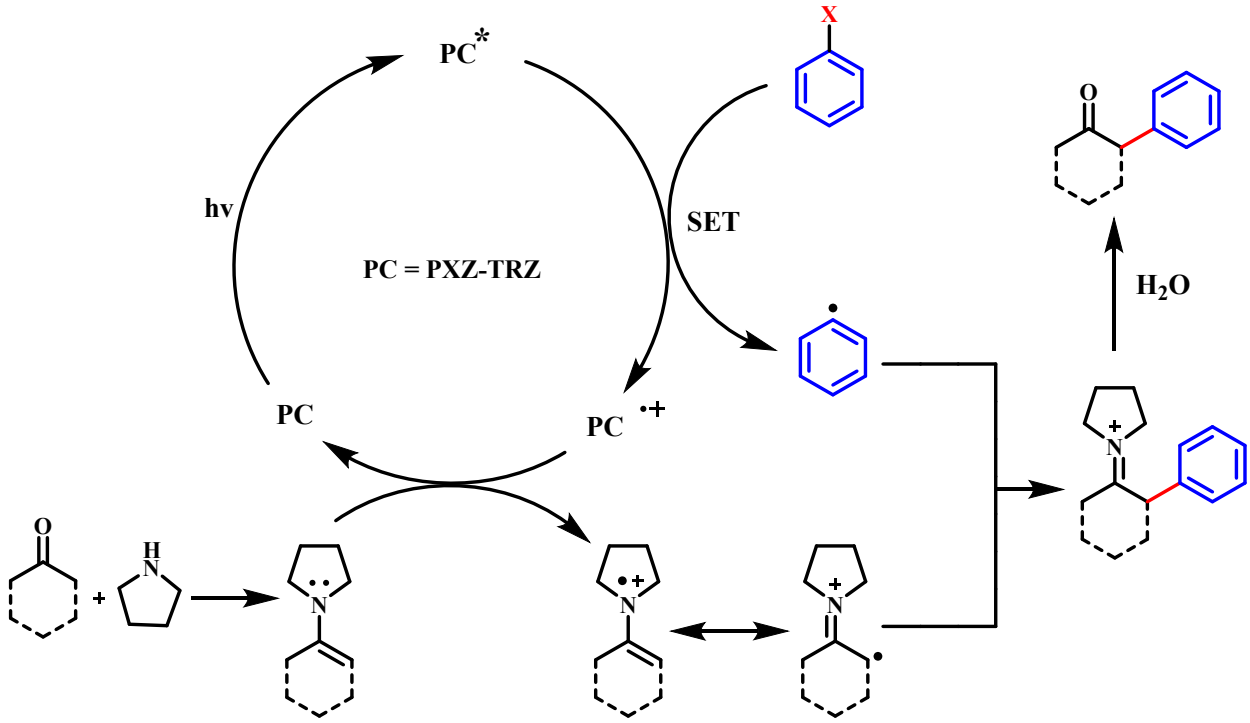
4j-4k **2** **5j-5k**

An oven-dried 10 mL screw capped pressure tube, equipped with a magnetic stir bar was charged with aryl halide (0.2 mmol), corresponding ketone (1 mmol), pyrrolidine (1 mmol) and PXZ-TRZ photocatalyst (0.5 mol%) in dry mixture solvent MeCN:THF (4:1, 2 mL) inside a nitrogen-filled glove box. The cap was tightly closed and taken out of the glove box. The reaction mixture was irradiated with two blue LED (30 W each) for 72 hours with 60 ^{0}C heat with continuous stirring. After completion of the reaction the solvent was evaporated in rotary evaporator. The crude reaction mixture was neutralized by 1.0 N HCl and the aqueous layer was extracted with Et_2O. The organic layer was collected, washed with brine, dried in $MgSO_4$ and evaporated in vacuum. The residue was purified by column chromatography using hexane: ethyl acetate (10:1 to 5:1) as eluent to obtain the pure product.

(c) Procedure for gram scale reaction

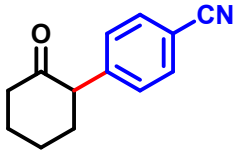
An oven-dried 100 mL Schlenk flask, equipped with a magnetic stir bar was charged with 4-iodo benzonitrile (5 mmol), cyclohexanone (25 mmol), pyrrolidine (25 mmol) and PXZ-TRZ photocatalyst (0.5 mol%) in dry mixture solvent MeCN:THF (4:1, 50 mL) inside a nitrogen-filled glove box. The Schlenk flask was taken out of the glove box and it was irradiated with two blue LED (30 W each) for 30 hours with continuous stirring. After completion of the reaction the solvent was evaporated in a rotary evaporator. The crude reaction mixture was neutralized by 1.0 N HCl and the aqueous layer was extracted with Et_2O. The organic layer was collected, washed

with brine, dried in $MgSO_4$ and evaporated under vacuum. The residue was purified by column chromatography using hexane: ethyl acetate (10:1 to 5:1) as eluent to obtain the pure product. Yield, 650 mg, 65%.



5. Plausible mechanistic cycle for α-arylation reaction

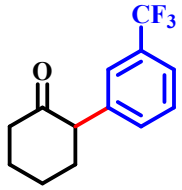
6. Characterization data



4-(2-oxocyclohexyl) benzonitrile (3a): The compound is prepared according to the general procedure described above. White solid, yield 91% from aryl iodide and 56% from aryl bromide substrates.

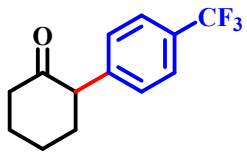
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.2 Hz, 2H), 3.67 (dd, *J* = 12.5, 5.3 Hz, 1H), 2.55 – 2.45 (m, 2H), 2.26 (d, *J* = 9.8 Hz, 1H), 2.18 (dd, *J* = 9.1, 3.7 Hz, 1H), 2.05 – 1.93 (m, 2H), 1.87 – 1.78 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.04, 144.33,

132.10, 129.61, 118.99, 110.73, 57.47, 42.25, 35.14, 27.75, 25.34. Spectroscopic data matched with the literature.[3]



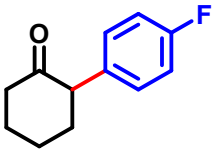
2-(3-(trifluoromethyl) phenyl) cyclohexan-1-one (3b): The compound is prepared according to the general procedure described. White solid, Yield 74%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 7.8 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 1H), 7.39 (s, 1H), 7.33 (d, *J* = 7.7 Hz, 1H), 3.68 (dd, *J* = 12.4, 5.4 Hz, 1H), 2.59 – 2.46 (m, 2H), 2.33 – 2.26 (m, 1H), 2.19 (d, *J* = 3.9 Hz, 1H), 2.01 (d, *J* = 9.8 Hz, 2H), 1.84 (t, *J* = 11.0 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.52, 132.31,130.83,130.52,128.50 125.49, 123.84, 42.34, 57.34 35.41, 27.87, 25.50. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.49. Spectroscopic data matched with the literature.[4]



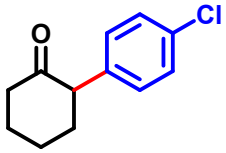
2-(4-(trifluoromethyl)phenyl)cyclohexan-1-one (3c): The compound is prepared according to the general procedure described. White solid, Yield 68%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 7.8 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 1H), 7.39 (s, 1H), 7.33 (d, *J* = 7.7 Hz, 1H), 3.68 (dd, *J* = 12.4, 5.4 Hz, 1H), 2.59 – 2.46 (m, 2H), 2.33 – 2.26 (m, 1H), 2.19 (d, *J* = 3.9 Hz, 1H), 2.01 (d, *J* = 9.8 Hz, 2H), 1.84 (t, *J* = 11.0 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.52, 132.31, 125.58 (d, *J* = 18.8 Hz), 123.90 (d, *J* = 3.7 Hz), 42.34, 35.41, 27.87, 25.50. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.49. Spectroscopic data matched with the literature.[3]



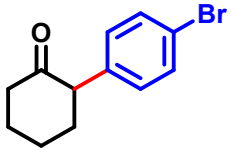
2-(4-fluorophenyl)cyclohexan-1-one (3d): The compound is prepared according to the general procedure described. White solid, Yield 77%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.10 (dd, *J* = 8.7, 5.5 Hz, 2H), 7.02 (t, *J* = 8.7 Hz, 2H), 3.60 (dd, *J* = 12.3, 5.3 Hz, 1H), 2.57 – 2.41 (m, 2H), 2.30 – 2.23 (m, 1H), 2.21 – 2.13 (m, 1H), 2.04 – 1.95 (m, 2H), 1.89 – 1.78 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.33, 163.12, 134.57, 115.42, 115.20, 56.81, 42.35, 35.59, 27.95, 25.56, 1.15. Spectroscopic data matched with the literature.[3]



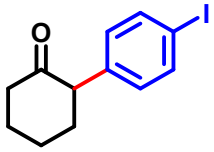
2-(4-chlorophenyl) cyclohexan-1-one (3e): The compound is prepared according to the general procedure described. White solid, Yield 71%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.4 Hz, 2H), 3.59 (dd, *J* = 12.3, 5.3 Hz, 1H), 2.50 (ddd, *J* = 18.4, 13.1, 8.7 Hz, 2H), 2.29 – 2.22 (m, 1H), 2.20 – 2.14 (m, 1H), 2.04 – 1.94 (m, 2H), 1.86 – 1.78 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.99, 137.32, 132.80, 130.05, 128.60, 56.93, 35.39, 27.91, 25.49. Spectroscopic data matched with the literature.[6]



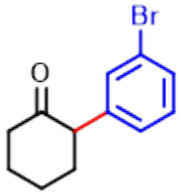
2-(4-bromophenyl) cyclohexan-1-one (3f): The compound is prepared according to the general procedure described. White solid, Yield 70%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 (d, *J* = 8.3 Hz, 2H), 7.01 (d, *J* = 8.3 Hz, 2H), 3.57 (dd, *J* = 12.3, 5.3 Hz, 1H), 2.52 (t, *J* = 11.2 Hz, 2H), 2.31 – 2.22 (m, 1H), 2.15 (s, 1H), 2.02 – 1.92 (m, 2H), 1.87 – 1.79 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.89, 137.84, 131.55, 130.44, 120.96, 57.00, 42.31, 35.34, 27.90, 25.48. Spectroscopic data matched with the literature.[6]



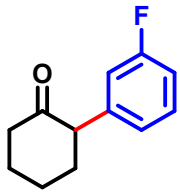
2-(4-iodophenyl) cyclohexan-1-one (3g): The compound is prepared according to the general procedure described. White solid, Yield 68%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 (s, 2H), 6.89 (d, *J* = 8.3 Hz, 2H), 3.56 (dd, *J* = 12.4, 5.3 Hz, 1H), 2.57 – 2.43 (m, 2H), 2.31 – 2.22 (m, 1H), 2.16 (d, *J* = 5.9 Hz, 1H), 2.03 – 1.93 (m, 2H), 1.85 – 1.77 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.87, 138.51, 137.49, 130.74, 92.56, 57.09, 42.30, 35.26, 27.88, 25.45. Spectroscopic data matched with the literature.[7]



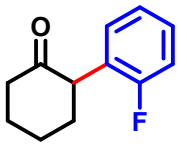
2-(3-bromophenyl) cyclohexan-1-one (3h): The compound is prepared according to the general procedure described. White solid, Yield 67%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 10.8 Hz, 1H), 7.29 (s, 1H), 7.20 (t, *J* = 7.8 Hz, 1H), 7.06 (d, *J* = 7.7 Hz, 1H), 3.57 (dd, *J* = 12.1, 5.3 Hz, 1H), 2.57 – 2.41 (m, 2H), 2.25 (d, *J* = 10.0 Hz, 1H), 2.20 – 2.13 (m, 1H), 2.04 – 1.93 (m, 2H), 1.81 (t, *J* = 10.5 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.66, 141.14, 131.75, 129.96, 127.47, 122.50, 57.15, 42.29, 35.23, 27.85, 25.43. Spectroscopic data matched with the literature.[8]



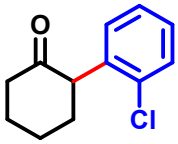
2-(3-fluorophenyl)cyclohexan-1-one (3i): The compound is prepared according to the general procedure described. White solid, Yield 70%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 – 7.25 (m, 1H), 6.98 – 6.83 (m, 3H), 3.61 (dd, *J* = 12.1, 5.4 Hz, 1H), 2.58 – 2.44 (m, 2H), 2.32 – 2.24 (m, 1H), 2.21 – 2.13 (m, 1H), 2.00 (d, *J* = 6.2 Hz, 2H), 1.88 – 1.78 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.82, 164.11, 141.36, 141.29, 129.86, 129.78, 124.41, 124.38, 115.76, 115.55, 114.01, 113.80, 57.17, 42.29, 35.16, 27.87, 25.40. Spectroscopic data matched with the literature.[6]



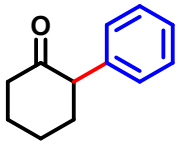
2-(2-fluorophenyl) cyclohexan-1-one (3j): The compound is prepared according to the general procedure described. White solid, Yield 69%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.26 – 7.21 (m, 1H), 7.18 – 7.09 (m, 2H), 7.06 – 7.00 (m, 1H), 3.84 (dd, *J* = 12.9, 5.4 Hz, 1H), 2.60 – 2.44 (m, 2H), 2.29 – 2.15 (m, 2H), 2.03 (d, *J* = 12.3 Hz, 2H), 1.87 – 1.77 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.57, 129.85, 128.62, 124.13, 115.52, 115.30, 51.15, 42.27, 33.84, 27.62, 25.71. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -116.86. Spectroscopic data matched with the literature.[6]



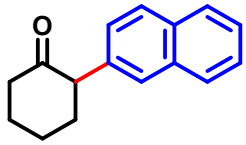
2-(2-chlorophenyl) cyclohexan-1-one (3k): The compound is prepared according to the general procedure described. White solid, Yield 60%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 6.1 Hz, 1H), 7.17 (d, *J* = 9.1 Hz, 2H), 4.08 (dd, *J* = 12.7, 5.3 Hz, 1H), 2.53 (d, *J* = 5.3 Hz, 2H), 2.29 – 2.15 (m, 2H), 2.01 (t, *J* = 11.5 Hz, 2H), 1.89 – 1.74 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.94, 136.82, 134.27, 129.50, 129.46, 128.22, 126.86, 54.11, 42.47, 34.00, 27.77, 25.76. Spectroscopic data matched with the literature.[6]



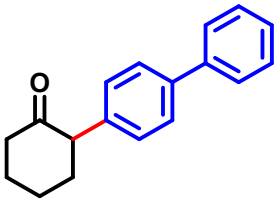
2-phenylcyclohexan-1-one (3l): The compound is prepared according to the general procedure described. White solid, Yield 73%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (t, *J* = 7.3 Hz, 2H), 7.27 (d, *J* = 6.3 Hz, 1H), 7.14 (d, *J* = 7.1 Hz, 2H), 3.61 (dd, *J* = 12.1, 5.4 Hz, 1H), 2.58 – 2.43 (m, 2H), 2.28 (dd, *J* = 9.5, 5.8 Hz, 1H), 2.21 – 2.12 (m, 1H), 2.08 – 1.97 (m, 2H), 1.89 – 1.78 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.53, 138.87, 128.67, 128.50, 127.05, 57.54, 42.35, 35.25, 27.97, 25.49. Spectroscopic data matched with the literature.[3]



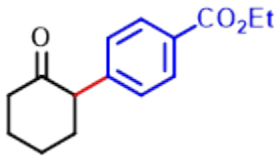
2-(naphthalen-2-yl) cyclohexan-1-one (3m): The compound is prepared according to the general procedure described. White solid, Yield 70%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 9.5 Hz, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.74 – 7.70 (m, 1H), 7.50 – 7.44 (m, 3H), 7.36 (d, *J* = 7.1 Hz, 1H), 4.37 (dd, *J* = 12.6, 5.3 Hz, 1H), 2.65 (q, *J* = 5.3, 4.9 Hz, 2H), 2.46 – 2.38 (m, 1H), 2.34 – 2.23 (m, 2H), 2.14 (d, *J* = 13.0 Hz, 1H), 2.02 – 1.90 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.22, 135.33, 133.95, 131.93, 129.16, 127.79, 126.03, 125.49, 123.40, 42.79, 28.06. Spectroscopic data matched with the literature.[4]



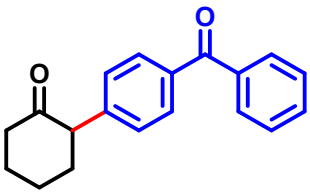
2-([1,1'-biphenyl]-4-yl) cyclohexan-1-one (3n): The compound is prepared according to the general procedure described. White solid, Yield 76%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59 (t, *J* = 8.1 Hz, 4H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 7.4 Hz, 1H), 7.23 (d, *J* = 8.2 Hz, 2H), 3.67 (dd, *J* = 12.3, 5.4 Hz, 1H), 2.61 – 2.47 (m, 2H), 2.37 – 2.28 (m, 1H), 2.18 (d, *J* = 5.7 Hz, 1H), 2.11 – 2.01 (m, 2H), 1.85 (t, *J* = 10.5 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.50, 141.13, 139.95, 137.95, 128.82, 127.24, 57.24, 42.37, 35.32, 29.84, 27.97, 25.52. Spectroscopic data matched with the literature.[6]



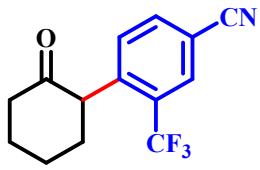
ethyl 4-(2-oxocyclohexyl) benzoate (3o): The compound is prepared according to the general procedure described. White solid, Yield 76% when X = I and 51% when X = Br

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.20 (d, *J* = 8.3 Hz, 2H), 4.35 (q, *J* = 7.1 Hz, 2H), 3.66 (dd, *J* = 12.3, 5.4 Hz, 1H), 2.57 – 2.42 (m, 2H), 2.27 (dd, *J* = 12.9, 5.4 Hz, 1H), 2.21 – 2.12 (m, 1H), 2.06 – 1.96 (m, 2H), 1.82 (t, *J* = 12.0 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.65, 166.58, 144.02, 129.65, 129.17, 128.71, 60.91, 57.48, 42.30, 35.13, 27.82, 25.37, 14.42. Spectroscopic data matched with the literature.[3]



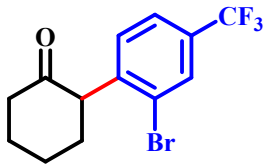
2-(4-benzoylphenyl) cyclohexan-1-one (3p): The compound is prepared according to the general procedure described. White solid, Yield 65%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 (t, *J* = 8.1 Hz, 4H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.26 (m, 2H), 3.72 (dd, *J* = 12.4, 5.4 Hz, 1H), 2.63 – 2.50 (m, 2H), 2.32 (d, *J* = 7.0 Hz, 1H), 2.21 (d, *J* = 9.1 Hz, 1H), 2.05 (t, *J* = 6.7 Hz, 2H), 1.86 (t, *J* = 11.6 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.74, 196.56, 143.82, 137.87, 136.26, 132.42, 130.38, 130.15, 128.76, 128.38, 57.61, 35.35, 27.91, 25.49. Spectroscopic data matched with the literature.[3]



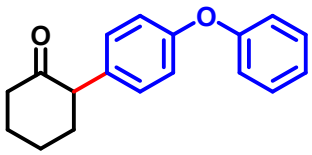
4-(2-oxocyclohexyl)-3-(trifluoromethyl)benzonitrile (3q): The compound is prepared according to the general procedure described. White solid, Yield 79%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 (s, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.48 (d, *J* = 8.2 Hz, 1H), 4.09 (dd, *J* = 12.2, 5.0 Hz, 1H), 2.61 – 2.48 (m, 2H), 2.32 – 2.21 (m, 2H), 1.95 (ddd, *J* = 49.6, 25.5, 10.0 Hz, 4H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.73, 143.34, 134.91, 132.02, 129.68, 129.62, 117.63, 111.37, 53.57, 42.29, 35.92, 27.87, 25.62. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -59.77. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{14}H_{13}F_3NO^+$ 268.0944, found 268.0947.



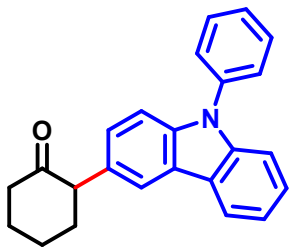
2-(2-bromo-4-(trifluoromethyl)phenyl)cyclohexan-1-one (3r): The compound is prepared according to the general procedure described. White solid, Yield 72%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (s, 1H), 7.58 (dd, *J* = 13.5, 7.1 Hz, 1H), 7.34 (t, *J* = 8.2 Hz, 1H), 4.15 (dd, *J* = 12.6, 5.2 Hz, 1H), 2.60 – 2.54 (m, 2H), 2.33 – 2.27 (m, 1H), 2.23 (d, *J* = 8.9 Hz, 1H), 2.08 (d, *J* = 14.7 Hz, 1H), 2.01 – 1.82 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.24, 142.65, 132.35, 130.86, 130.07, 129.74, 129.70, 129.22, 125.41, 124.38, 124.34, 56.70, 42.51, 34.33, 27.80, 25.68. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{13}H_{13}BrF_3O^+$ 321.0097, found 321.0092.



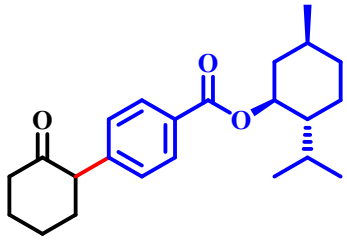
2-(4-phenoxyphenyl) cyclohexan-1-one (3s): The compound is prepared according to the general procedure described. White solid, Yield 68%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.30 (m, 2H), 7.10 (dd, *J* = 8.0, 3.4 Hz, 3H), 7.06 – 7.01 (m, 2H), 6.98 (s, 2H), 3.61 (dd, *J* = 12.1, 5.5 Hz, 1H), 2.59 – 2.42 (m, 2H), 2.29 (d, *J* = 9.0 Hz, 1H), 2.21 – 2.12 (m, 1H), 2.07 – 1.95 (m, 2H), 1.83 (t, *J* = 11.9 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 210.54, 157.25, 156.19, 133.64, 129.89, 129.81, 123.32, 119.13, 118.67, 56.83, 42.33, 35.52, 27.96, 25.52. Spectroscopic data matched with the literature.[6]



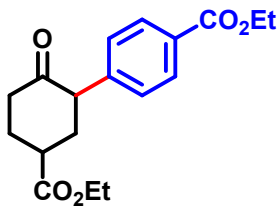
2-(9-phenyl-9H-carbazol-3-yl)cyclohexan-1-one (3t): The compound is prepared according to the general procedure described. White solid, Yield 62%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.17 (d, *J* = 7.7 Hz, 1H), 7.99 (s, 1H), 7.63 (t, *J* = 8.8 Hz, 4H), 7.53 – 7.43 (m, 4H), 7.36 – 7.30 (m, 1H), 7.26 (s, 1H), 3.88 (dd, *J* = 12.1, 5.2 Hz, 1H), 2.69 – 2.53 (m, 2H), 2.45 (d, *J* = 13.3 Hz, 1H), 2.31 – 2.20 (m, 2H), 2.11 (s, 1H), 1.95 (t, *J* = 9.7 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 211.24, 141.21, 140.09, 137.87, 130.49, 129.94, 127.45, 127.16, 126.73, 125.94, 123.54, 123.43, 120.38, 120.12, 119.90, 109.83, 109.76, 57.59, 42.39, 35.84, 28.03, 25.64. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{24}H_{22}NO^+$ 340.1696, found 340.1689.



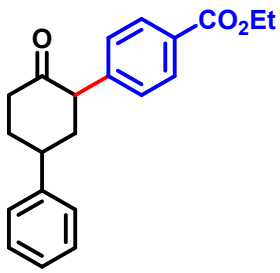
(1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 4-(2-oxocyclohexyl)benzoate (3u): The compound is prepared according to the general procedure described. White solid, Yield 73%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 4.92 (td, *J* = 10.8, 4.3 Hz, 1H), 3.67 (dd, *J* = 12.2, 5.4 Hz, 1H), 2.58 – 2.43 (m, 2H), 2.36 – 2.27 (m, 1H), 2.23 – 2.14 (m, 1H), 2.02 (ddd, *J* = 28.4, 22.6, 9.7 Hz, 4H), 1.88 – 1.79 (m, 2H), 1.72 (d, *J* = 11.4 Hz, 2H), 1.61 (s, 1H), 1.54 (dd, *J* = 16.1, 7.0 Hz, 2H), 1.17 – 1.03 (m, 2H), 0.92 (t, *J* = 6.1 Hz, 6H), 0.78 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.67, 166.08, 129.78, 129.66, 128.73, 74.81, 57.59, 47.43, 42.36, 41.10, 35.24, 35.13, 34.48, 31.58, 27.87, 26.56, 25.47, 23.75, 23.73, 22.19, 20.91, 16.60. Spectroscopic data matched with the literature.[3]



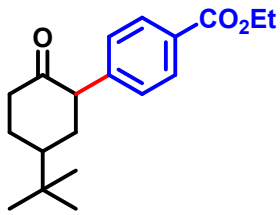
Ethyl 4-(5-(ethoxycarbonyl)-2-oxocyclohexyl) benzoate (5a): The compound is prepared according to the general procedure described. White solid, Yield 74%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.1 Hz, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.76 – 3.68 (m, 1H), 2.97 (t, *J* = 13.8 Hz, 1H), 2.66 – 2.41 (m, 5H), 2.27 – 2.13 (m, 1H), 2.04 (d, *J* = 34.0 Hz, 1H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 207.63, 173.75, 166.54, 142.96, 129.81, 129.59, 128.88, 61.03, 55.93, 42.46, 40.79, 36.99, 29.72, 14.47, 14.31. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{18}H_{23}O_5^+$ 319.1540, found 319.1547.



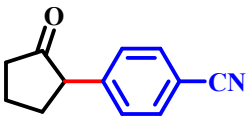
Ethyl 4-(2-oxo-5-phenylcyclohexyl) benzoate (5b): The compound is prepared according to the general procedure described. White solid, Yield 70%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 8.2 Hz, 2H), 7.30 (ddd, *J* = 23.5, 15.7, 7.9 Hz, 7H), 4.41 – 4.34 (m, 2H), 3.91 (dd, *J* = 13.3, 5.3 Hz, 1H), 2.78 – 2.63 (m, 2H), 2.42 (d, *J* = 8.3 Hz, 1H), 2.40 – 2.31 (m, 2H), 2.24 (d, *J* = 13.0 Hz, 1H), 2.11 (d, *J* = 4.9 Hz, 1H), 1.39 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 208.63, 166.48, 144.07, 129.63, 129.28, 128.83, 128.72, 126.82, 126.70, 60.87, 56.93, 43.47, 42.41, 41.89, 34.60, 14.38. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{21}H_{23}O_3^+$ 323.1642, found 323.1640.



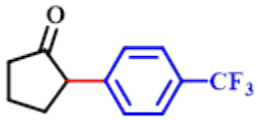
Ethyl 4-(5-(tert-butyl)-2-oxocyclohexyl) benzoate (5c): The compound is prepared according to the general procedure described. White solid, Yield 64%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.03 – 7.99 (m, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 4.36 (q, *J* = 7.1 Hz, 2H), 3.66 (d, *J* = 12.6 Hz, 1H), 2.54 (d, *J* = 4.3 Hz, 2H), 2.26 (t, *J* = 13.9 Hz, 2H), 1.80 – 1.70 (m, 2H), 1.67 – 1.56 (m, 1H), 1.37 (t, *J* = 7.1 Hz, 3H), 0.94 (s, 10H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 209.55, 166.28, 144.01, 129.37, 128.93, 128.58, 60.61, 56.67, 47.10, 41.44, 36.42, 32.36, 28.28, 27.43, 14.14. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{19}H_{27}O_3^+$ 303.1955, found 303.1951.



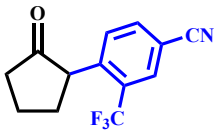
4-(2-oxocyclopentyl) benzonitrile (5d): The compound is prepared according to the general procedure described. White solid, Yield 62% from aryl iodide, and 43% from aryl bromide.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.3 Hz, 2H), 3.42 – 3.34 (m, 1H), 2.58 – 2.45 (m, 2H), 2.29 (ddd, *J* = 18.6, 10.6, 8.9 Hz, 1H), 2.19 (ddd, *J* = 12.5, 9.6, 5.3 Hz, 1H), 2.09 (td, *J* = 12.2, 11.8, 5.9 Hz, 1H), 2.01 – 1.88 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 216.33, 143.67, 132.28, 129.00, 118.85, 110.73, 55.21, 38.25, 31.14, 20.75. Spectroscopic data matched with the literature.[3]



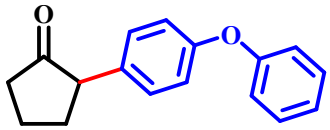
2-(4-(trifluoromethyl) phenyl) cyclopentan-1-one (5e): The compound is prepared according to the general procedure described. White solid, Yield 68%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 3.43 – 3.35 (m, 1H), 2.52 (t, *J* = 9.9 Hz, 2H), 2.37 – 2.26 (m, 1H), 2.22 – 2.10 (m, 2H), 2.02 – 1.92 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 217.16, 142.41, 128.64, 125.66,122.95, 55.21, 38.43, 20.92. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.53. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.5 Spectroscopic data matched with the literature.[3]



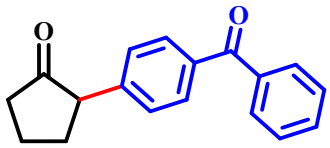
4-(2-oxocyclopentyl)-3-(trifluoromethyl) benzonitrile (5f): The compound is prepared according to the general procedure described. White solid, Yield 73%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (s, 1H), 7.80 (d, *J* = 8.1 Hz, 1H), 7.27 (d, *J* = 4.6 Hz, 1H), 3.76 (dd, *J* = 11.9, 8.2 Hz, 1H), 2.60 (dd, *J* = 18.8, 8.5 Hz, 2H), 2.34 (d, *J* = 28.2 Hz, 1H), 2.27 – 2.20 (m, 1H), 2.07 – 1.93 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 216.22, 144.27, 144.26, 135.51, 131.15, 129.86, 129.80, 117.48, 111.40, 53.11, 38.49, 34.36, 20.96. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{13}H_{11}F_3NO^+$ 254.0788, found 254.0782.



2-(4-phenoxyphenyl) cyclopentan-1-one (5g): The compound is prepared according to the general procedure described. White solid, Yield 66%.

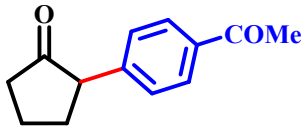
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 (t, *J* = 7.9 Hz, 2H), 7.16 (d, *J* = 8.6 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H), 6.99 (dd, *J* = 12.9, 8.5 Hz, 4H), 3.35 – 3.27 (m, 1H), 2.49 (dd, *J* = 18.0, 9.3 Hz, 2H), 2.35 – 2.24 (m, 1H), 2.20 – 2.06 (m, 2H), 2.00 – 1.90 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 157.32, 156.25, 133.26, 129.85, 129.53, 123.35, 54.82, 38.48, 31.95. HRMS (EI) m/z: $[M+H]^+$ calcd for $C_{17}H_{17}O_2^+$ 253.1224, found 253.1228.



2-(4-benzoylphenyl) cyclopentan-1-one (5h): The compound is prepared according to the general procedure described. White solid, Yield 58%.

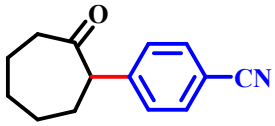
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 (ddd, *J* = 6.7, 4.1, 1.5 Hz, 4H), 7.61 – 7.56 (m, 1H), 7.48 (dd, *J* = 8.3, 7.0 Hz, 2H), 7.34 – 7.30 (m, 2H), 3.43 (dd, *J* = 11.3, 8.4 Hz, 1H), 2.58 – 2.49 (m, 2H), 2.38 – 2.29 (m, 1H), 2.17 (dt, *J* = 16.8, 6.1 Hz, 2H), 2.07 – 1.95 (m, 2H ^{13}C NMR (101

MHz, Chloroform-*d*) δ 217.68, 196.82, 143.63, 138.08, 136.59, 132.82, 130.89, 130.47, 128.72, 128.57, 55.79, 38.88, 32.02.). Spectroscopic data matched with the literature.[5]



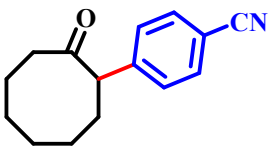
2-(4-acetylphenyl) cyclopentan-1-one (5i): The compound is prepared according to the general procedure described. White solid, Yield 61% from aryl iodide, and 40% from aryl bromide.

^{1}H NMR (400 MHz, Chloroform-d) δ 7.95 (d, J = 8.4 Hz, 2H), 7.31 (d, J = 8.2 Hz, 2H), 3.46 – 3.37 (m, 1H), 2.60 (s, 3H), 2.59 – 2.52 (m, 1H), 2.55 – 2.47 (m, 1H), 2.34 (d, J = 8.6 Hz, 1H), 2.31 – 2.16 (m, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 217.48, 198.23, 144.27, 136.21, 129.06, 128.83, 38.79, 31.85, 27.07, 21.28. Spectroscopic data matched with the literature.[9]



4-(2-oxocycloheptyl) benzonitrile (5j): The compound is prepared according to the general procedure described along with slight modification. The reaction mixture was heated to 60 ^{0}C while being irradiated with two blue LED. White solid, Yield 55%.

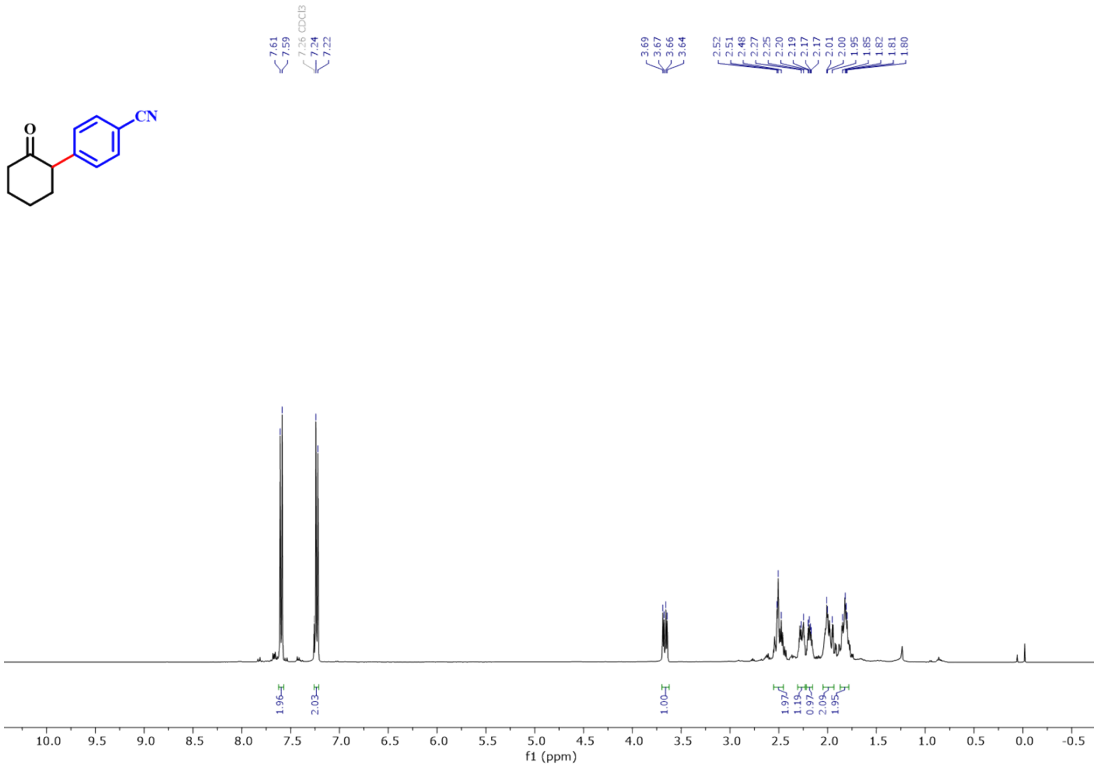
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.59 (d, *J* = 7.9 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 3.82 (dd, *J* = 11.1, 3.3 Hz, 1H), 2.60 (d, *J* = 10.2 Hz, 2H), 2.11 – 1.87 (m, 5H), 1.76 – 1.62 (m, 1H), 1.48 (dt, *J* = 32.1, 10.7 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 212.02, 146.04, 132.24, 129.05, 118.94, 110.75, 58.35, 43.37, 32.31, 29.58, 29.07, 24.61. Spectroscopic data matched with the literature.[3]



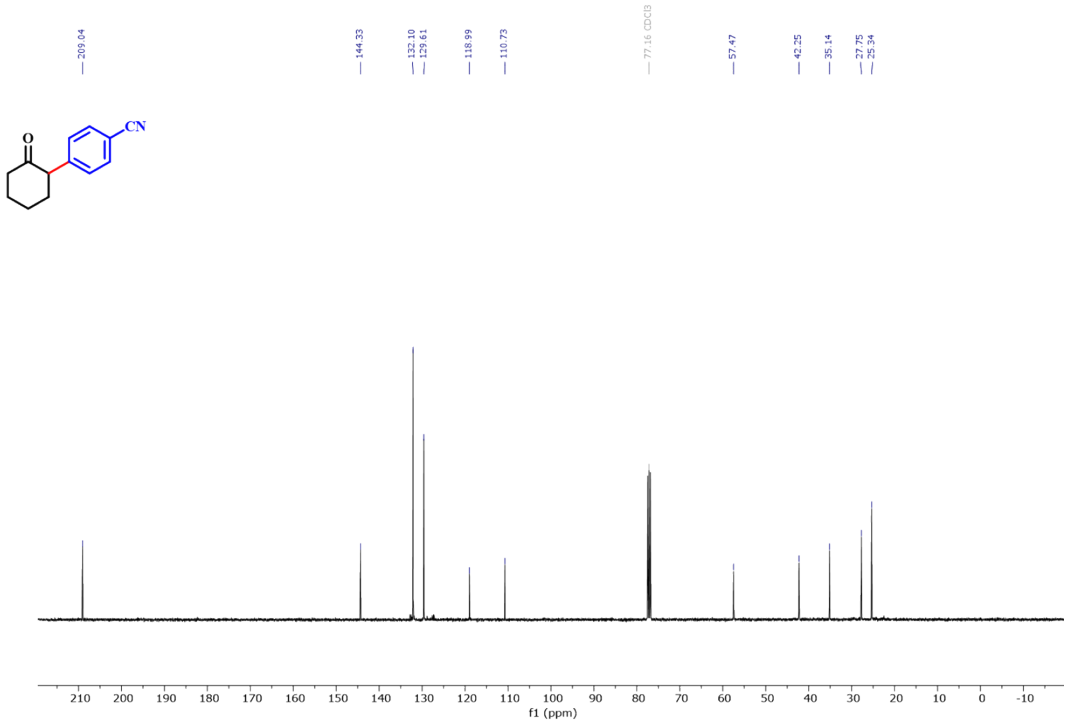
4-(2-oxocyclooctyl) benzonitrile (5k): The compound is prepared according to the general procedure described along with slight modification. The reaction mixture was heated to 60 ^{0}C while being irradiated with two blue LED. White solid, Yield 47%.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.3 Hz, 2H), 3.93 (dd, *J* = 12.0, 3.0 Hz, 1H), 2.52 (td, *J* = 12.7, 12.1, 3.6 Hz, 1H), 2.41 – 2.32 (m, 1H), 2.22 (td, *J* = 11.6, 3.6 Hz, 1H), 2.10 – 1.87 (m, 3H), 1.84 – 1.68 (m, 2H), 1.62 – 1.46 (m, 5H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 215.32, 145.09, 132.36, 128.96, 118.95, 111.02, 56.66, 41.80, 33.41, 27.32, 26.23, 25.82, 24.69. Spectroscopic data matched with the literature.[3]

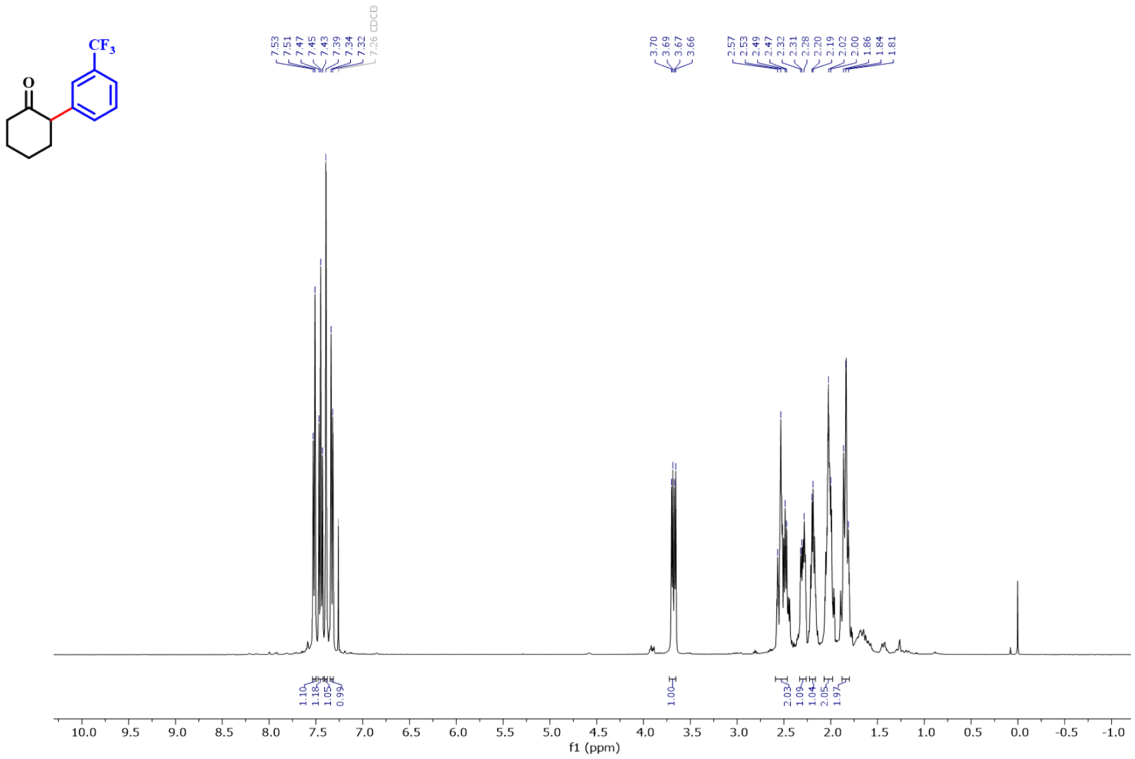
7. ^{1}H and ^{13}C NMR Spectra



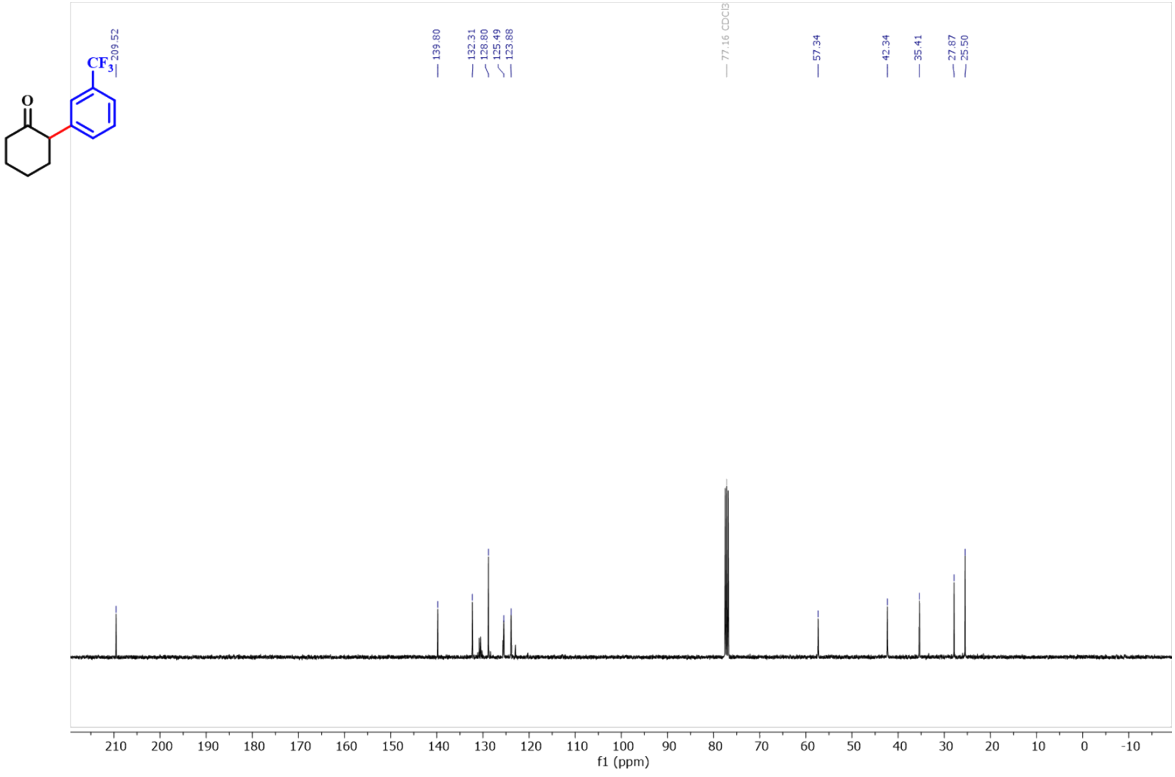
^{1}H NMR spectrum (400 MHz) of **3a** in $CDCl_3$



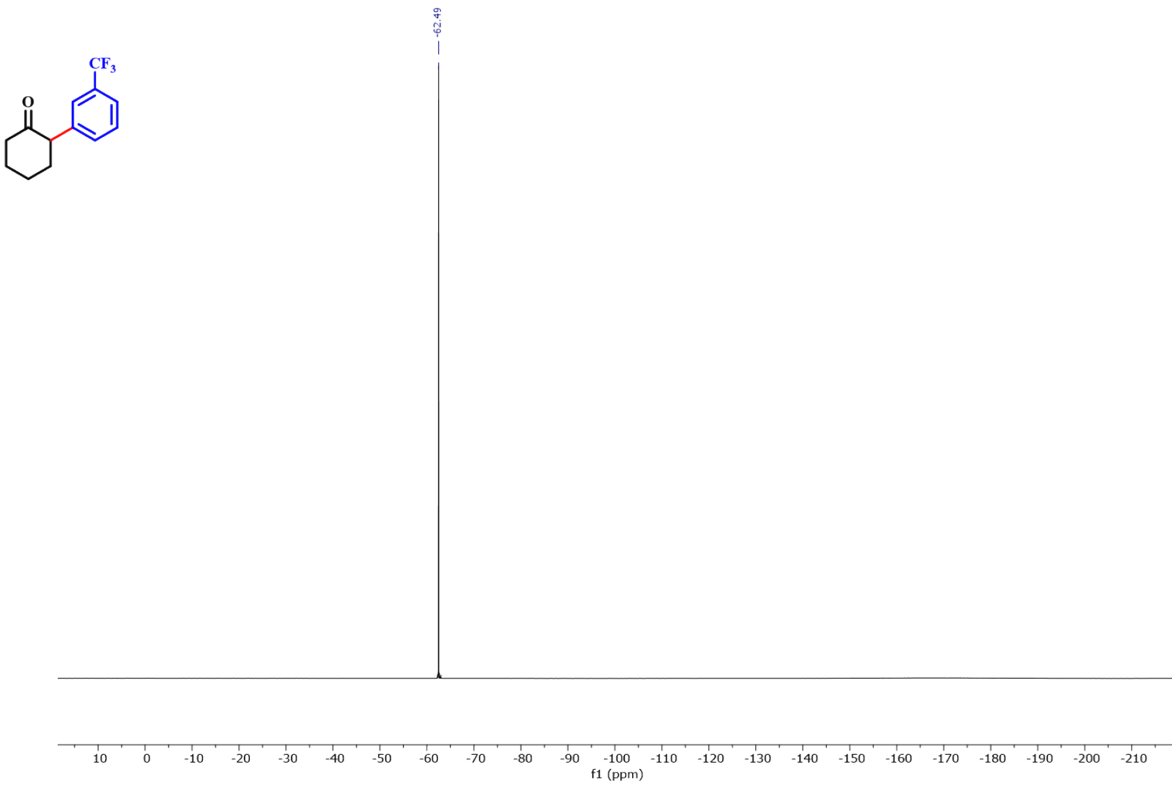
^{13}C NMR spectrum (101 MHz) of **3a** in $CDCl_3$



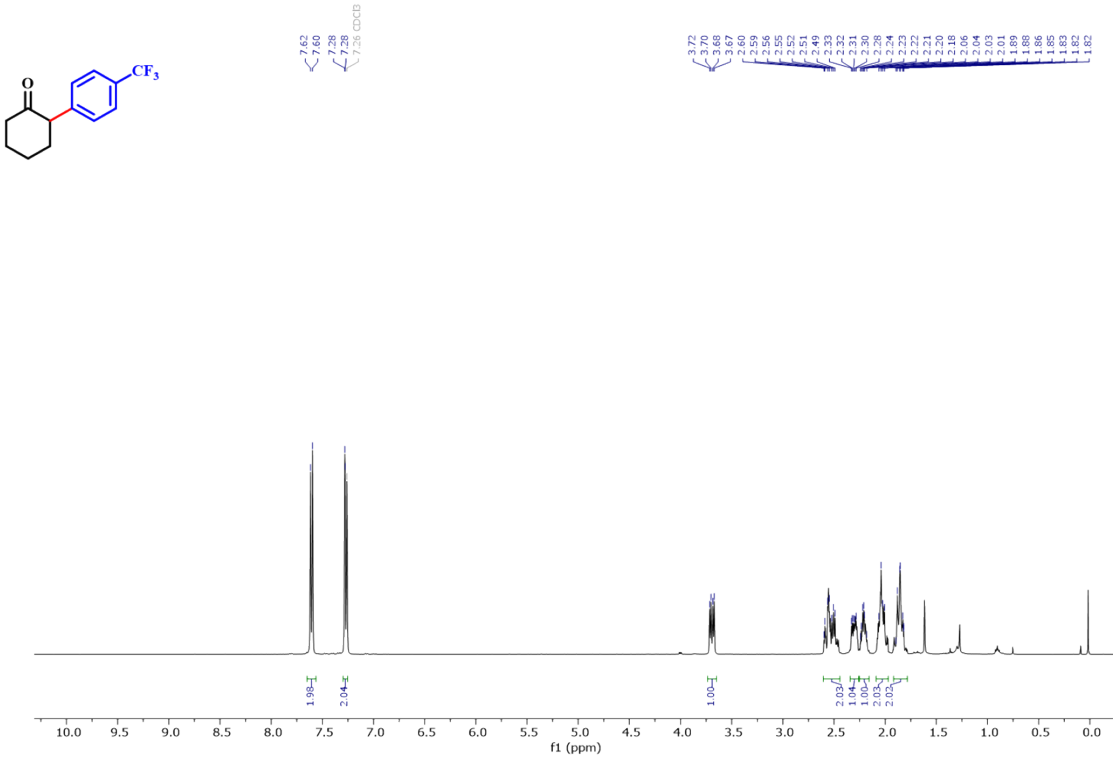
^{1}H NMR spectrum (400 MHz) of **3b** in $CDCl_3$



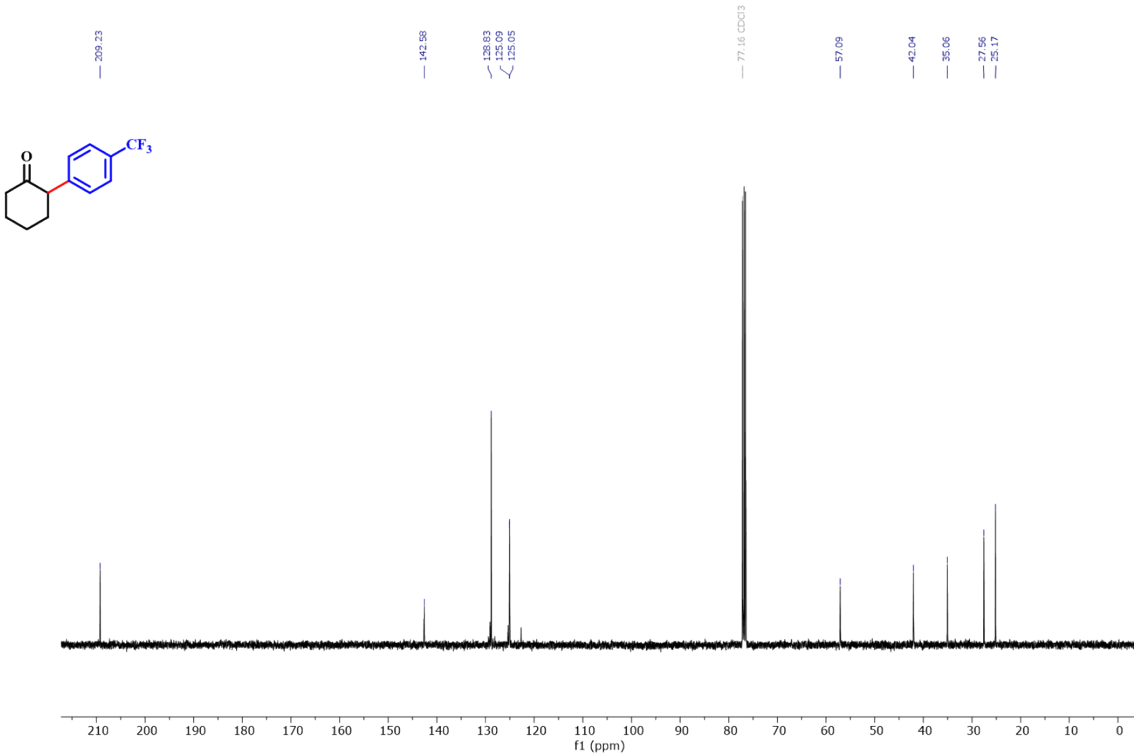
^{13}C NMR spectrum (101 MHz) of **3b** in $CDCl_3$



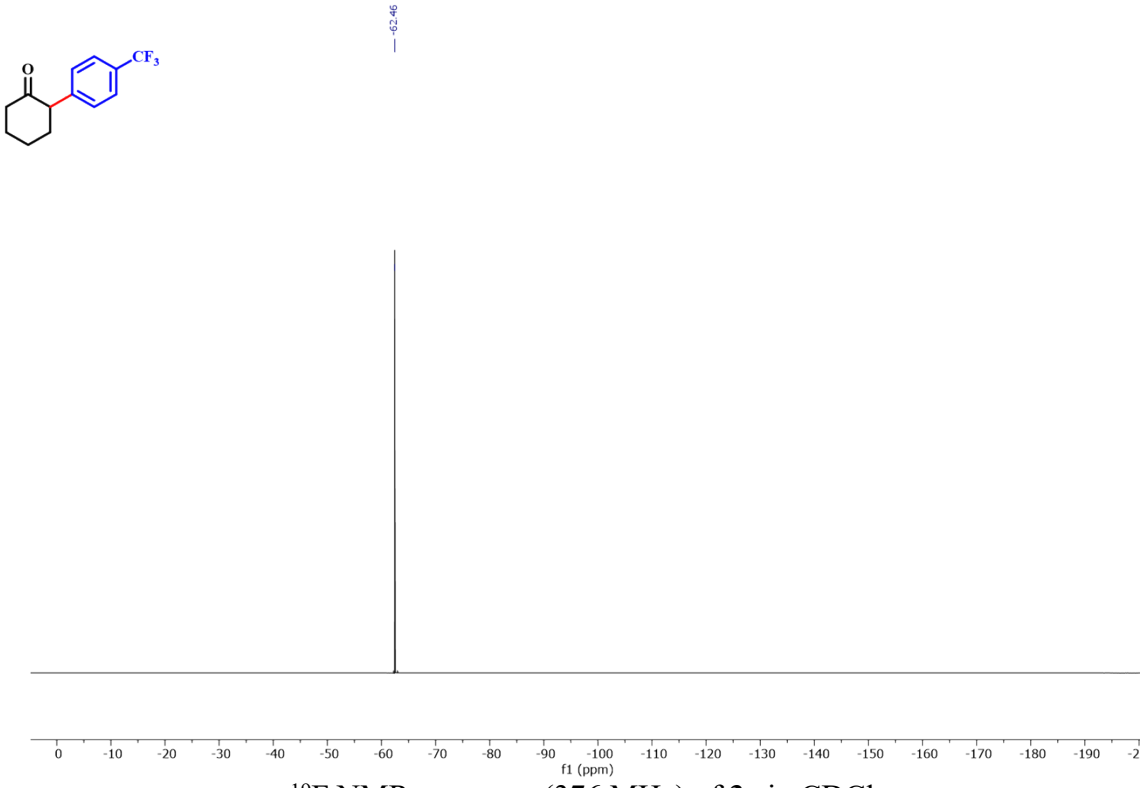
^{19}F NMR spectrum (376 MHz) of **3b** in $CDCl_3$



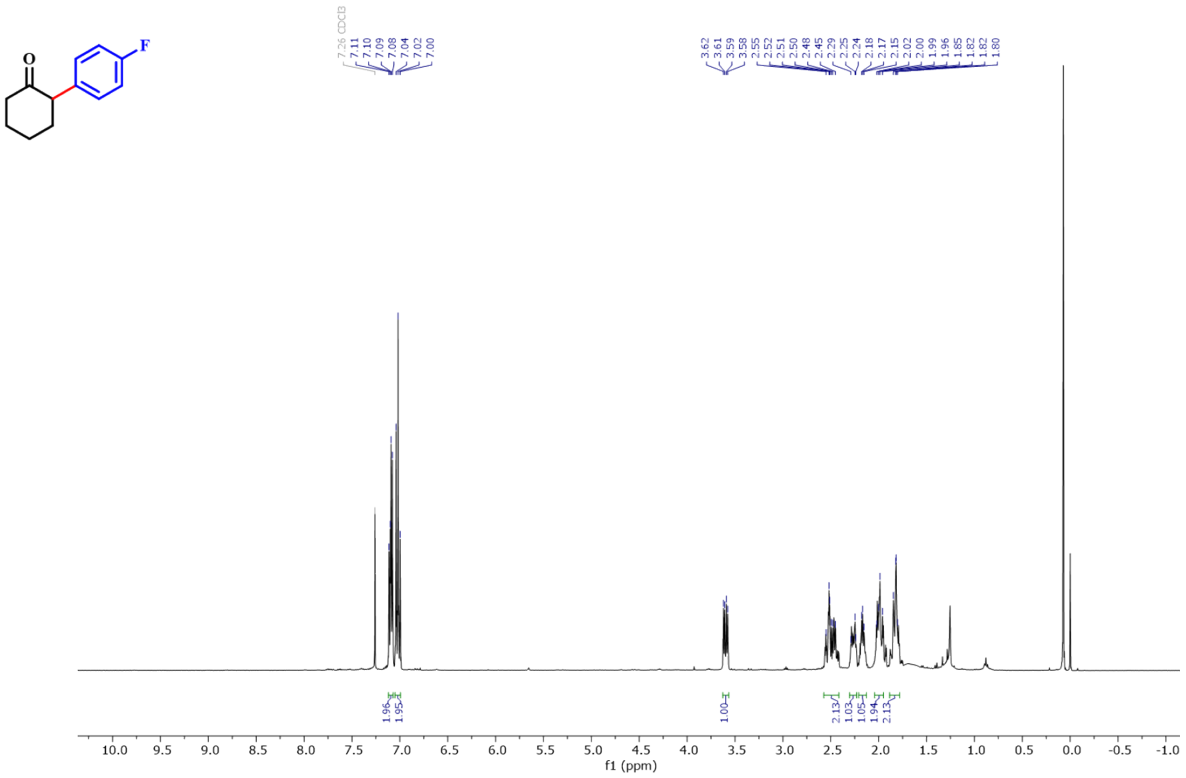
^{1}H NMR spectrum (400 MHz) of **3c** in $CDCl_3$



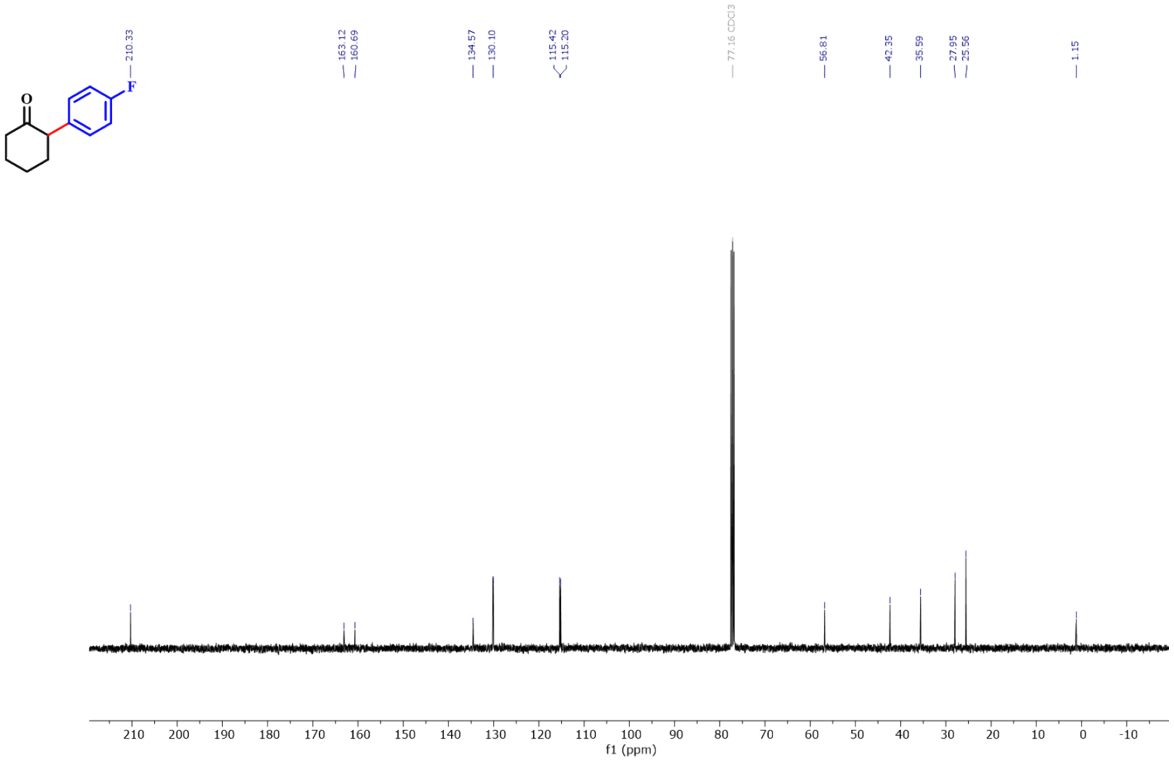
^{13}C NMR spectrum (101 MHz) of **3c** in $CDCl_3$



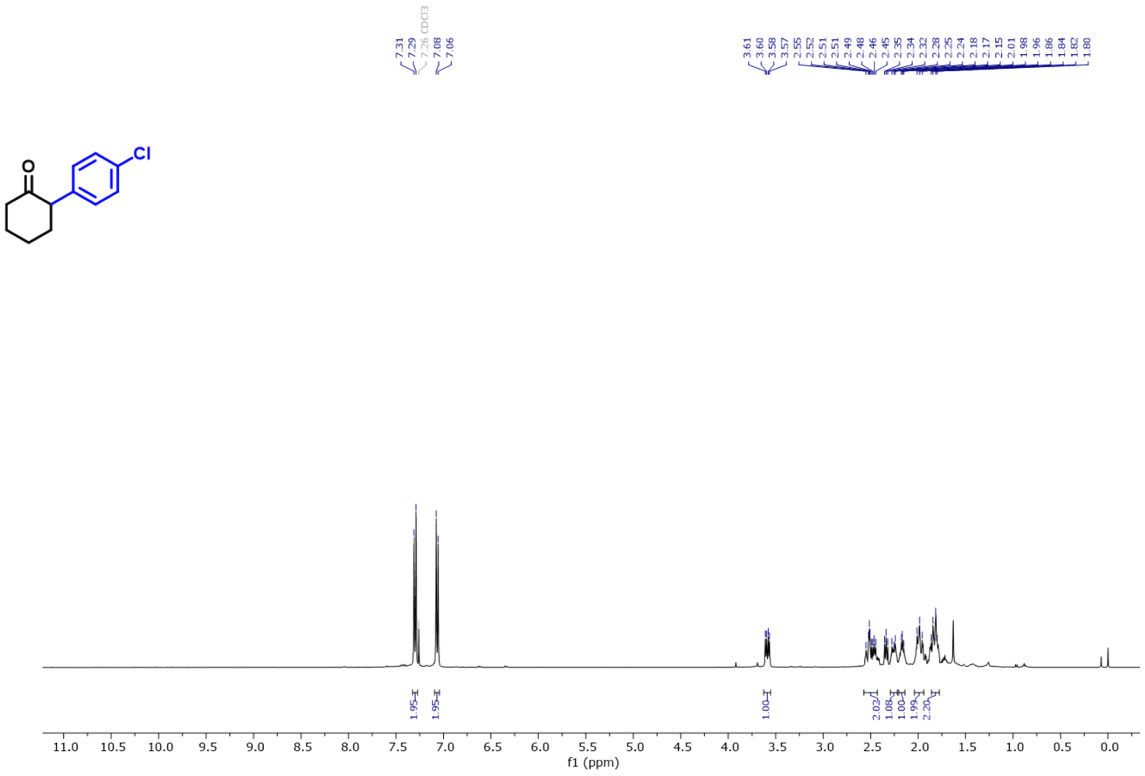
^{19}F NMR spectrum (376 MHz) of **3c** in $CDCl_3$



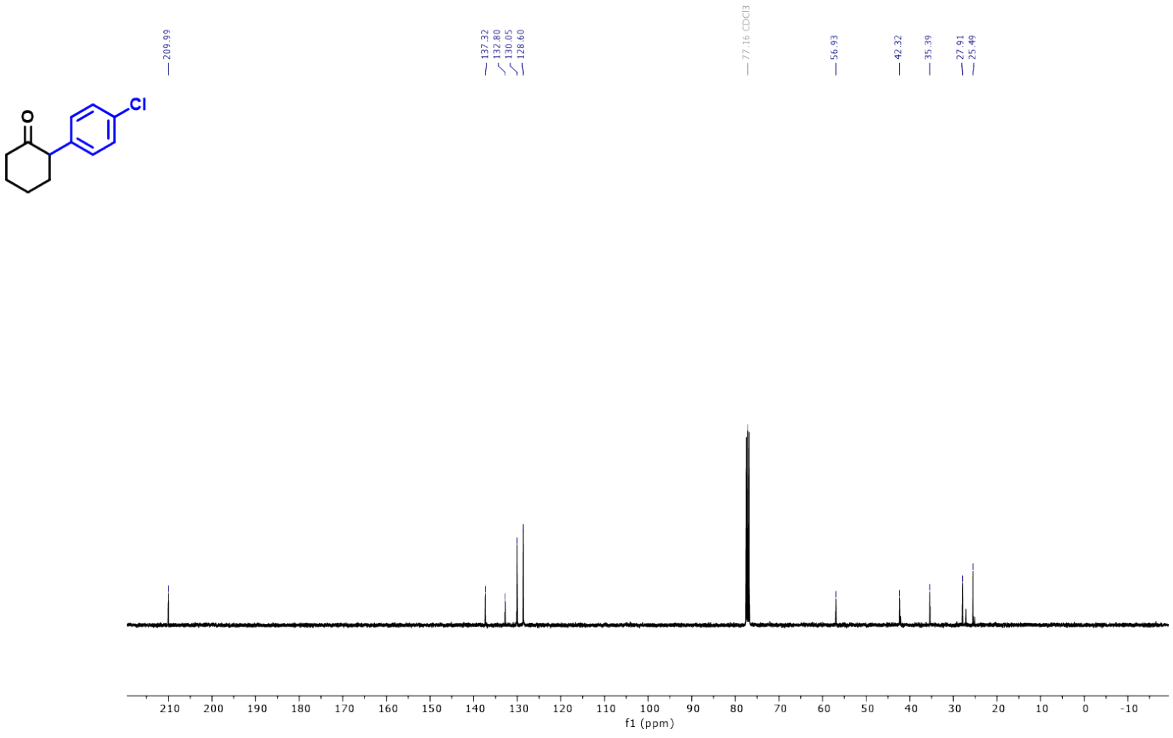
^{1}H NMR spectrum (400 MHz) of **3d** in $CDCl_3$



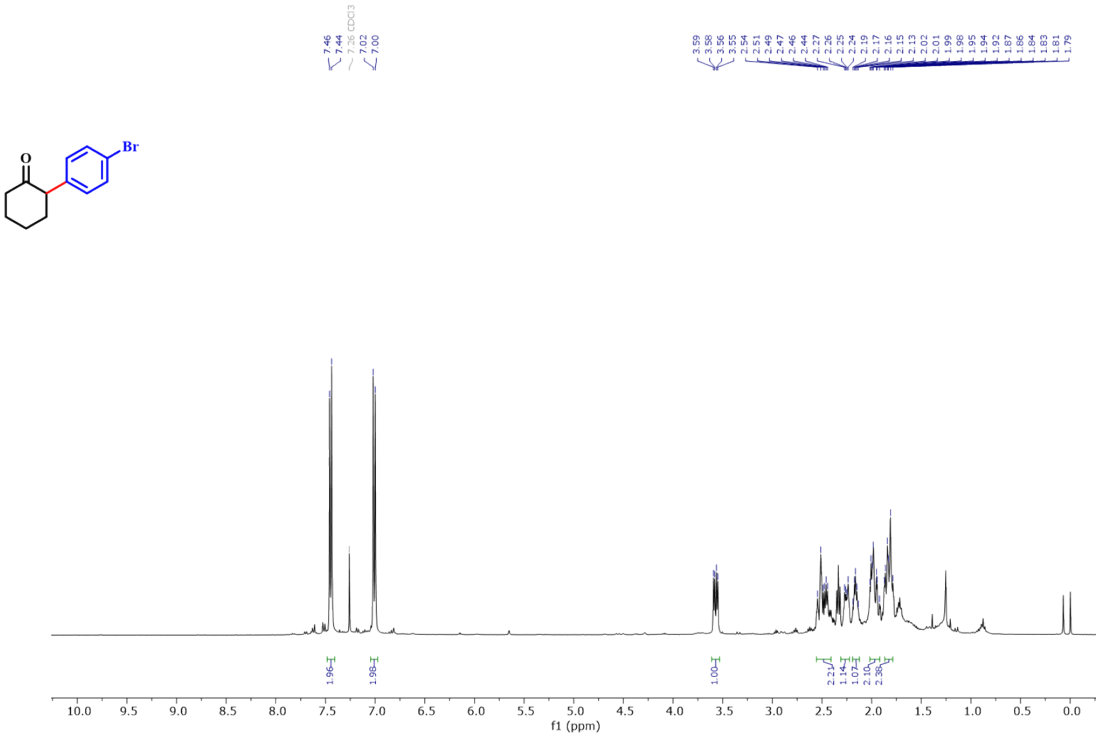
^{13}C NMR spectrum (101 MHz) of **3d** in $CDCl_3$



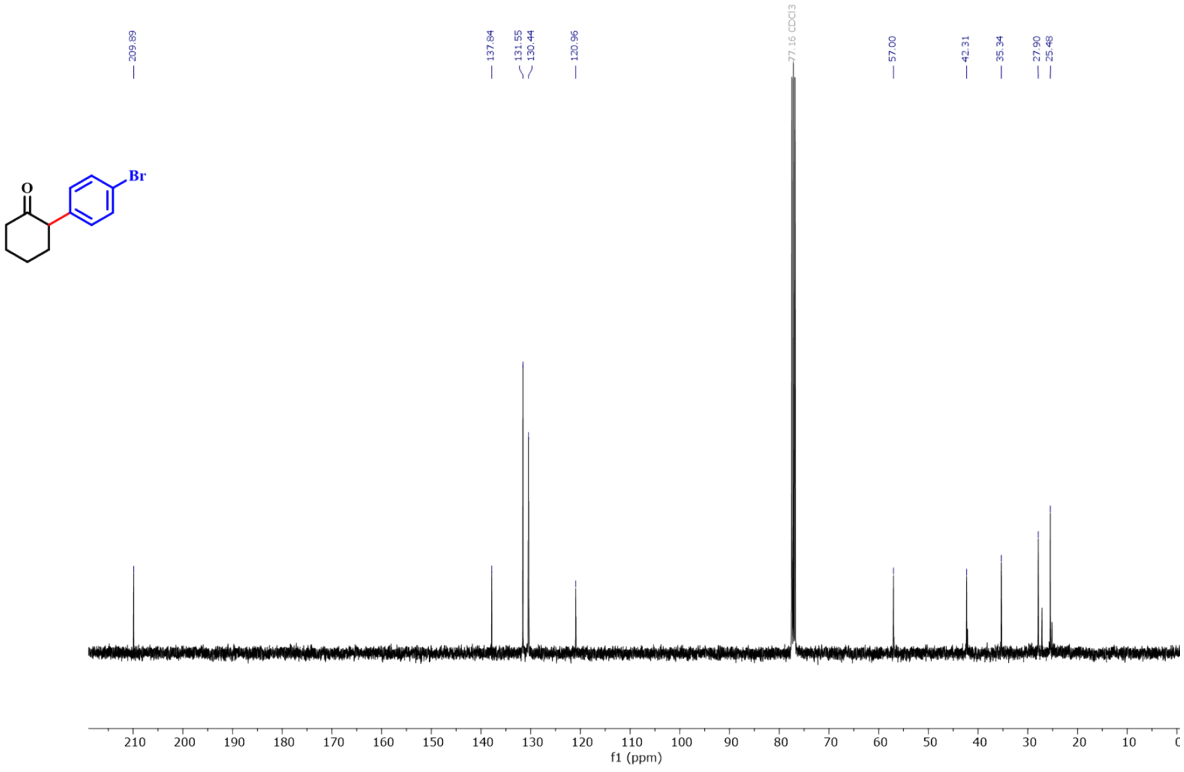
^{1}H NMR spectrum (400 MHz) of **3e** in $CDCl_3$



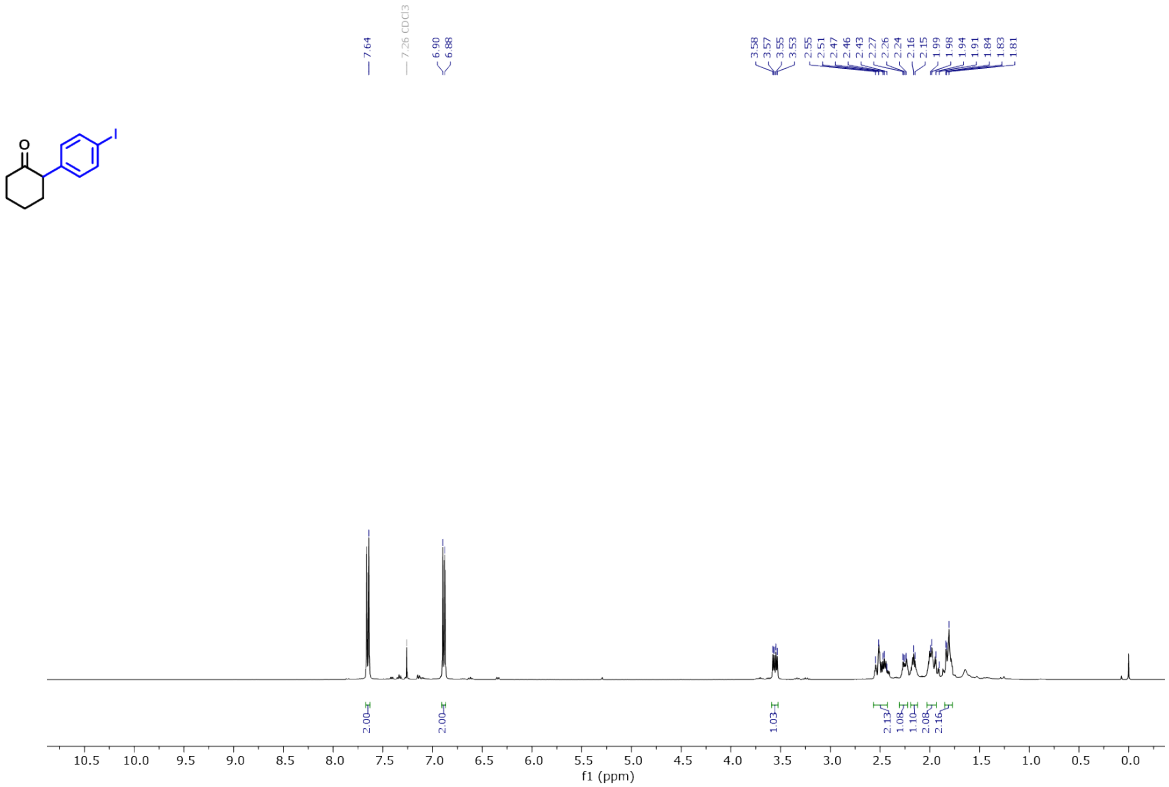
^{13}C NMR spectrum (101 MHz) of **3e** in $CDCl_3$



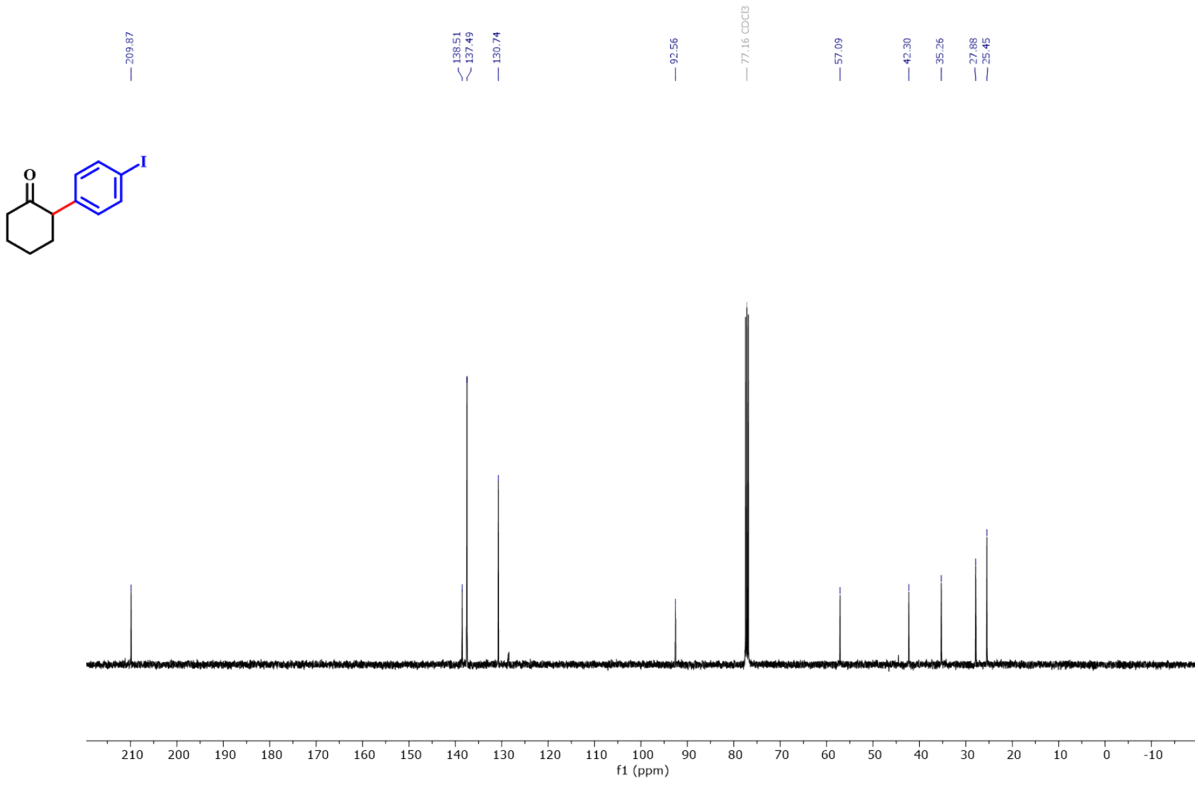
^{1}H NMR spectrum (400 MHz) of **3f** in $CDCl_3$



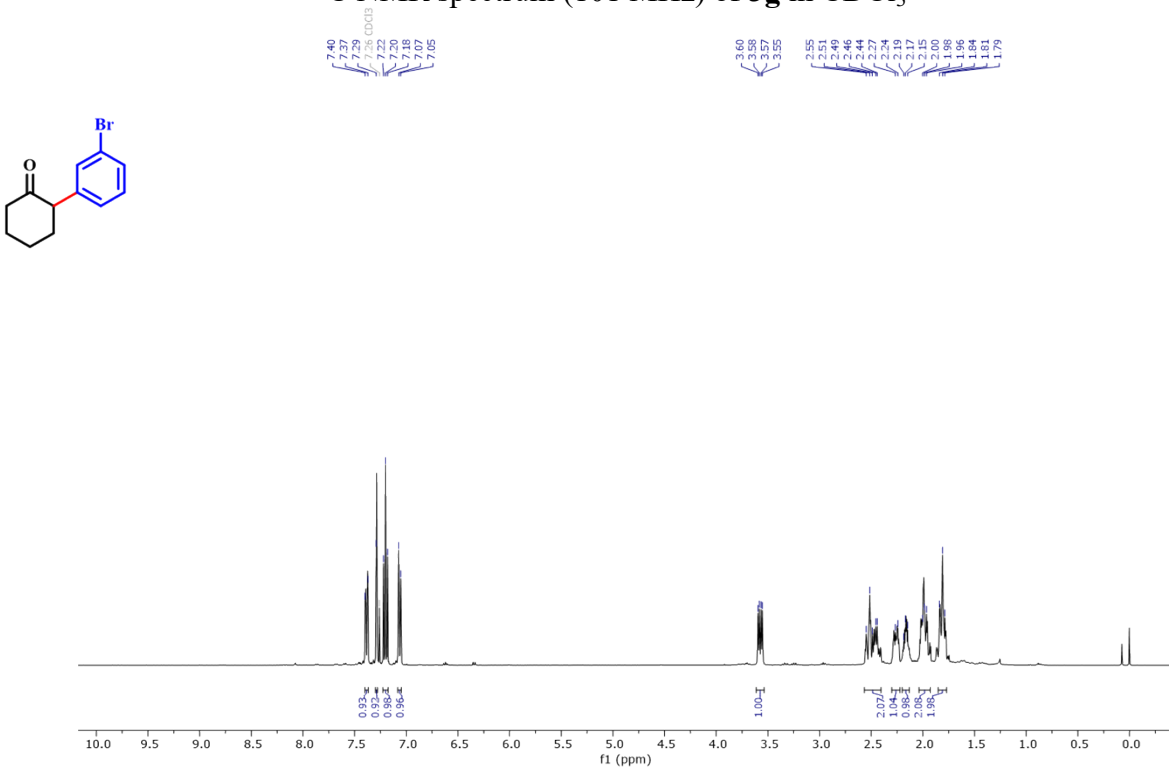
^{13}C NMR spectrum (101 MHz) of **3f** in $CDCl_3$



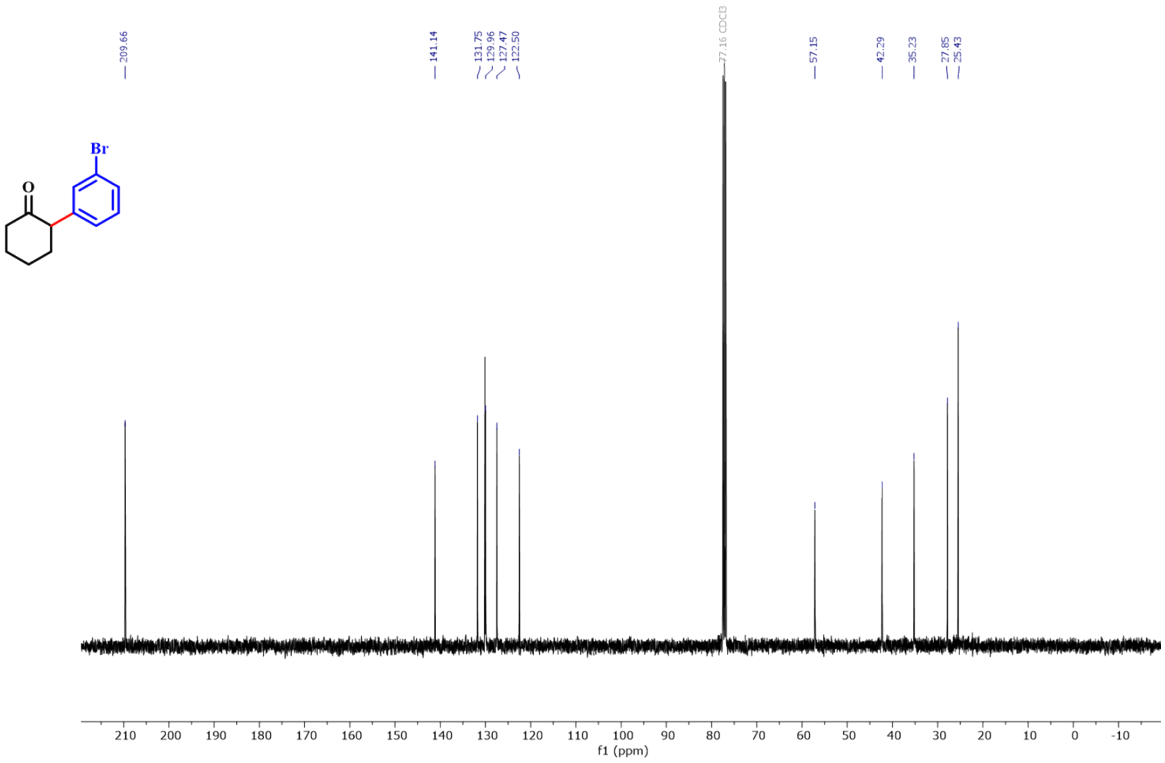
^{1}H NMR spectrum (400 MHz) of **3g** in $CDCl_3$



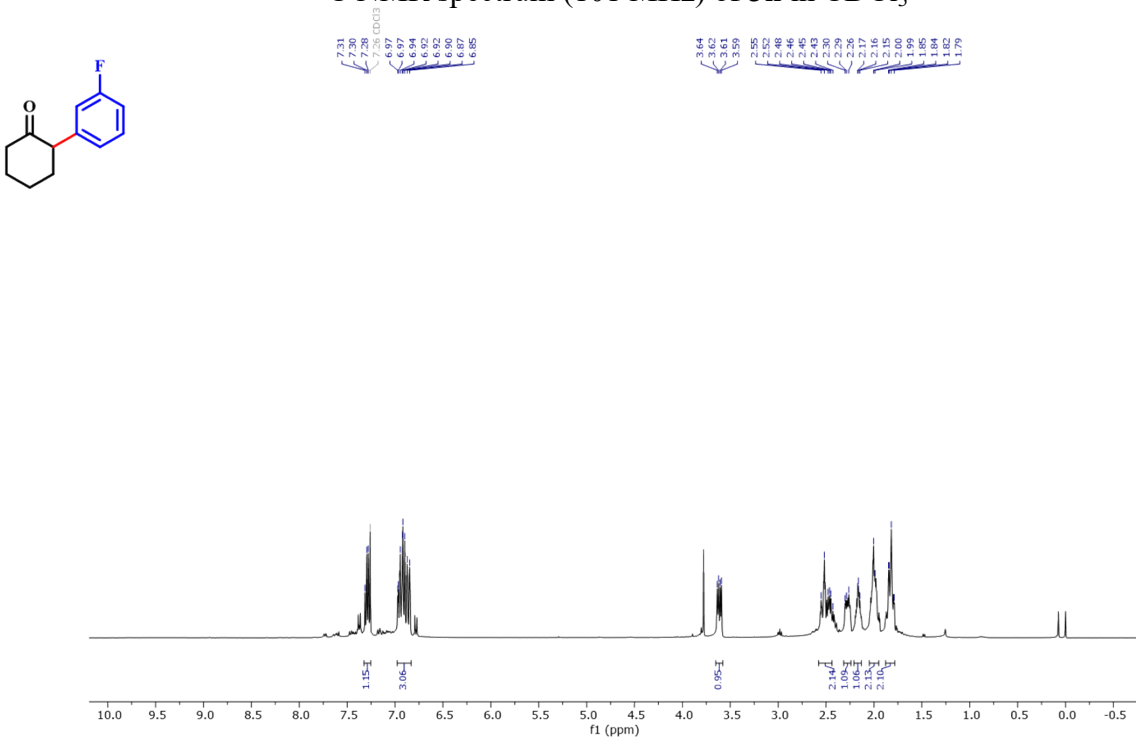
^{13}C NMR spectrum (101 MHz) of **3g** in $CDCl_3$



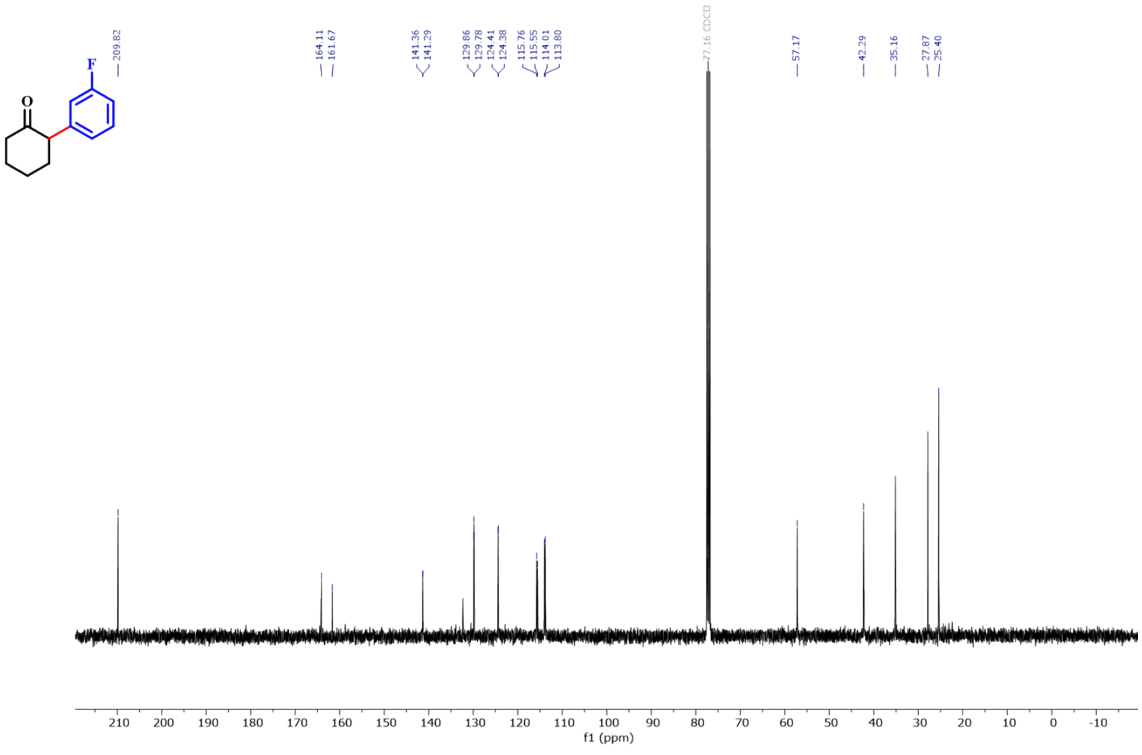
^{1}H NMR spectrum (400 MHz) of **3h** in $CDCl_3$



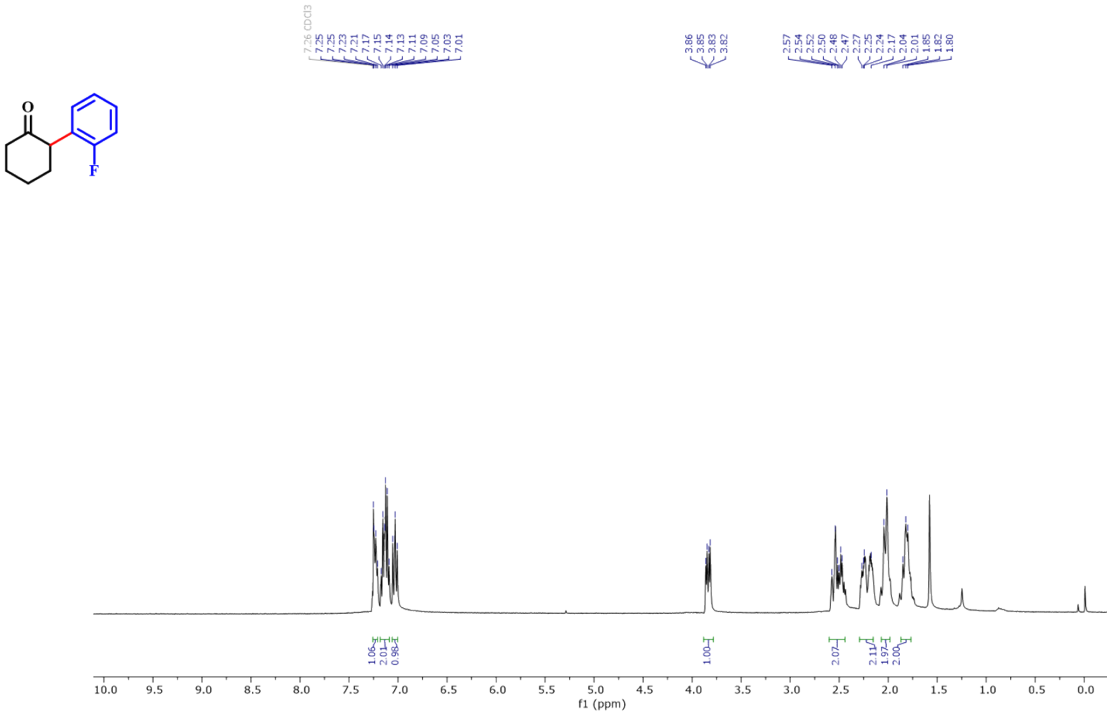
^{13}C NMR spectrum (101 MHz) of **3h** in $CDCl_3$



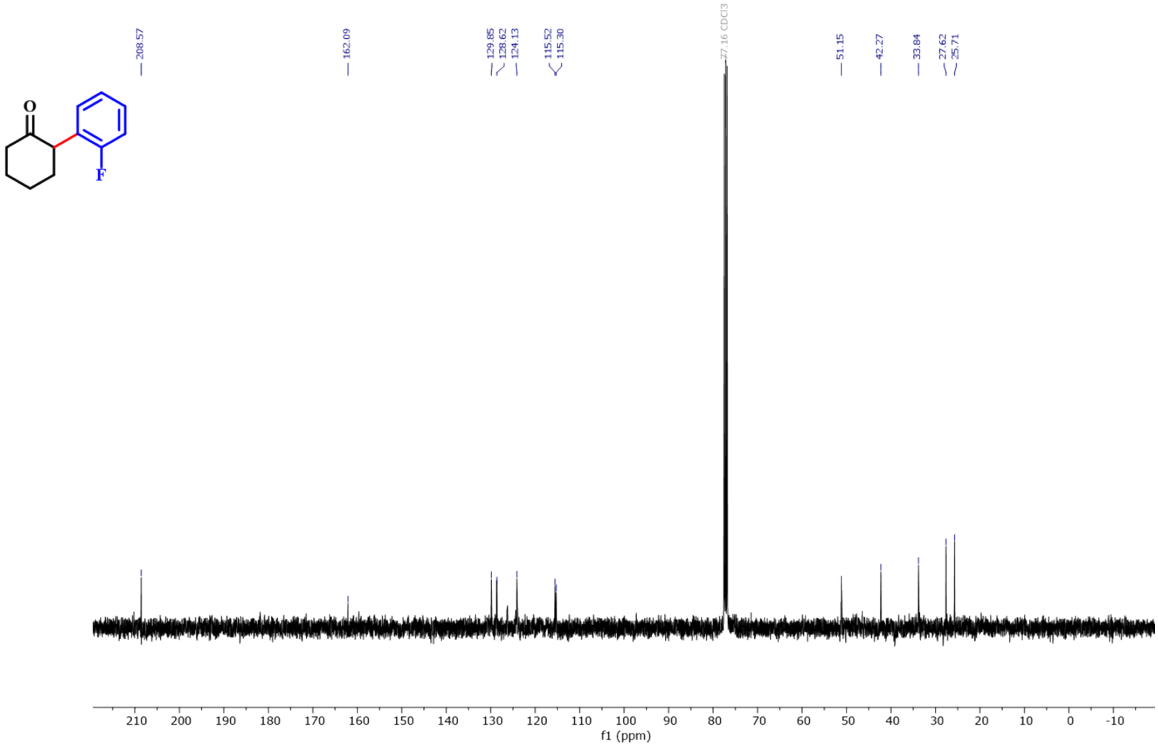
^{1}H NMR spectrum (400 MHz) of **3i** in $CDCl_3$



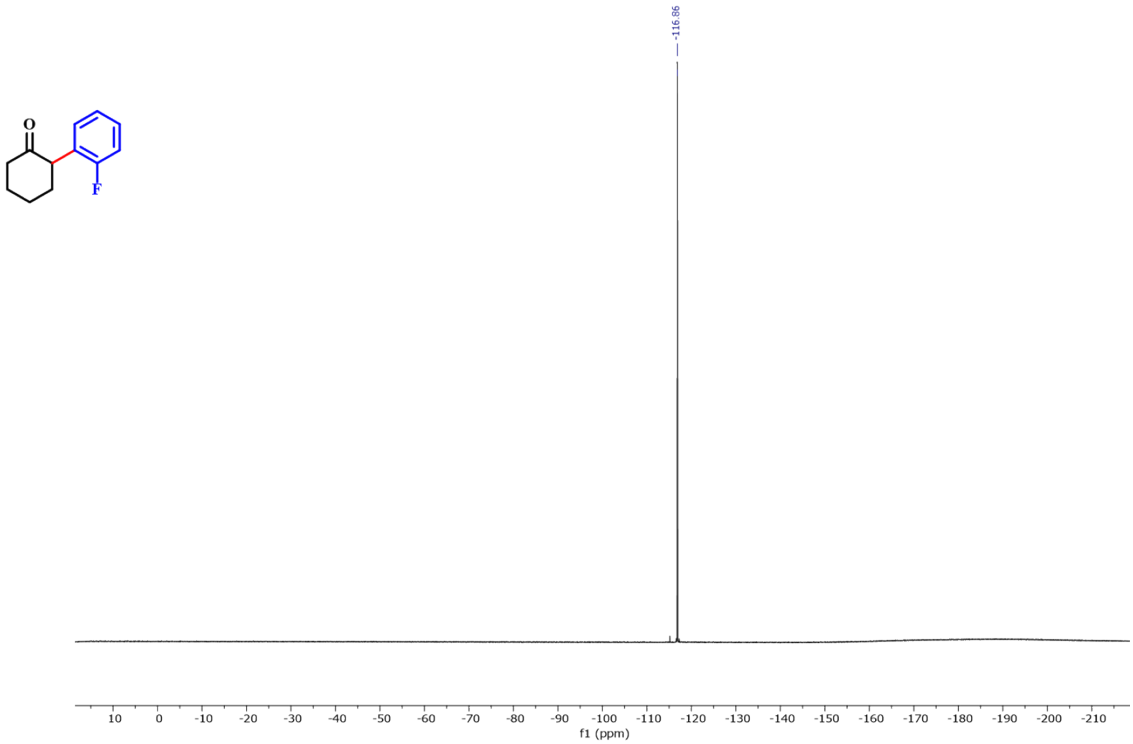
^{13}C NMR spectrum (101 MHz) of **3i** in $CDCl_3$



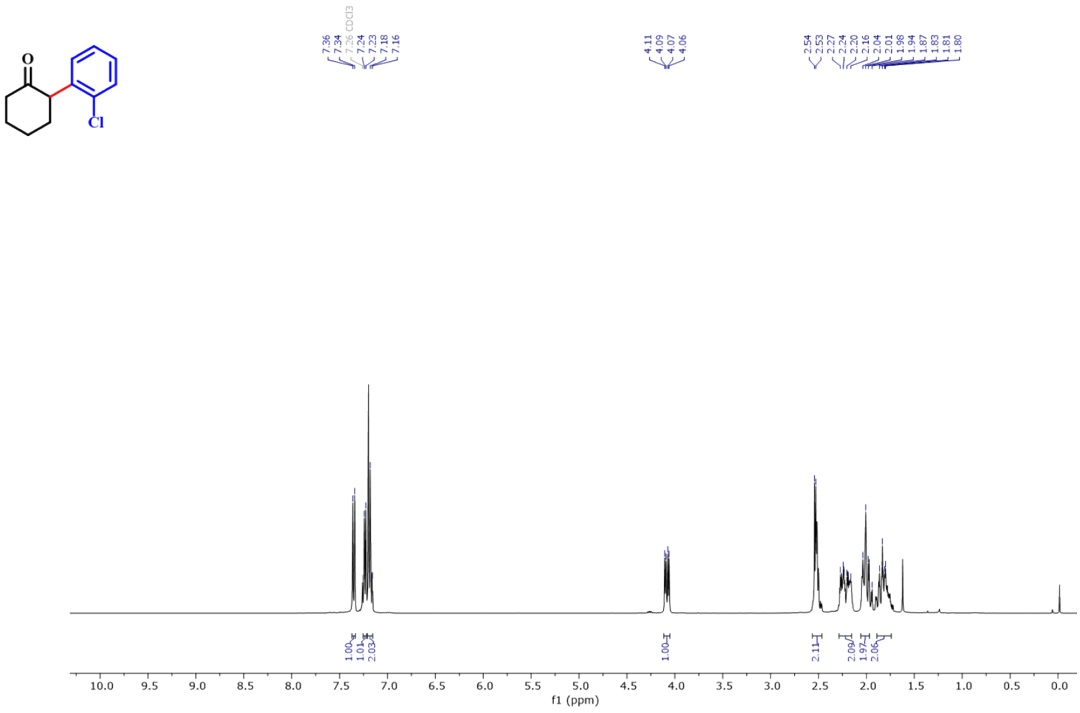
^{1}H NMR spectrum (400 MHz) of **3j** in $CDCl_3$



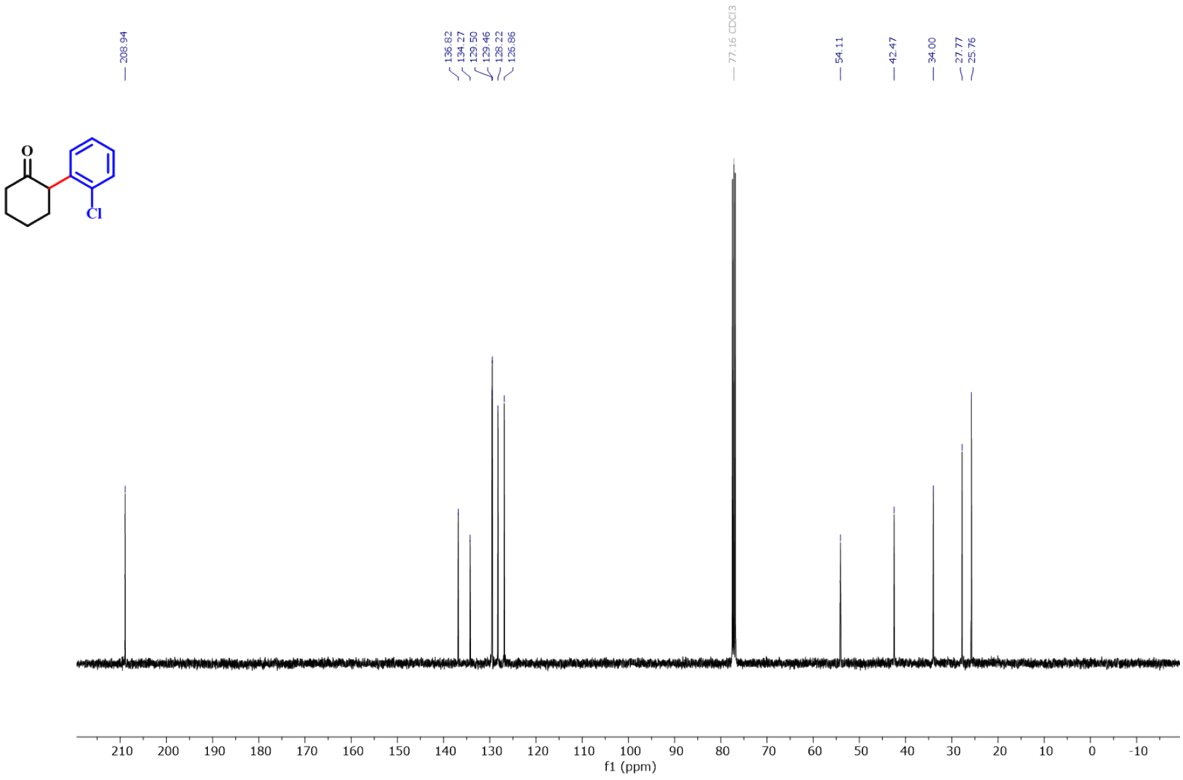
^{13}C NMR spectrum (101 MHz) of **3j** in $CDCl_3$



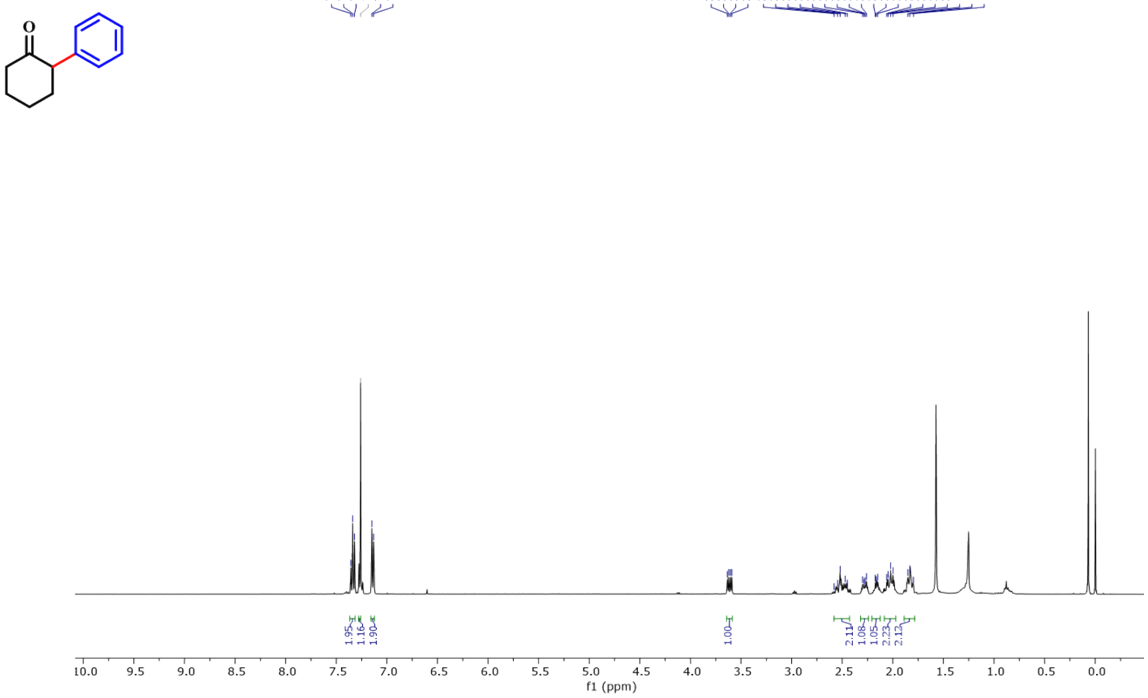
^{19}F NMR spectrum (376 MHz) of **3j** in $CDCl_3$



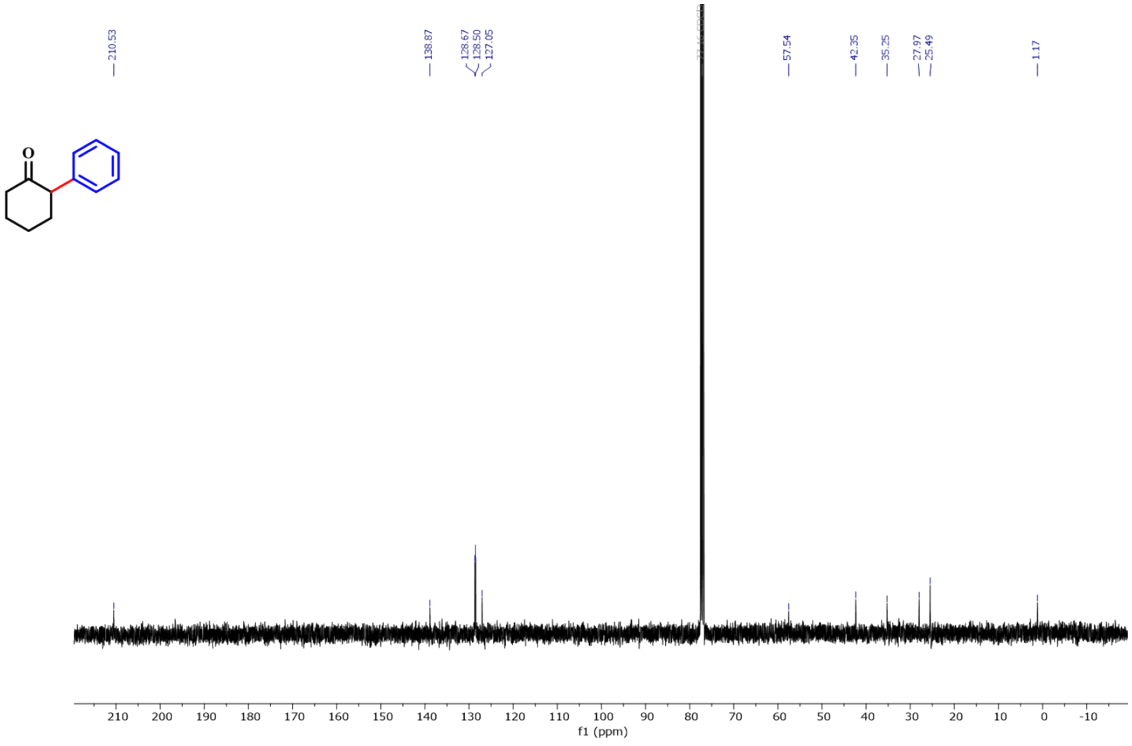
^{1}H NMR spectrum (400 MHz) of **3k** in $CDCl_3$



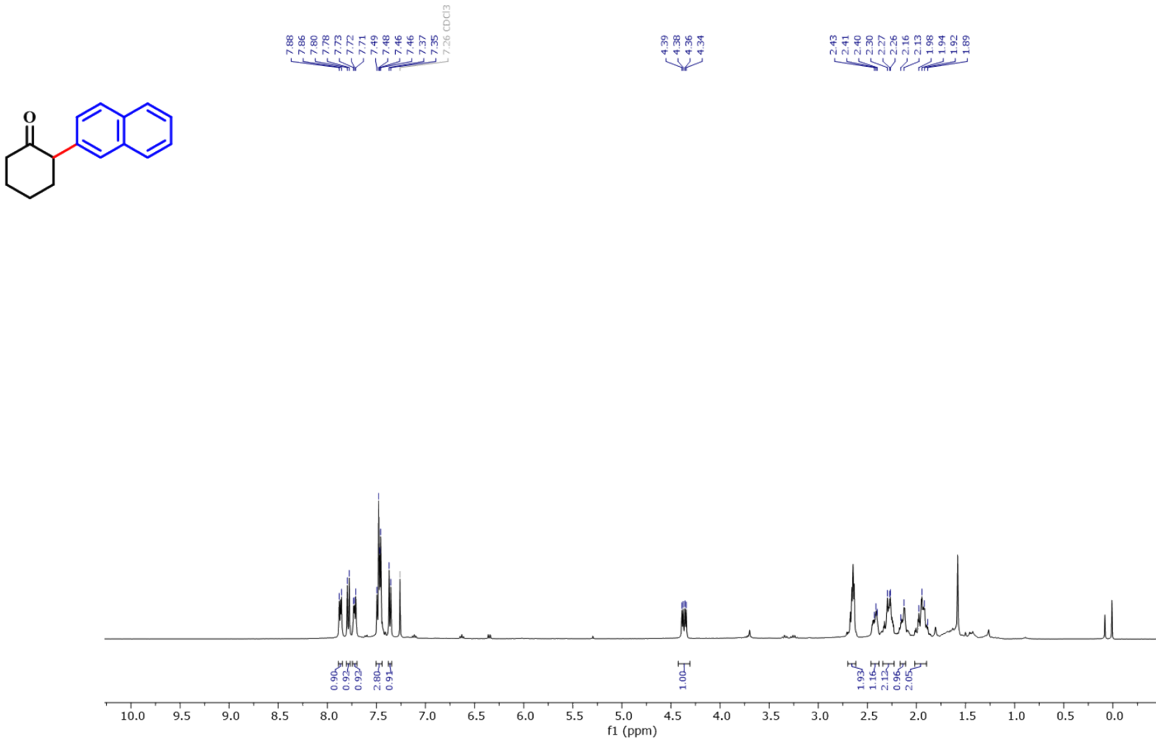
^{13}C NMR spectrum (101 MHz) of **3k** in $CDCl_3$



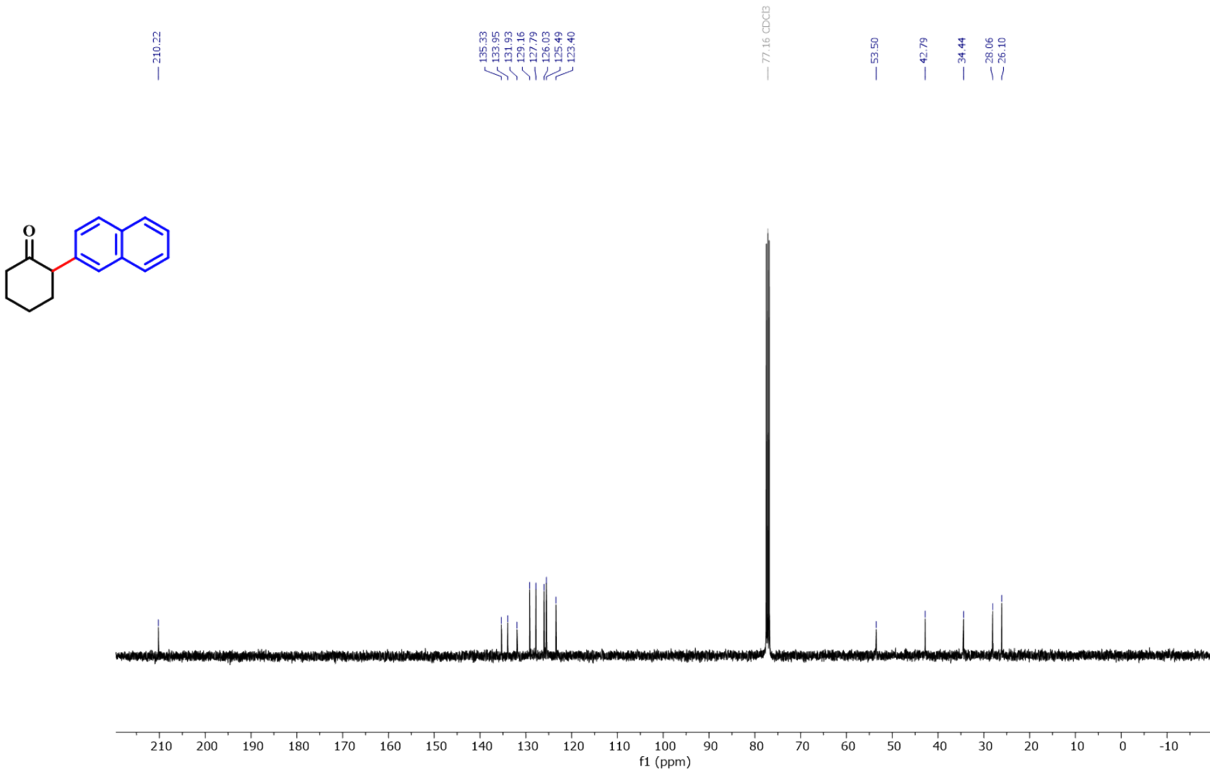
^{1}H NMR spectrum (400 MHz) of **3l** in $CDCl_3$



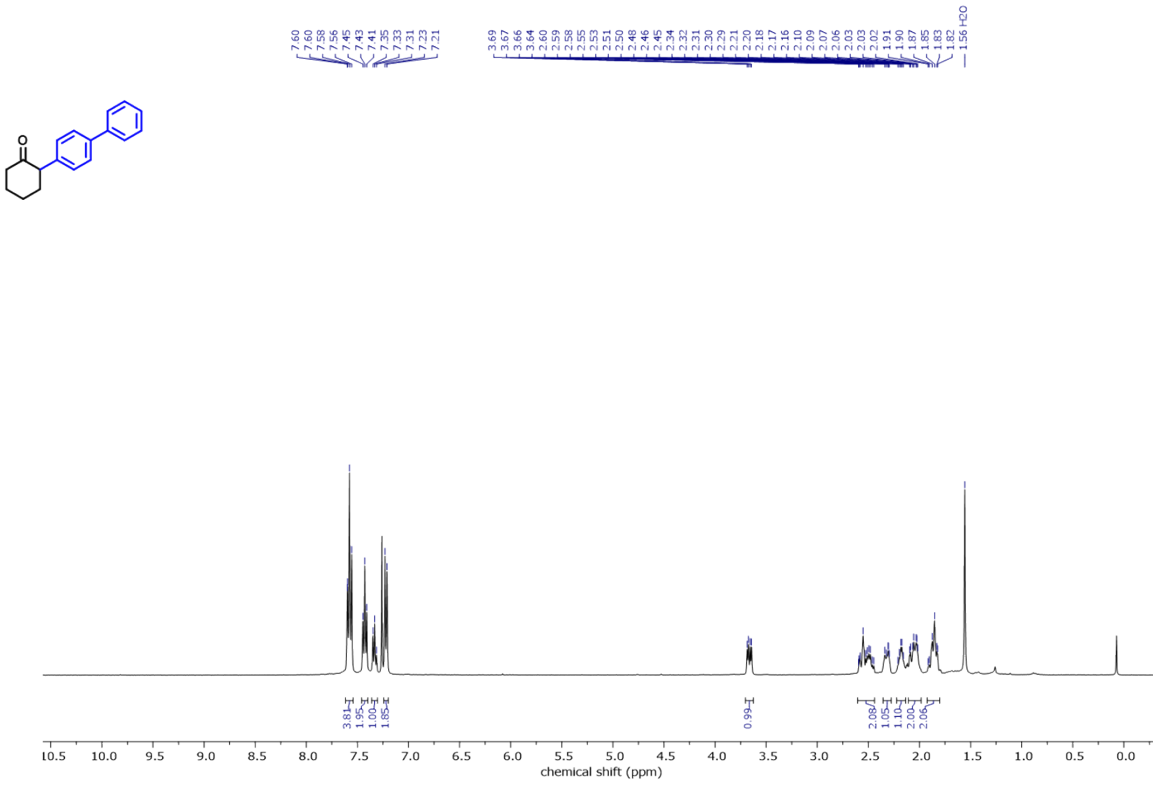
^{13}C NMR spectrum (101 MHz) of **3l** in $CDCl_3$



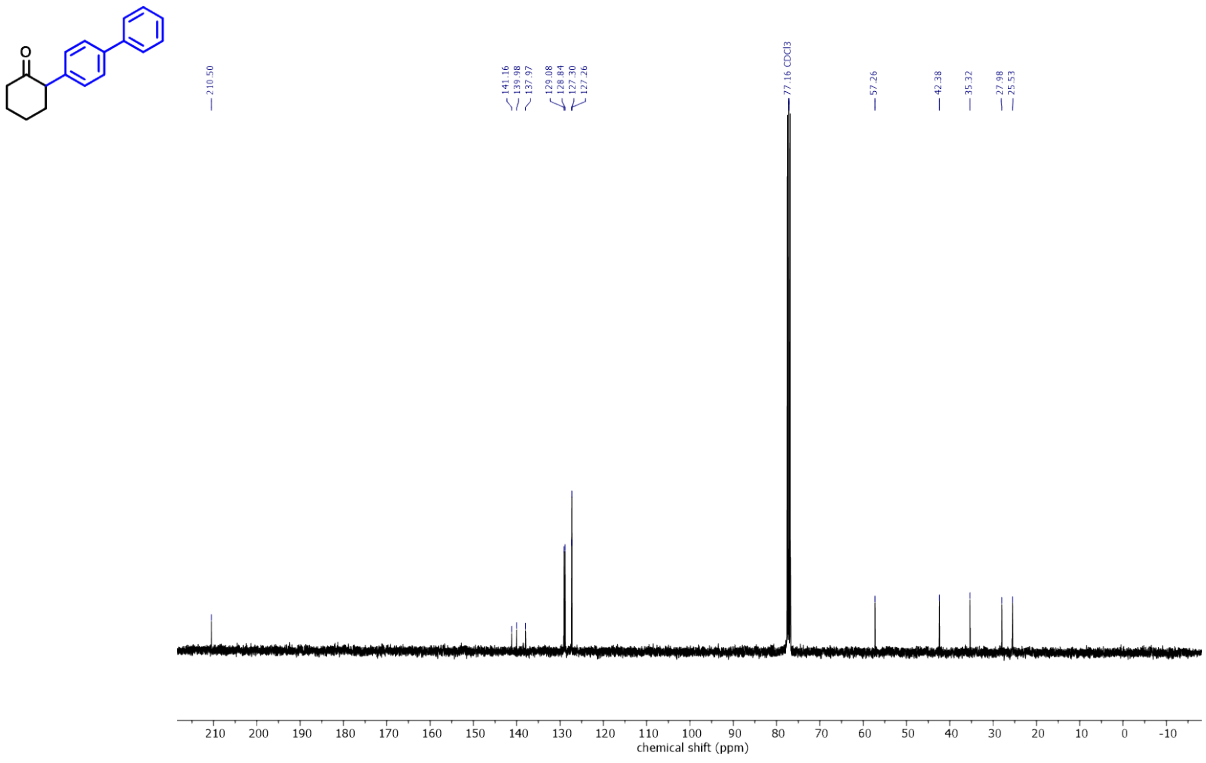
^{1}H NMR spectrum (400 MHz) of **3m** in $CDCl_3$



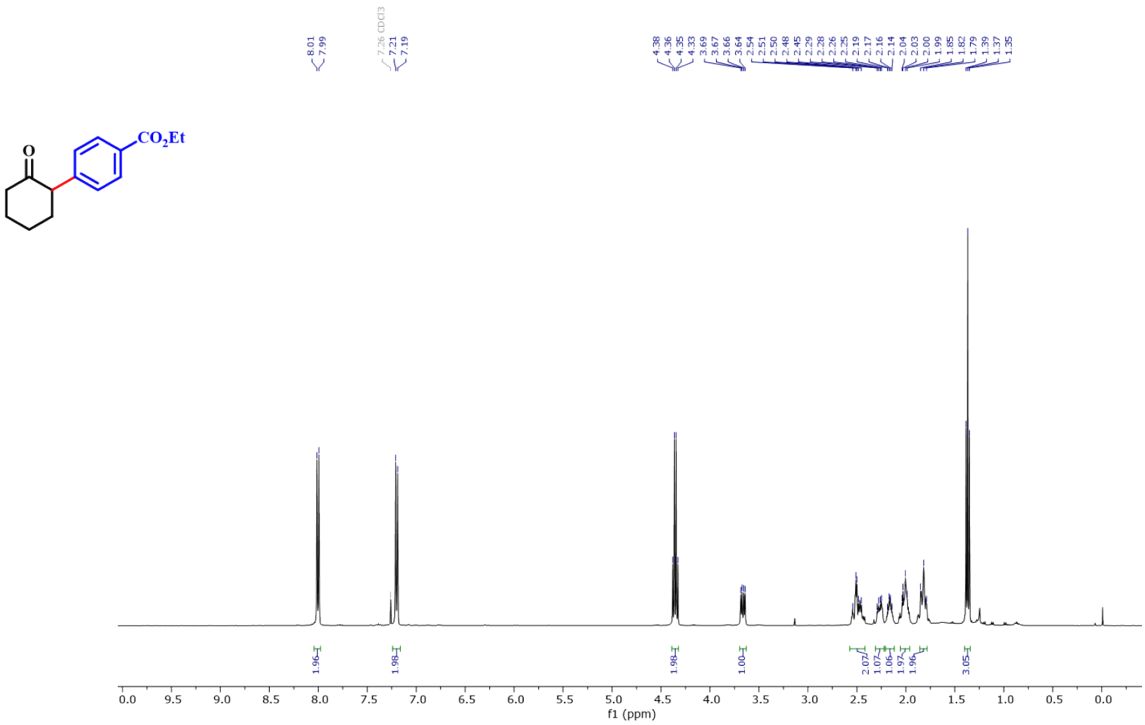
^{13}C NMR spectrum (101 MHz) of **3m** in $CDCl_3$



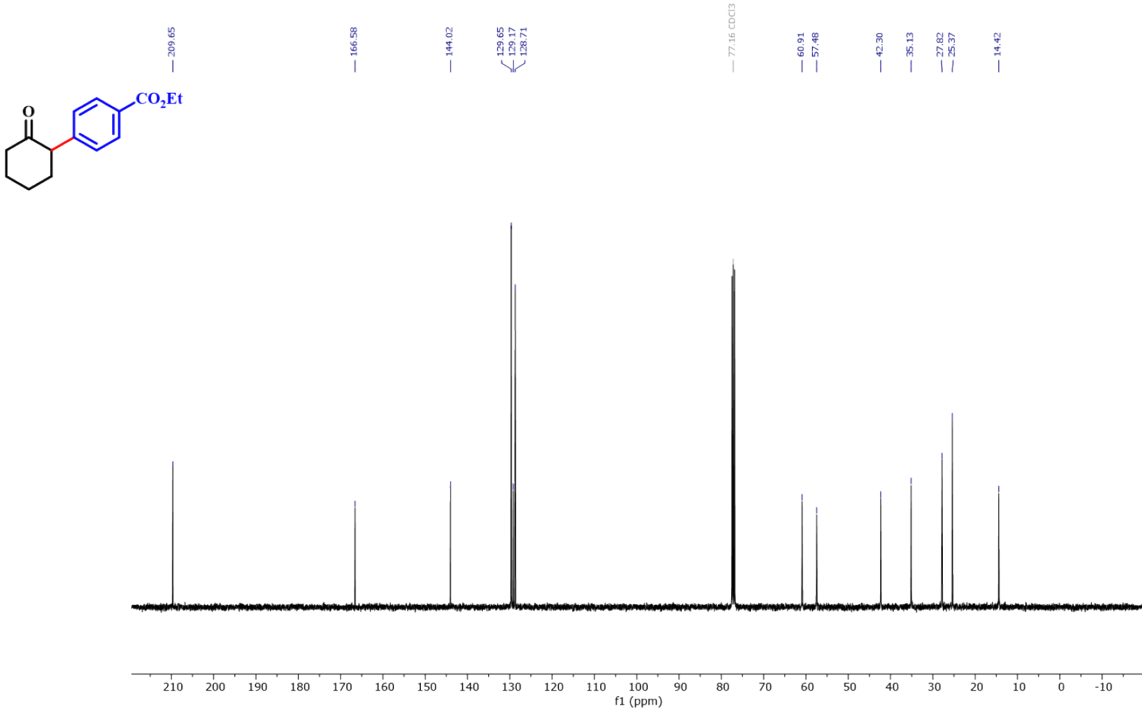
^{1}H NMR spectrum (400 MHz) of **3n** in $CDCl_3$



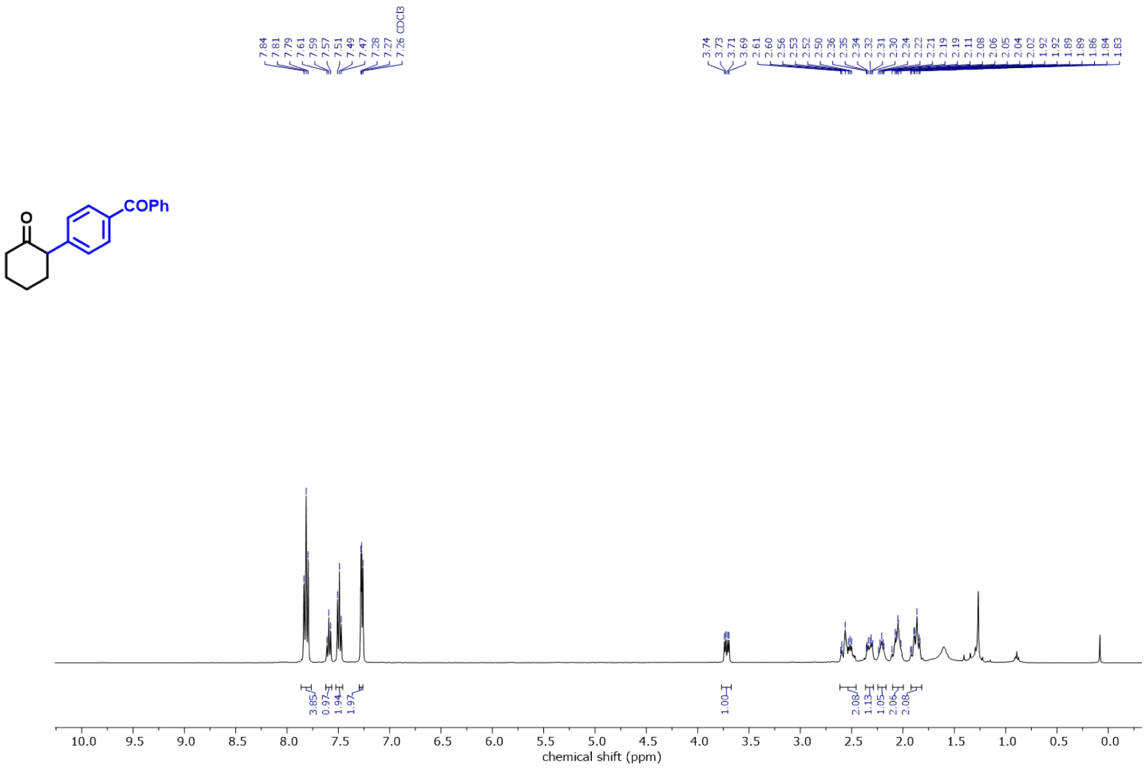
^{13}C NMR spectrum (101 MHz) of **3n** in $CDCl_3$



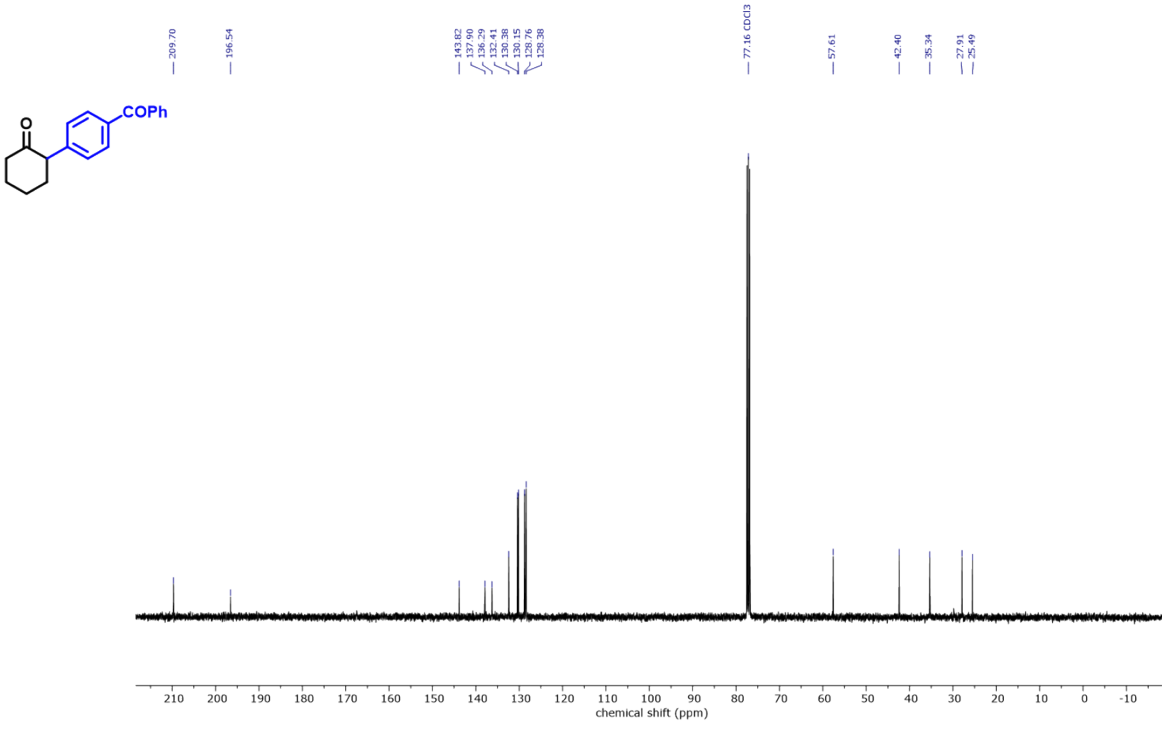
^{1}H NMR spectrum (400 MHz) of **3o** in $CDCl_3$



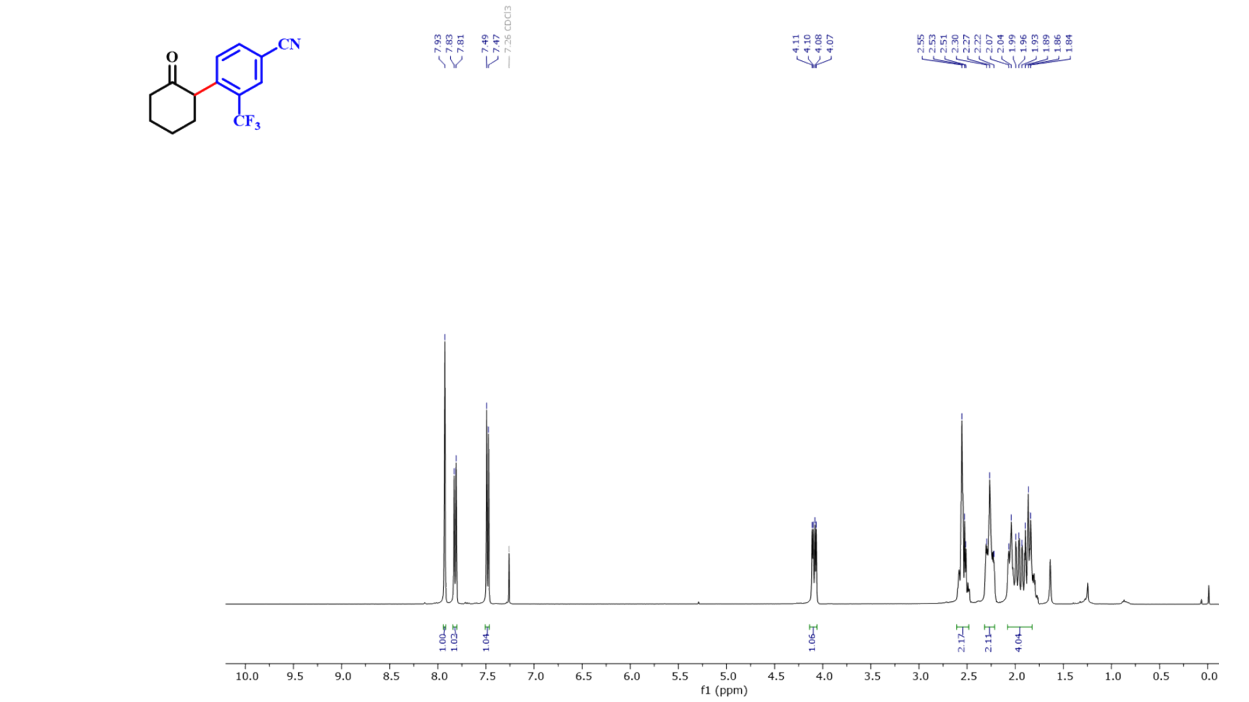
^{13}C NMR spectrum (101 MHz) of **3o** in $CDCl_3$



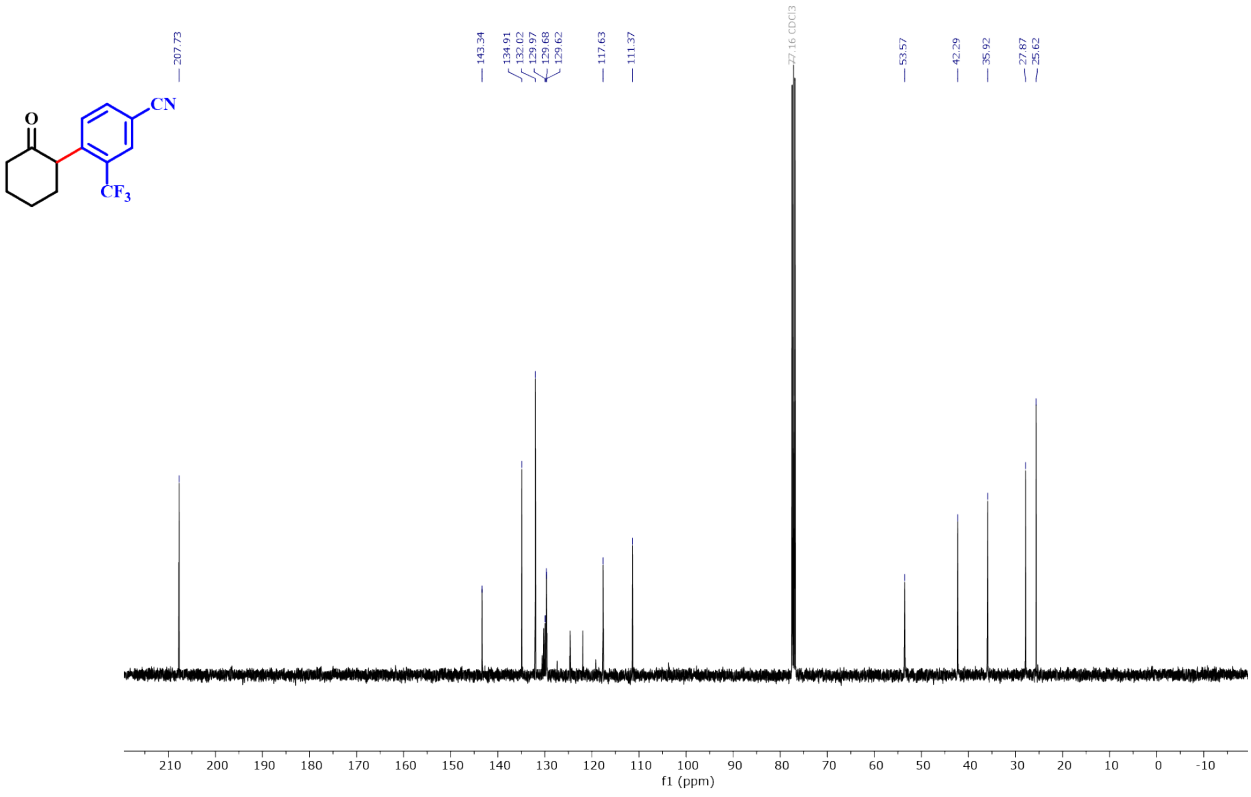
^{1}H NMR spectrum (400 MHz) of **3p** in $CDCl_3$



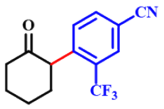
^{13}C NMR spectrum (101 MHz) of **3p** in $CDCl_3$

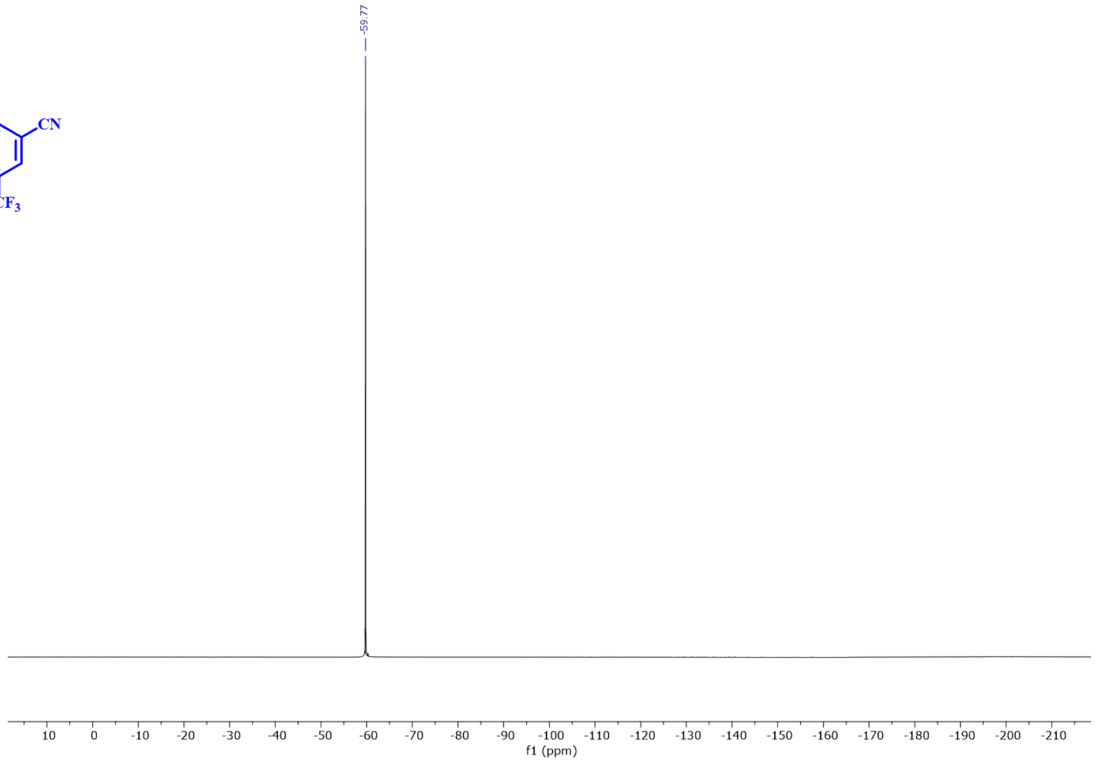


^{1}H NMR spectrum (400 MHz) of **3q** in $CDCl_3$

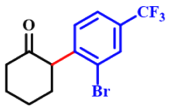


^{13}C NMR spectrum (101 MHz) of **3q** in $CDCl_3$

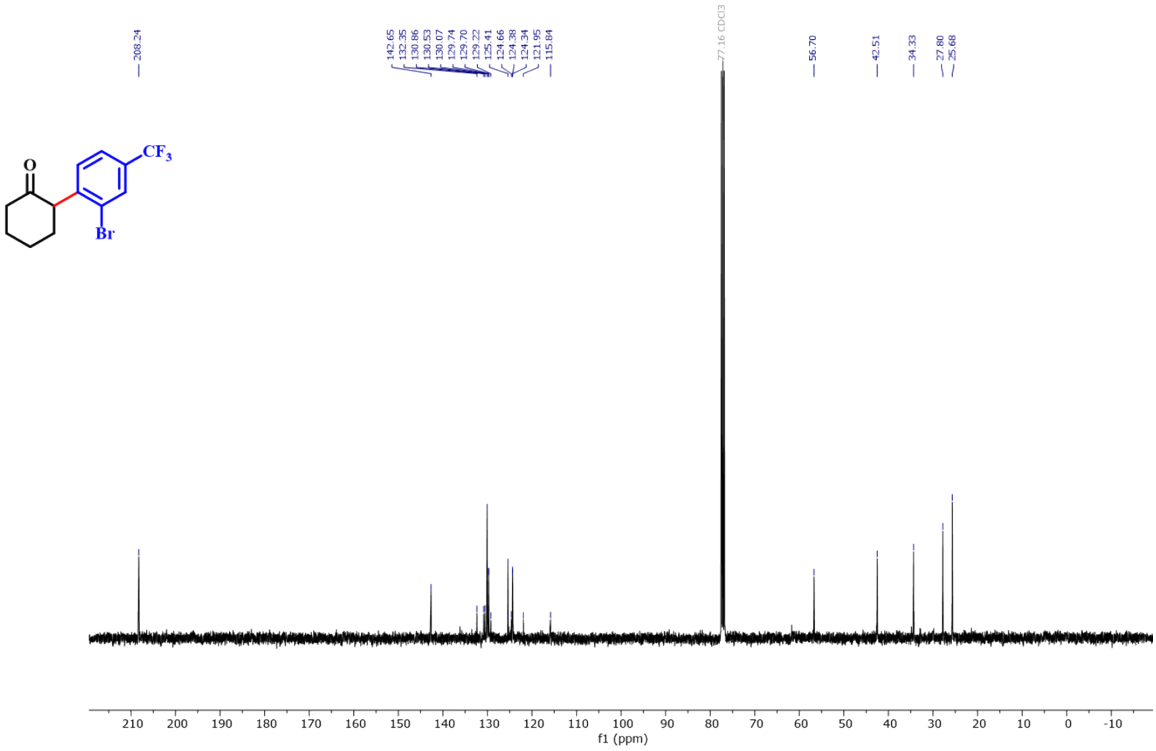




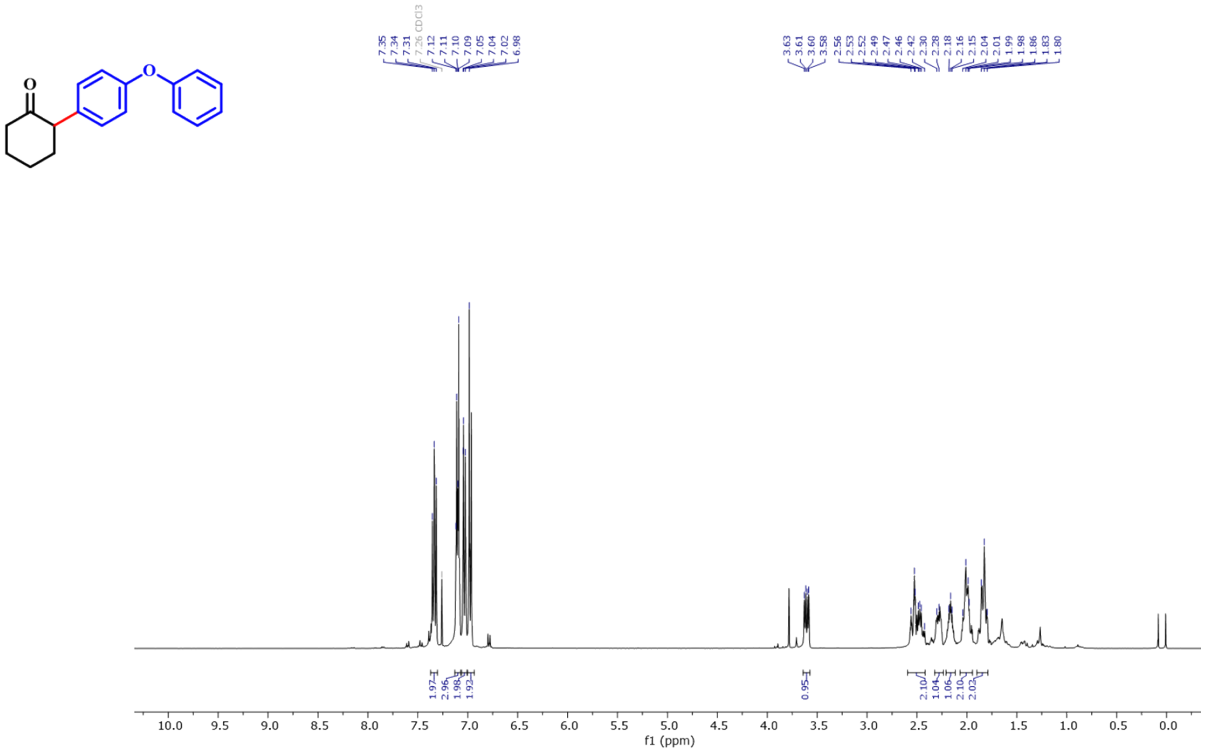
^{19}F NMR spectrum (376 MHz) of **3q** in $CDCl_3$



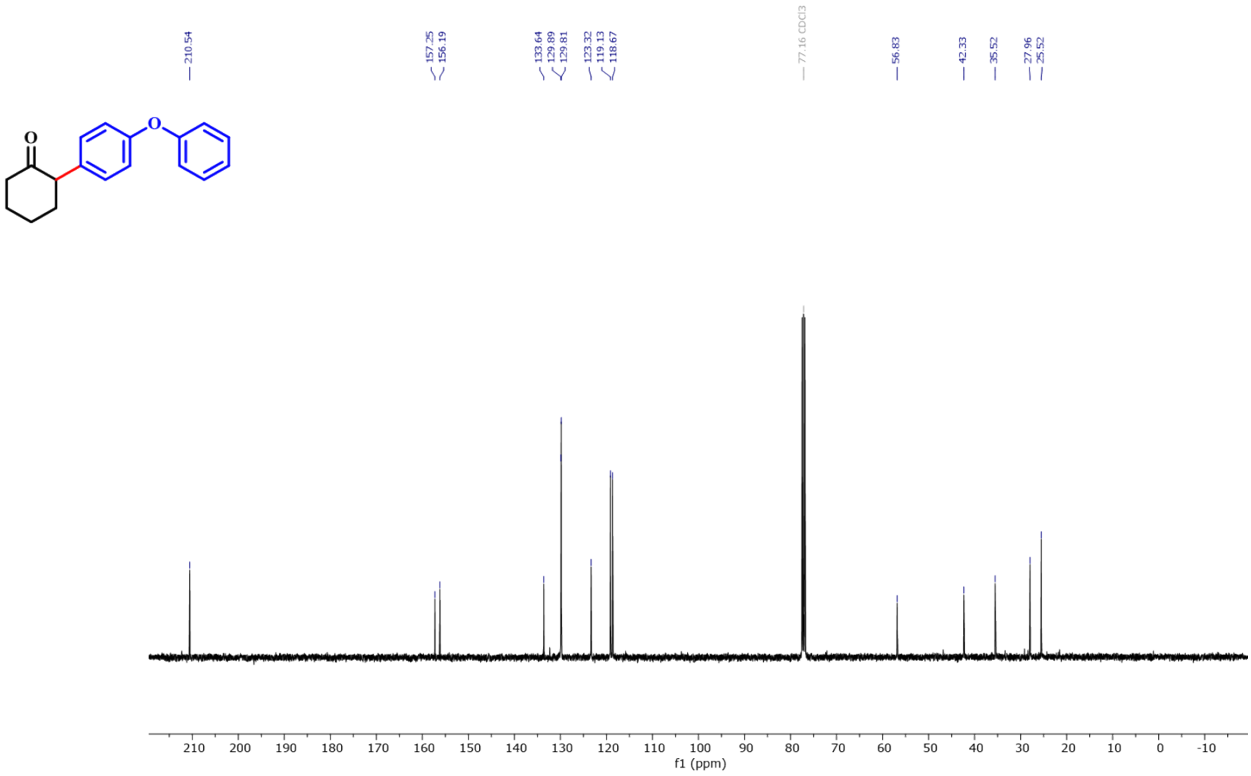
^{1}H NMR spectrum (400 MHz) of **3r** in $CDCl_3$



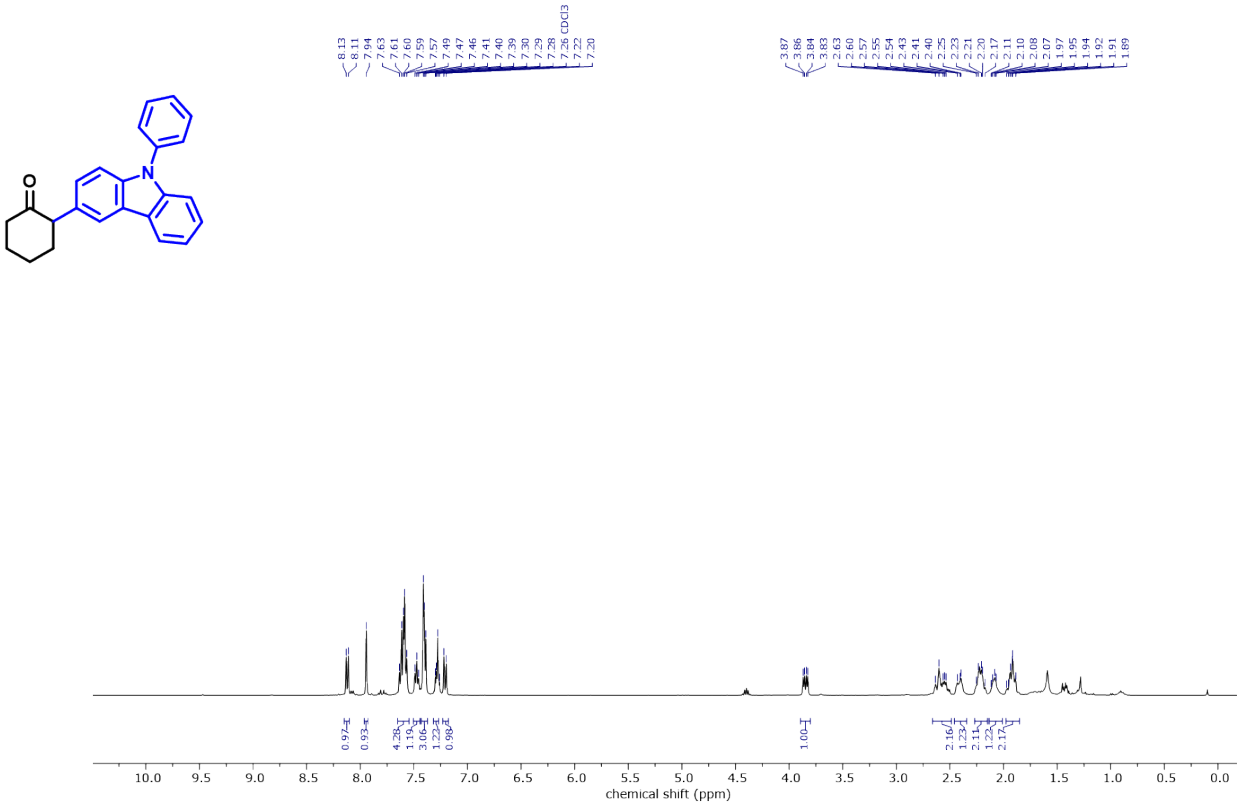
^{13}C NMR spectrum (101 MHz) of **3r** in $CDCl_3$



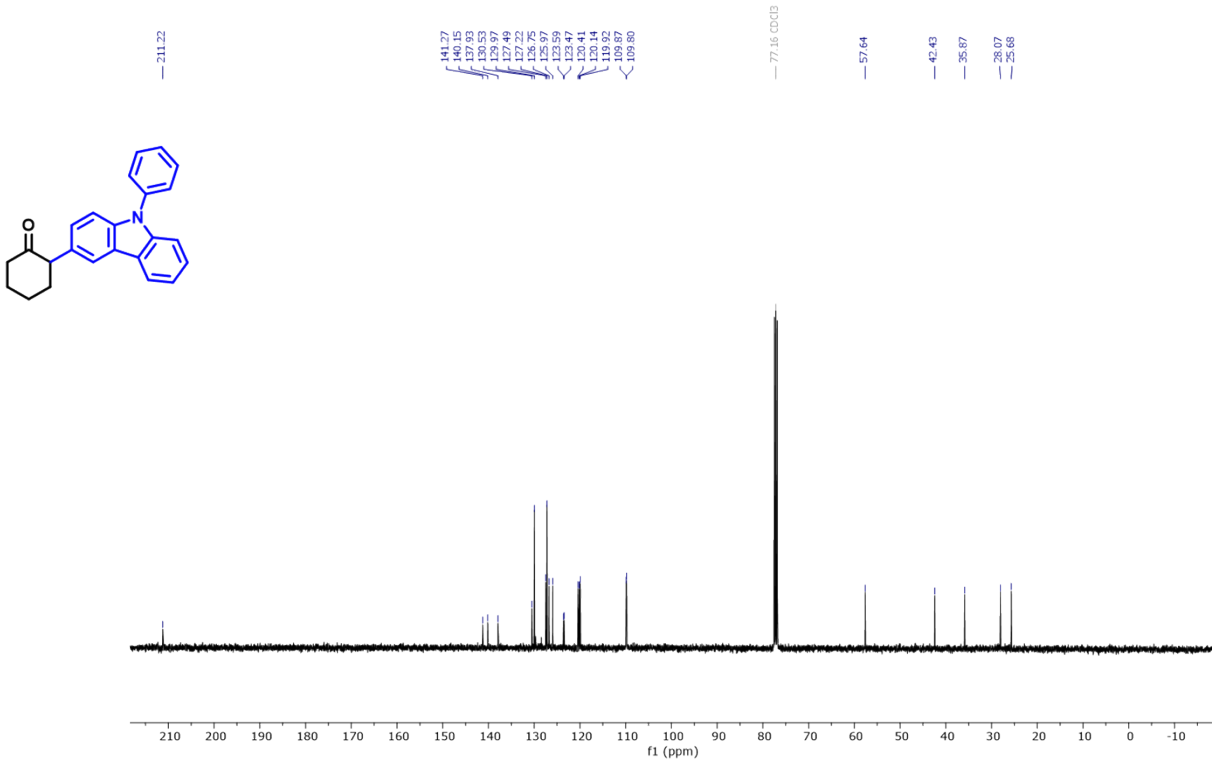
^{1}H NMR spectrum (400 MHz) of **3s** in $CDCl_3$



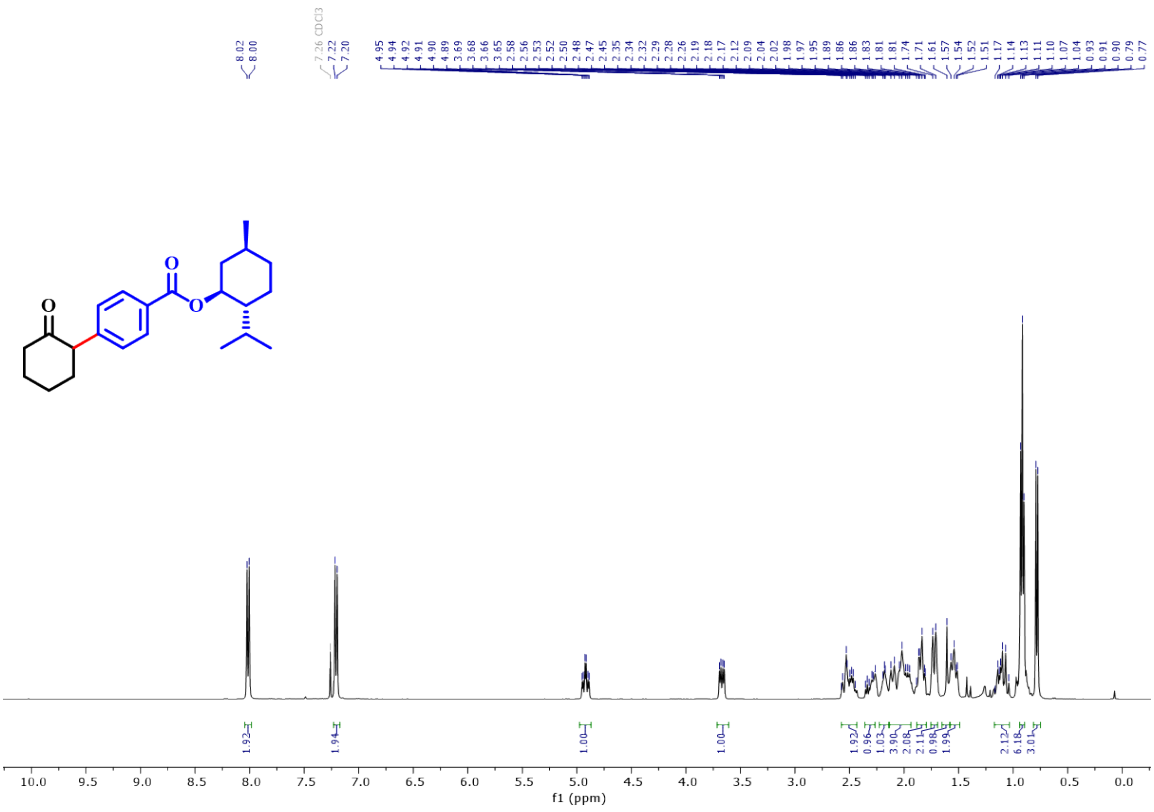
^{13}C NMR spectrum (101 MHz) of **3s** in $CDCl_3$



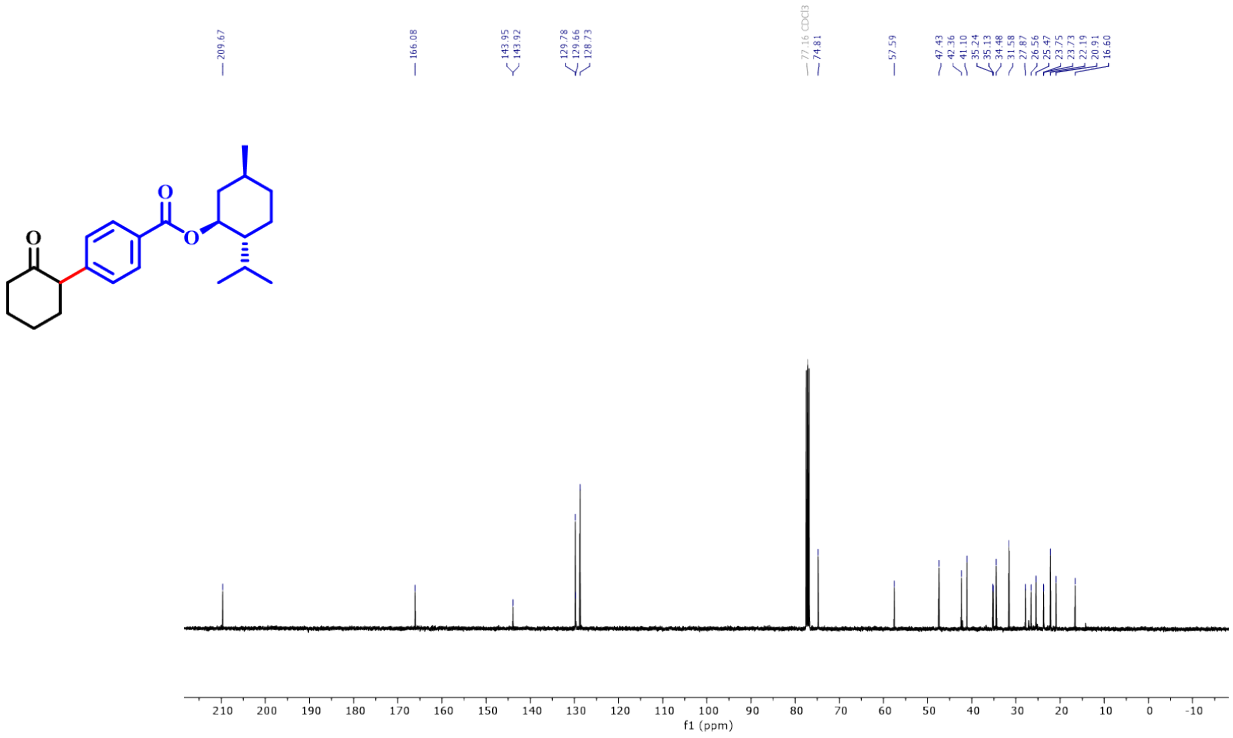
^{1}H NMR spectrum (400 MHz) of **3t** in $CDCl_3$



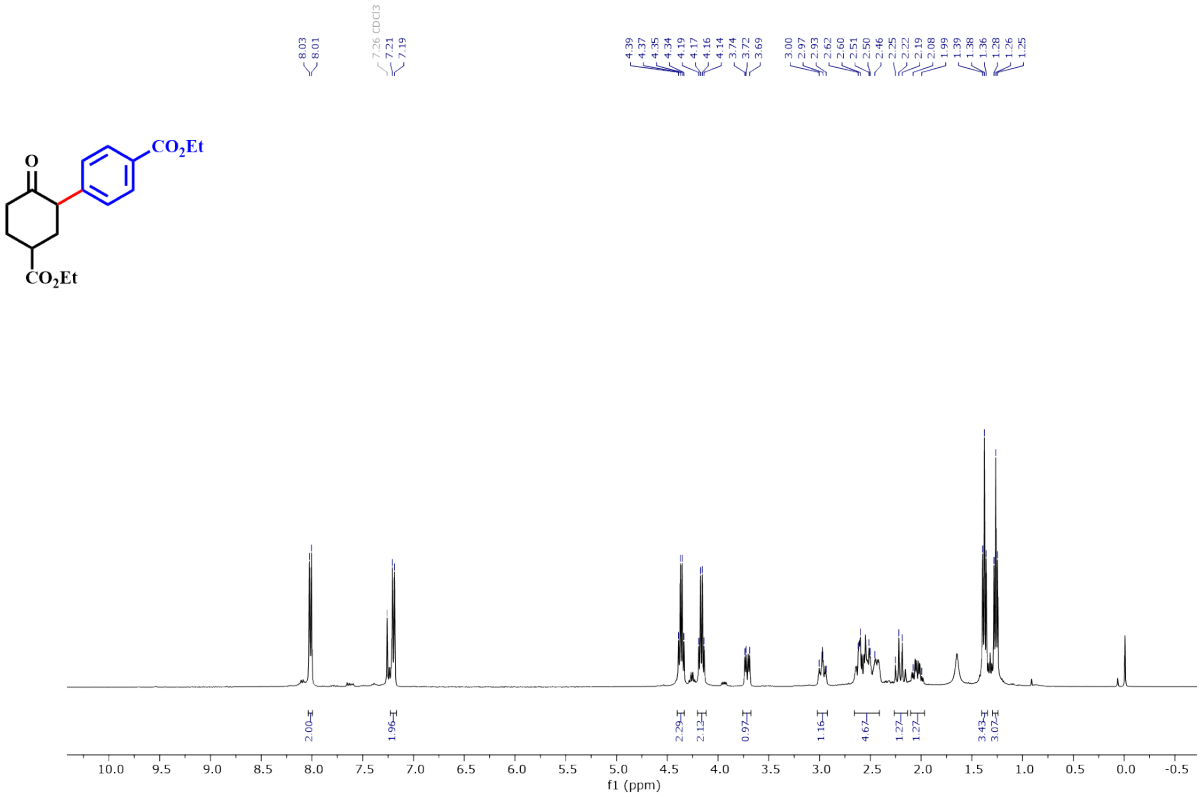
^{13}C NMR spectrum (101 MHz) of **3t** in $CDCl_3$



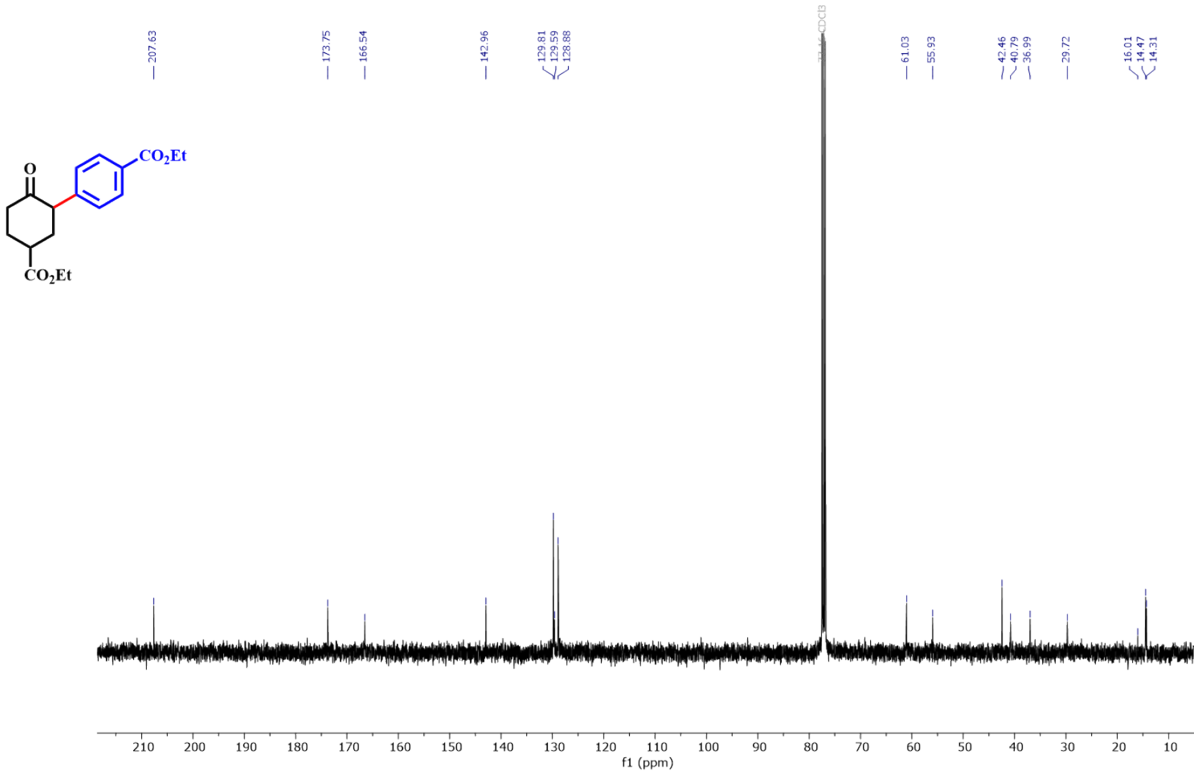
^{1}H NMR spectrum (400 MHz) of **3u** in $CDCl_3$



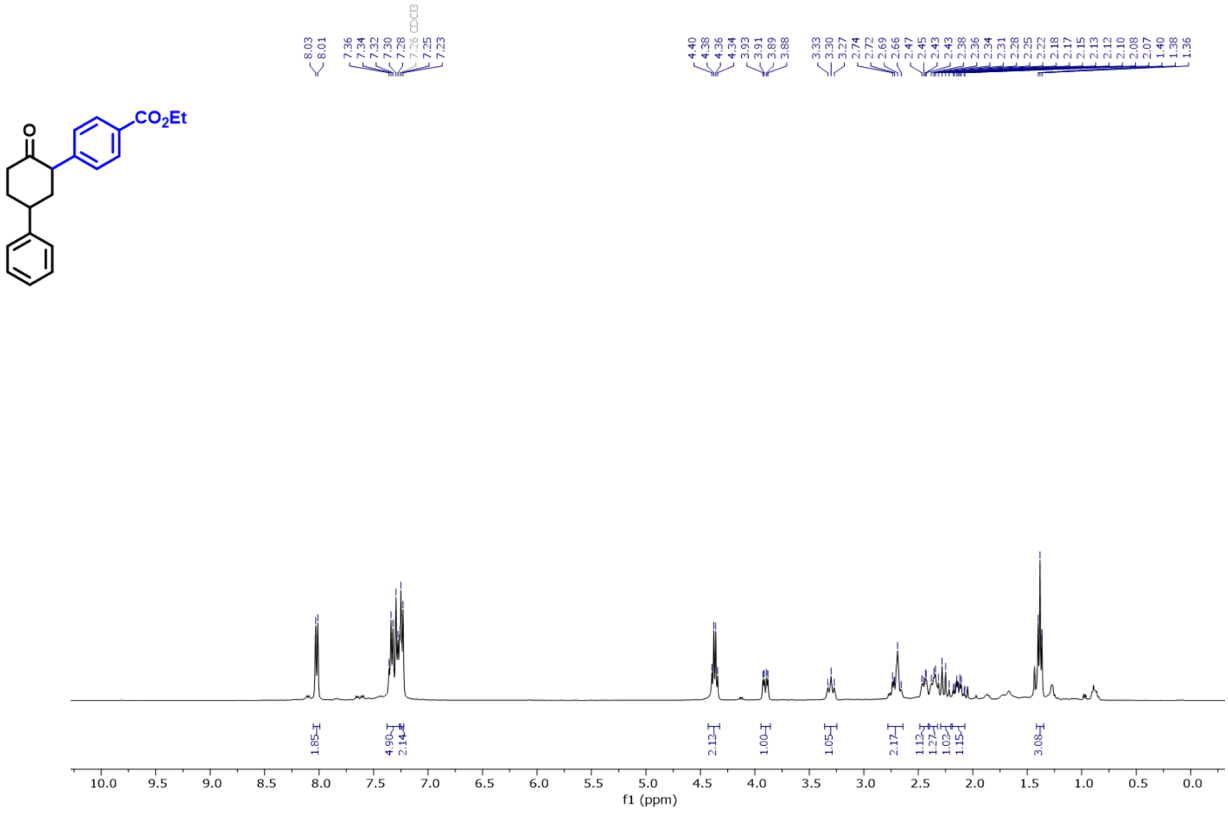
^{13}C NMR spectrum (101 MHz) of **3u** in $CDCl_3$



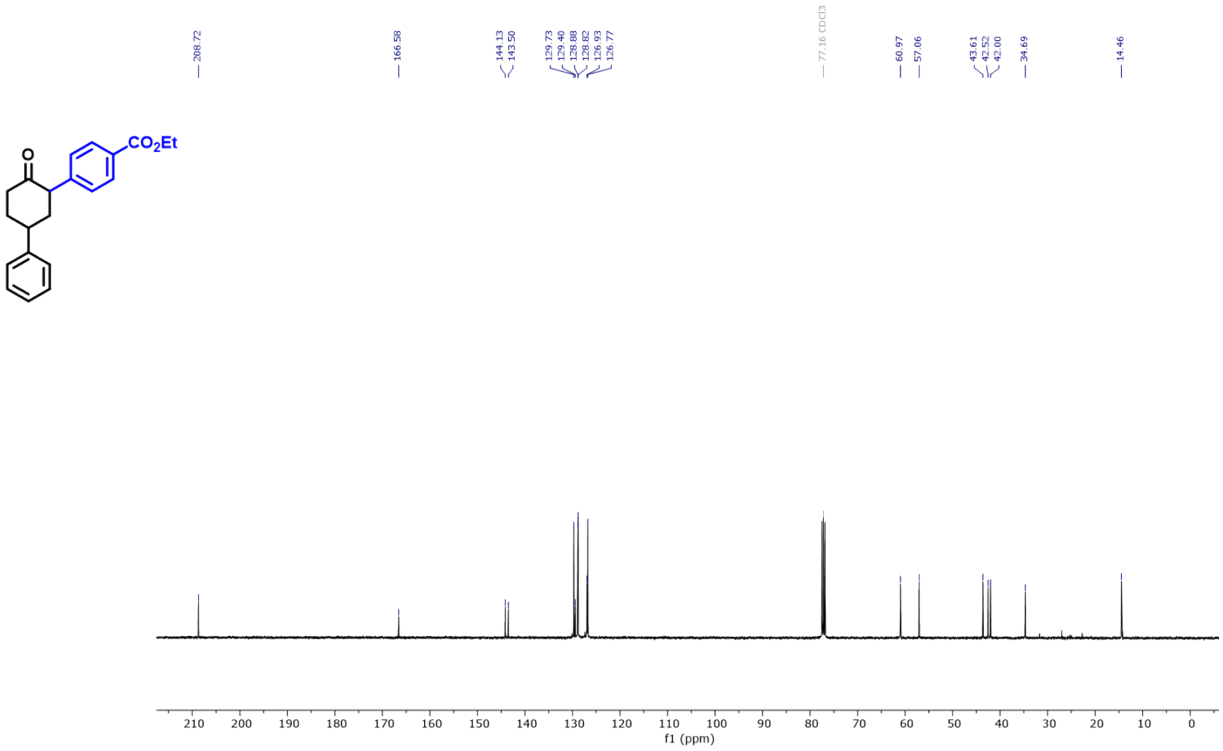
^{1}H NMR spectrum (400 MHz) of **5a** in $CDCl_3$



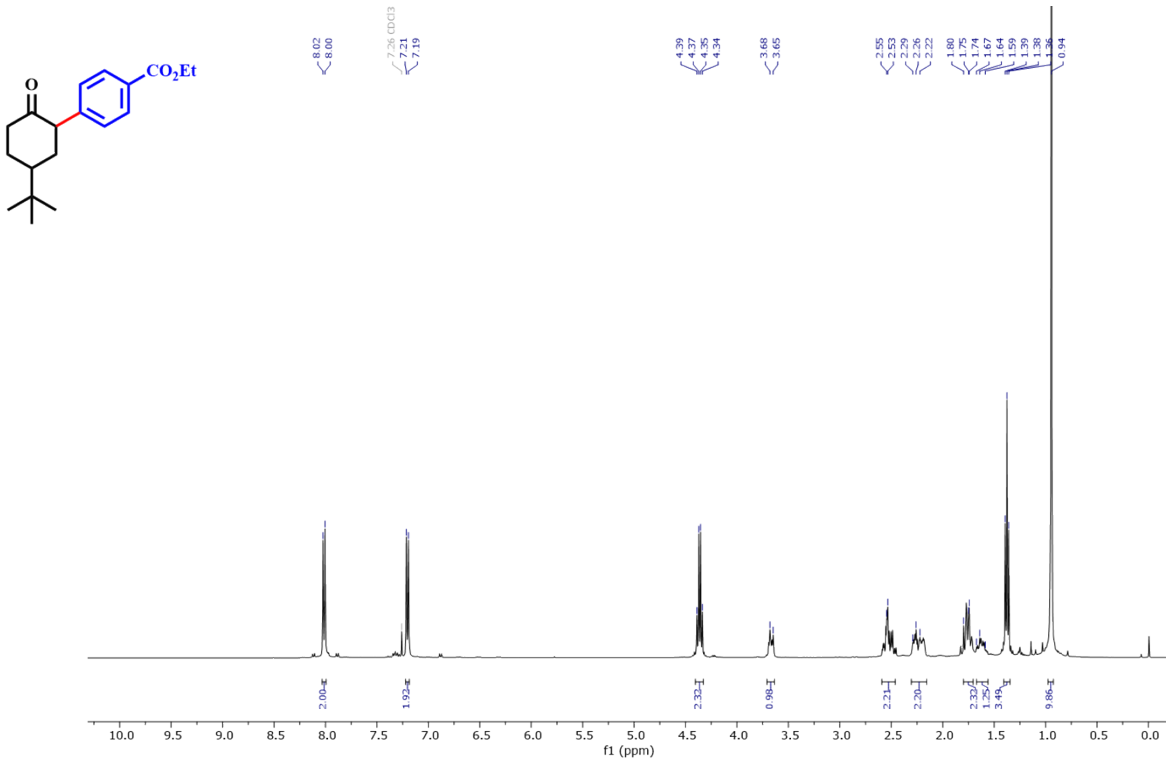
^{13}C NMR spectrum (101 MHz) of **5a** in $CDCl_3$



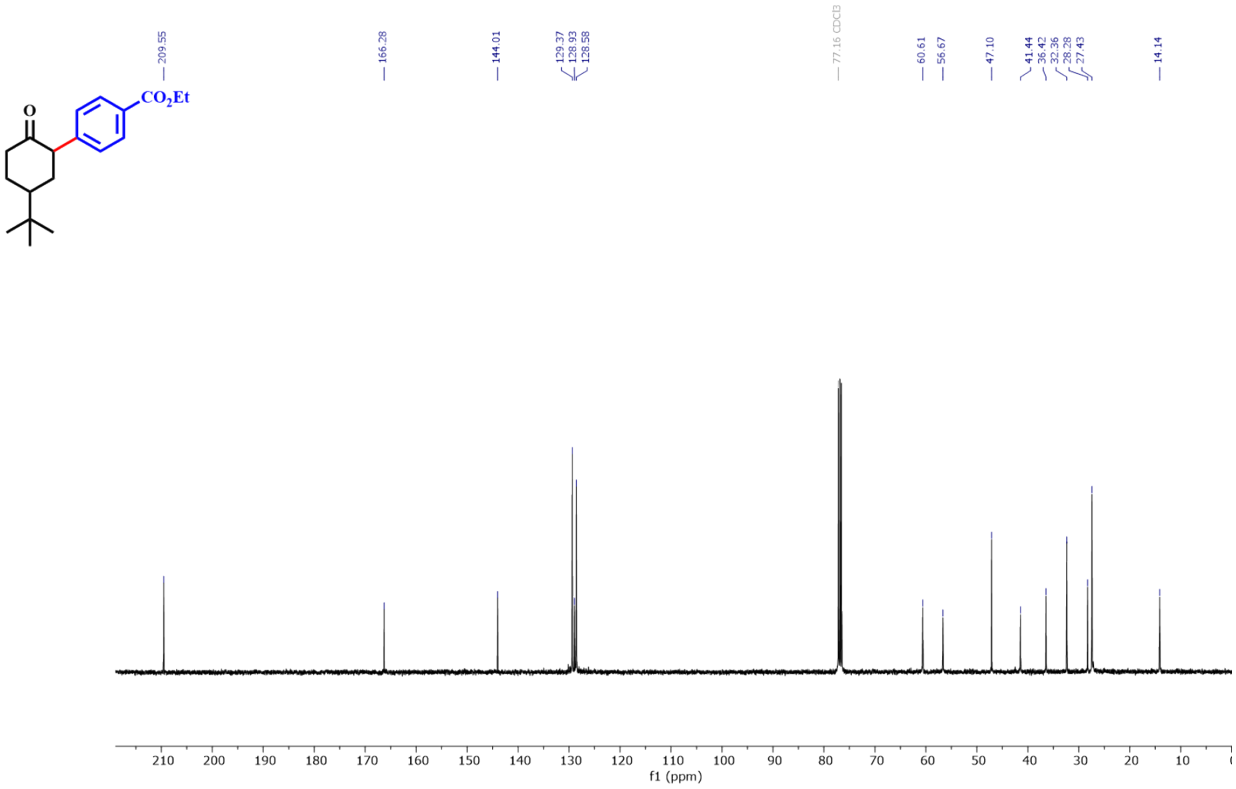
^{1}H NMR spectrum (400 MHz) of **5b** in $CDCl_3$



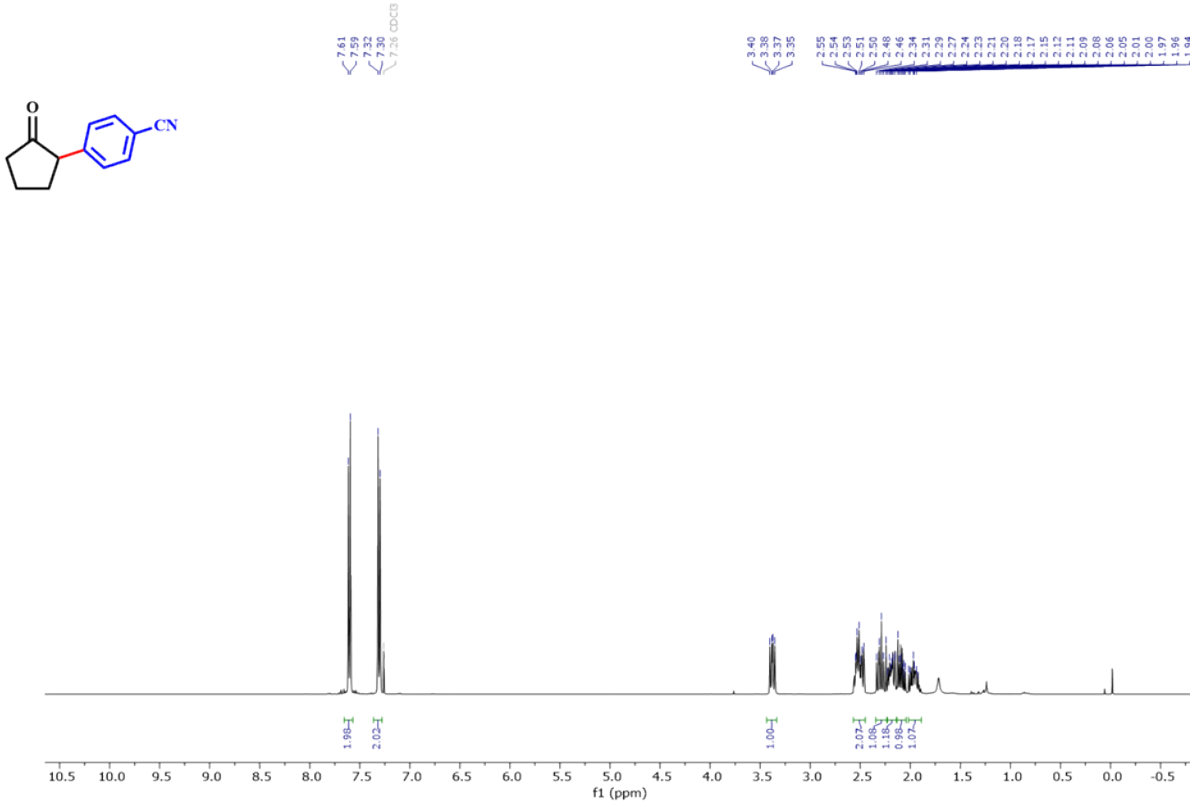
^{13}C NMR spectrum (101 MHz) of **5b** in $CDCl_3$



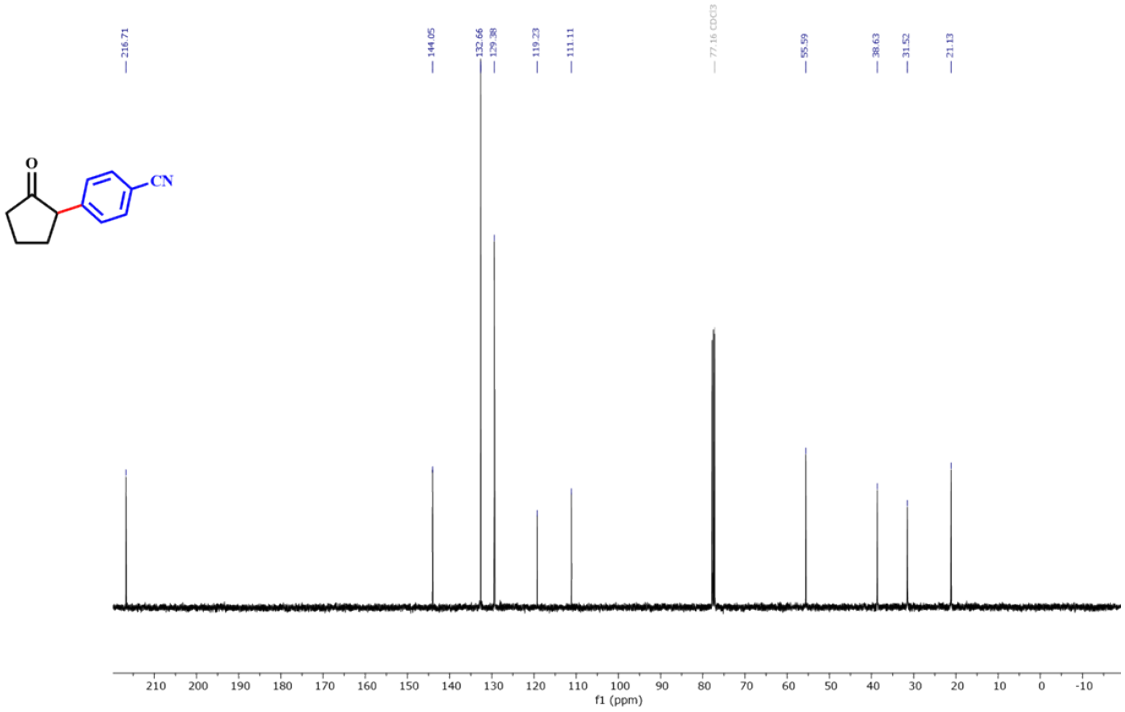
^{1}H NMR spectrum (400 MHz) of **5c** in $CDCl_3$



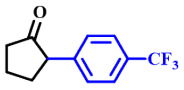
^{13}C NMR spectrum (101 MHz) of **5c** in $CDCl_3$

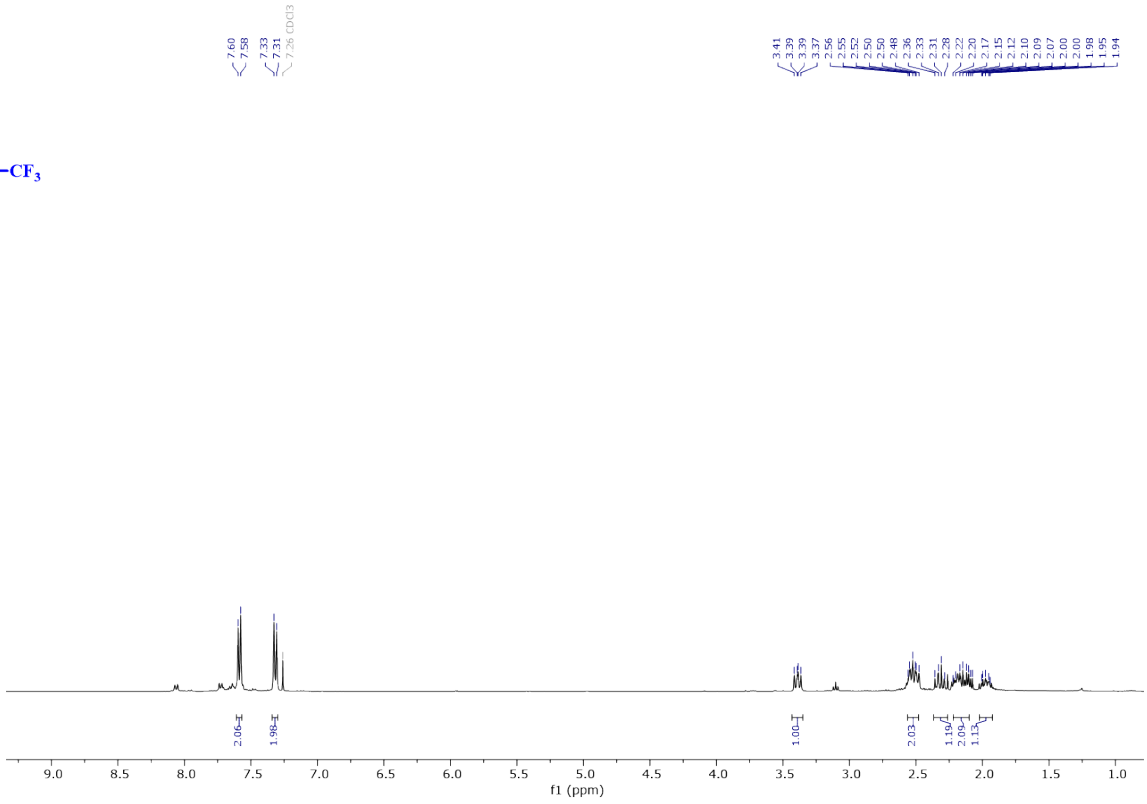


^{1}H NMR spectrum (400 MHz) of **5d** in $CDCl_3$

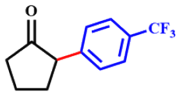


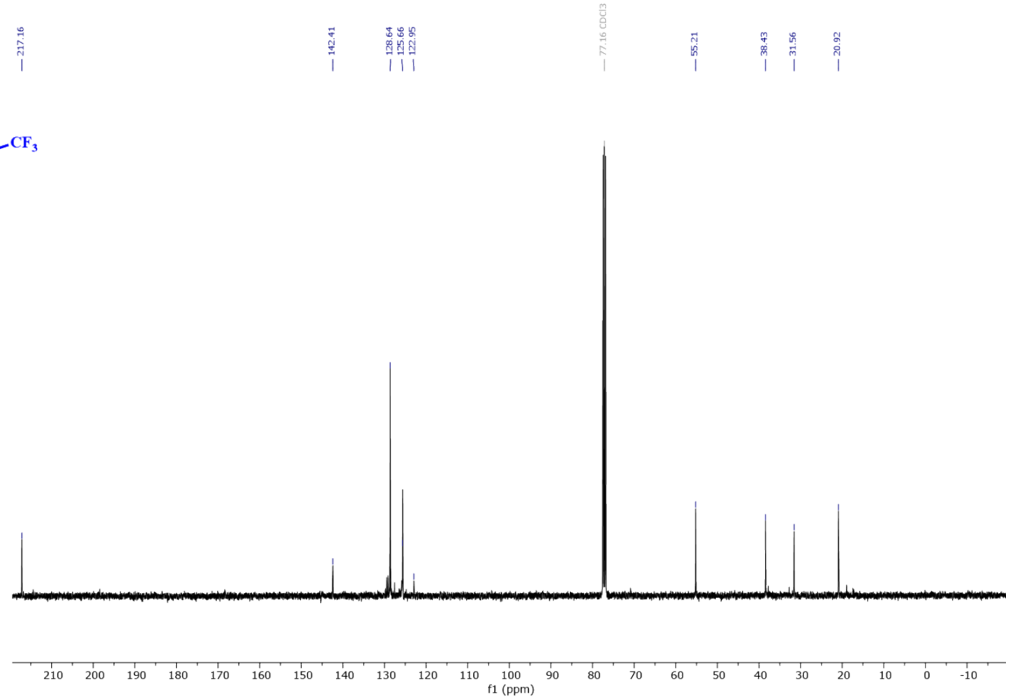
^{13}C NMR spectrum (101 MHz) of **5d** in $CDCl_3$



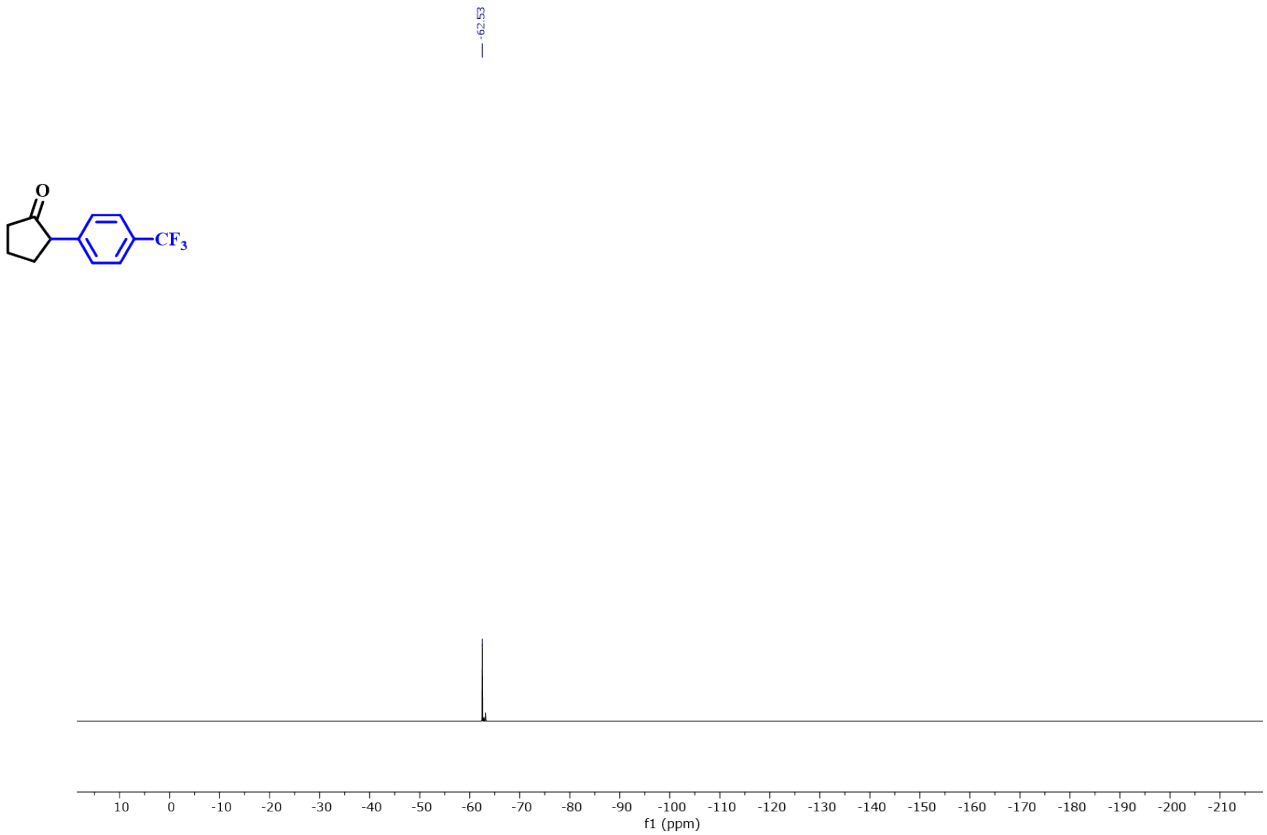


^{1}H NMR spectrum (400 MHz) of **5e** in $CDCl_3$

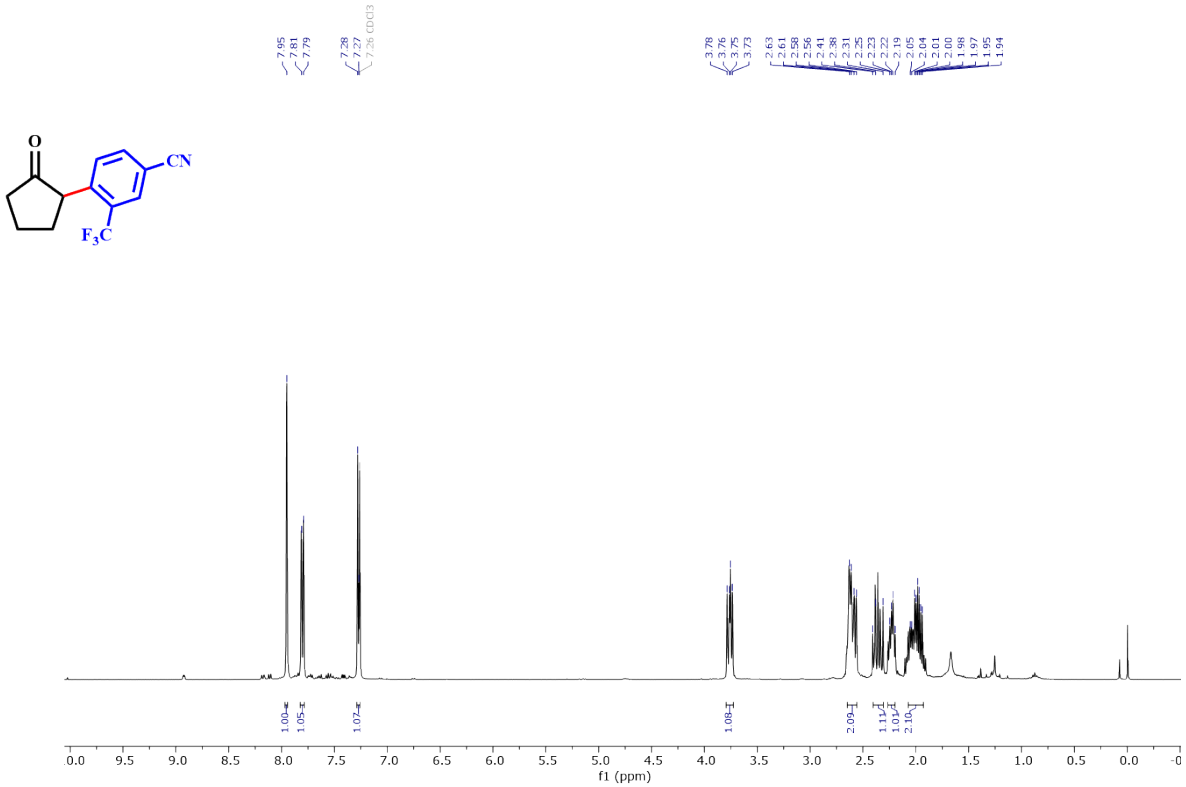




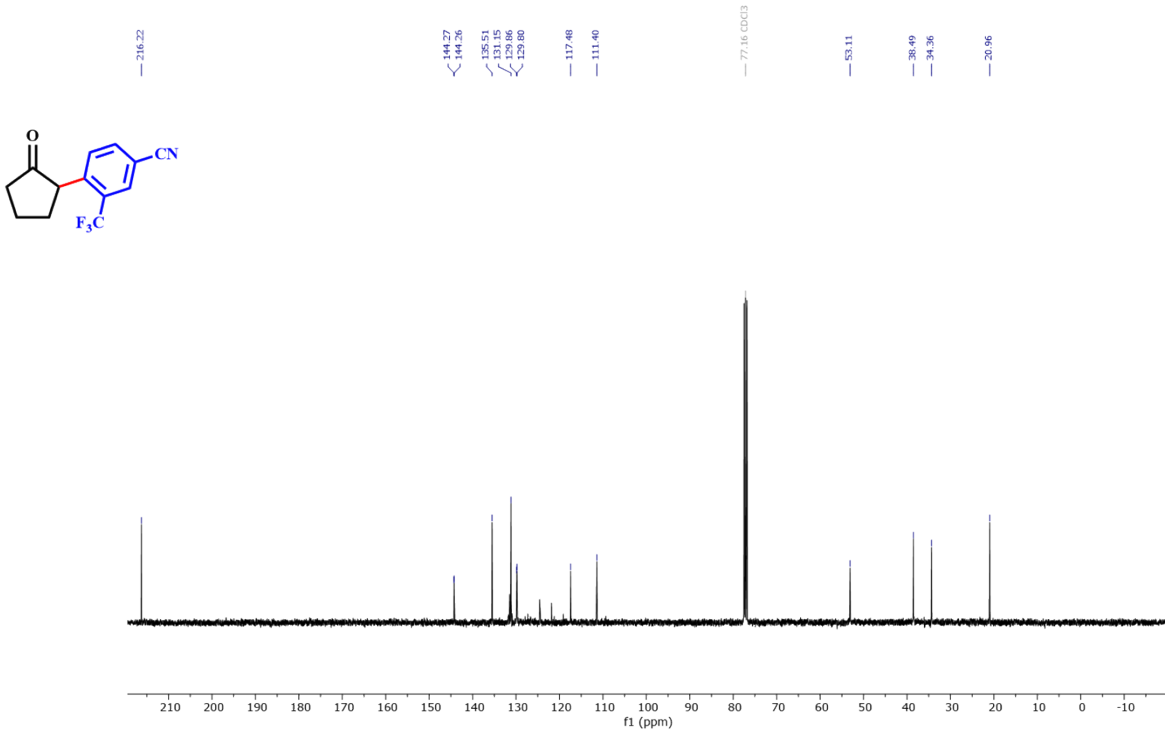
^{13}C NMR spectrum (101 MHz) of **5e** in $CDCl_3$



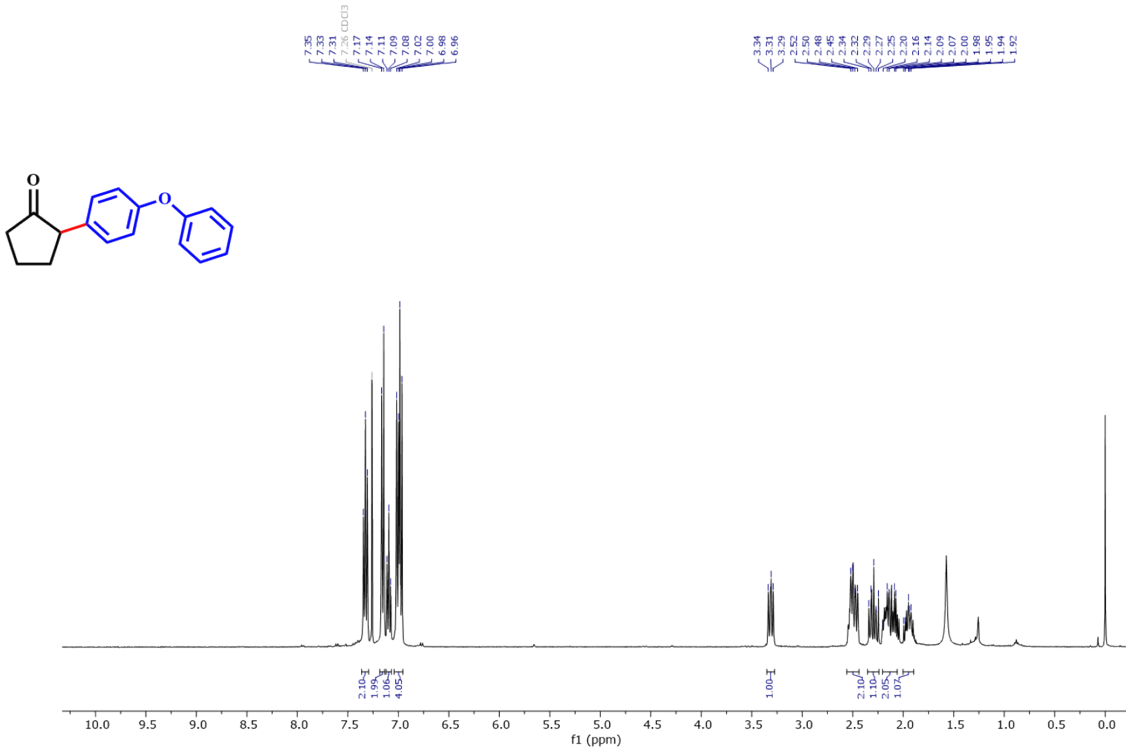
^{19}F NMR spectrum (376 MHz) of **5e** in $CDCl_3$



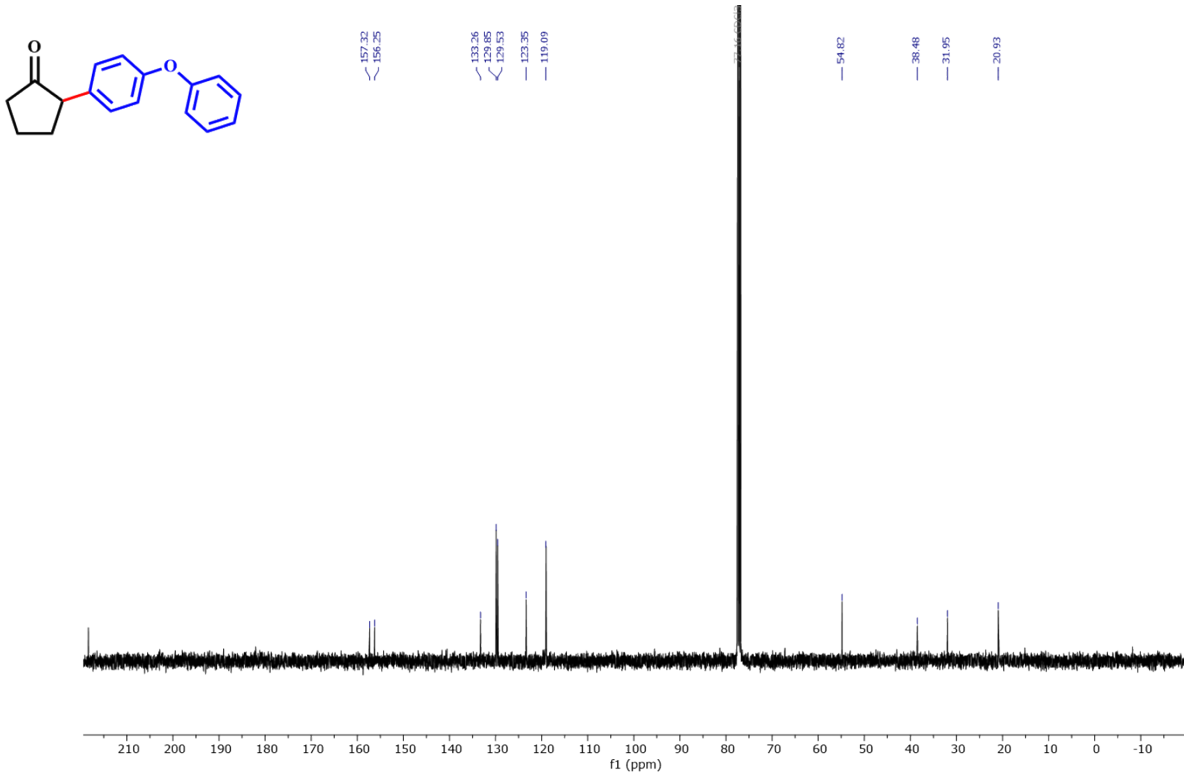
^{1}H NMR spectrum (400 MHz) of **5f** in $CDCl_3$



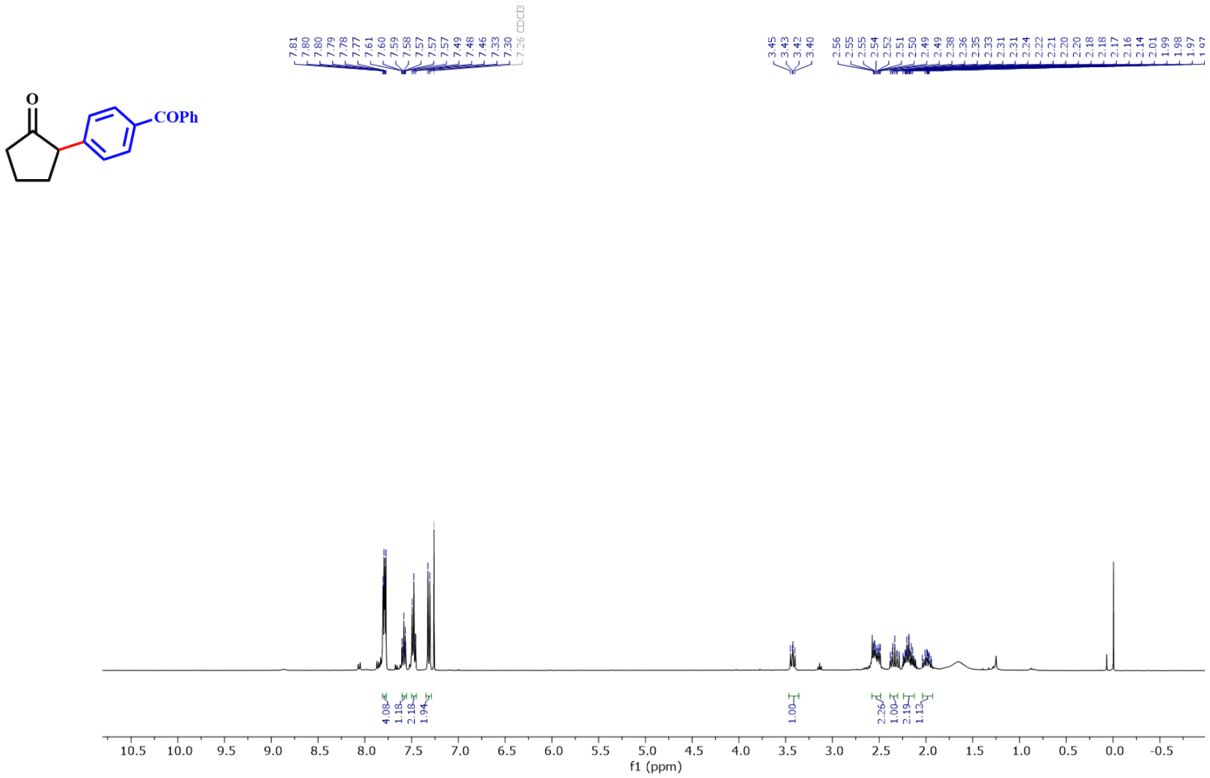
^{13}C NMR spectrum (101 MHz) of **5f** in $CDCl_3$



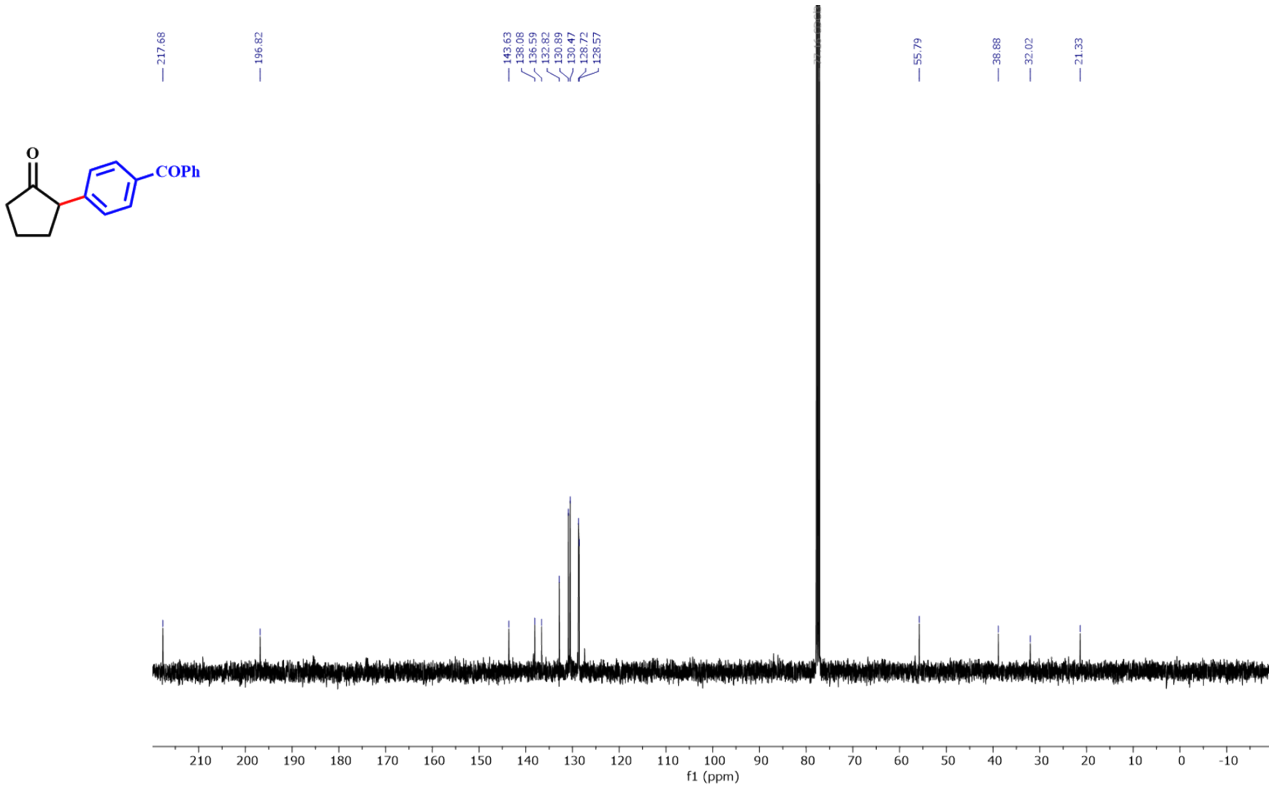
^{1}H NMR spectrum (400 MHz) of **5g** in $CDCl_3$



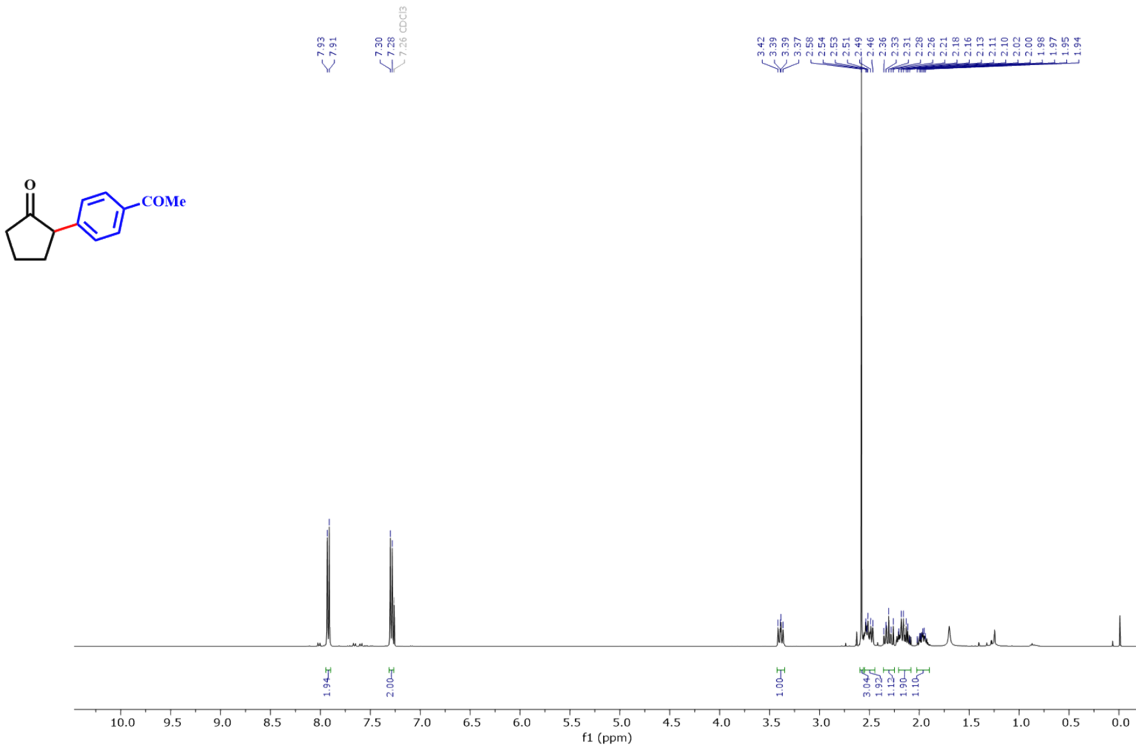
^{13}C NMR spectrum (101 MHz) of **5g** in $CDCl_3$



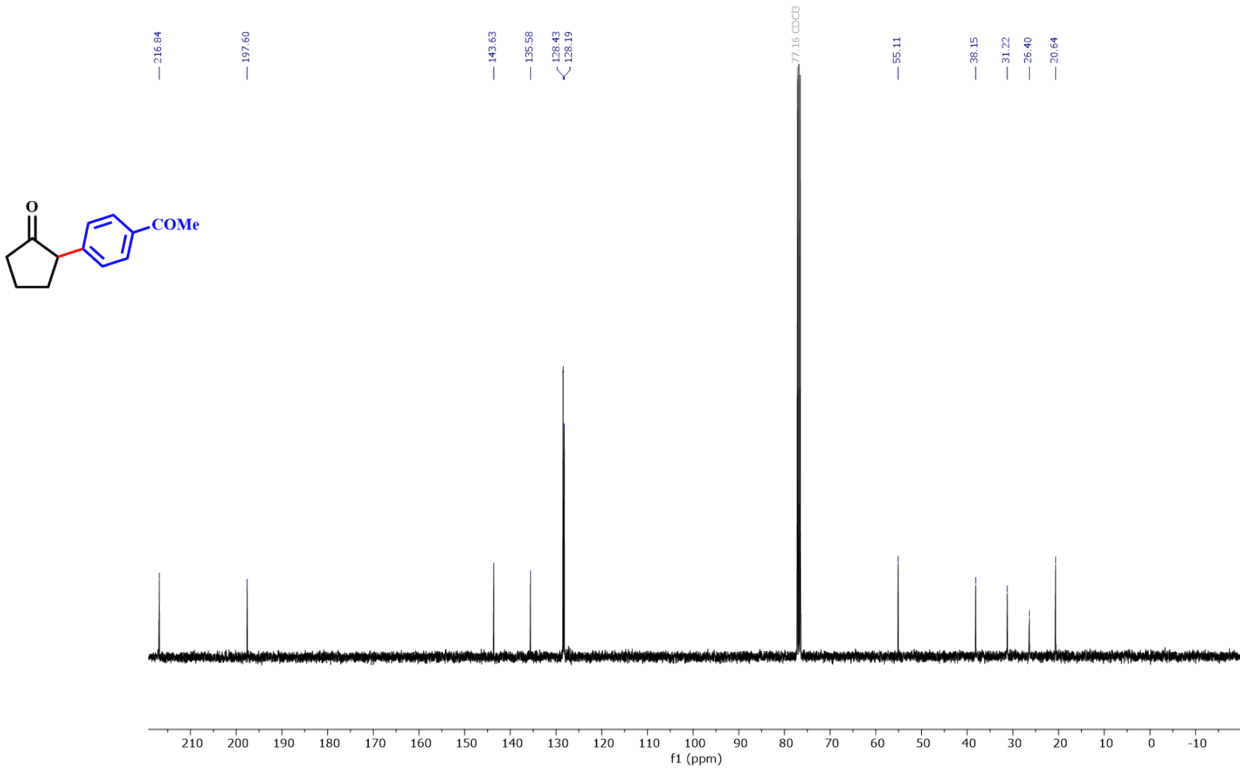
^{1}H NMR spectrum (400 MHz) of **5h** in $CDCl_3$



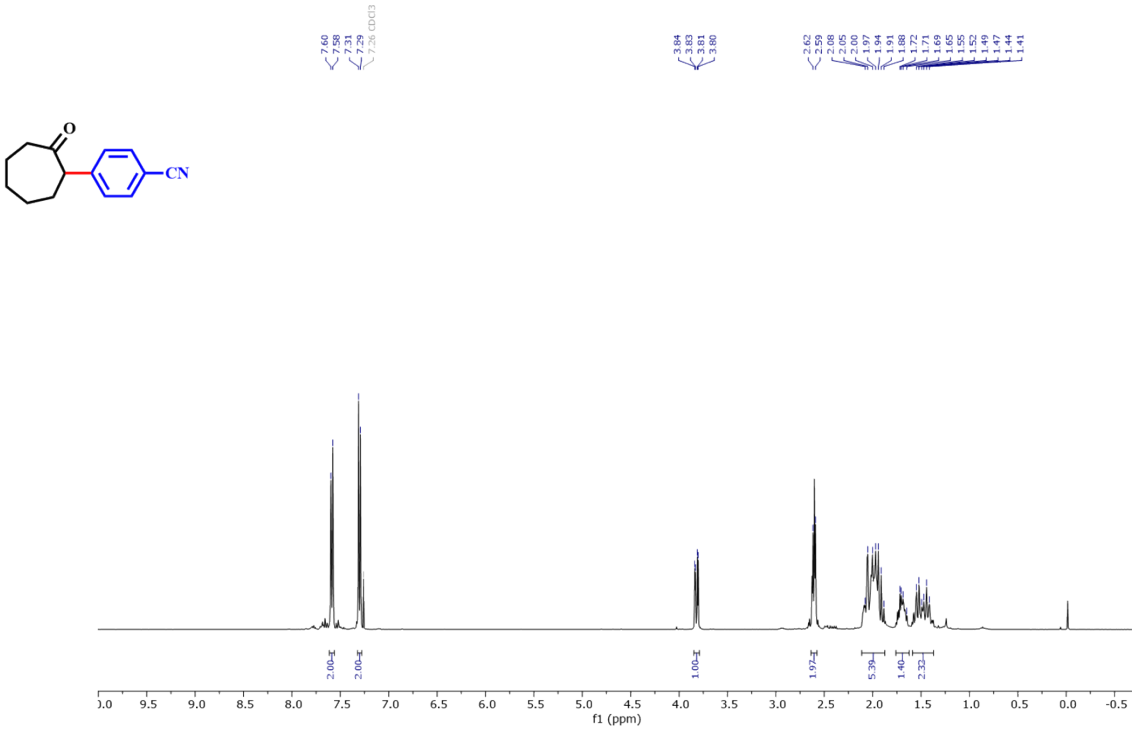
^{13}C NMR spectrum (101 MHz) of **5h** in $CDCl_3$



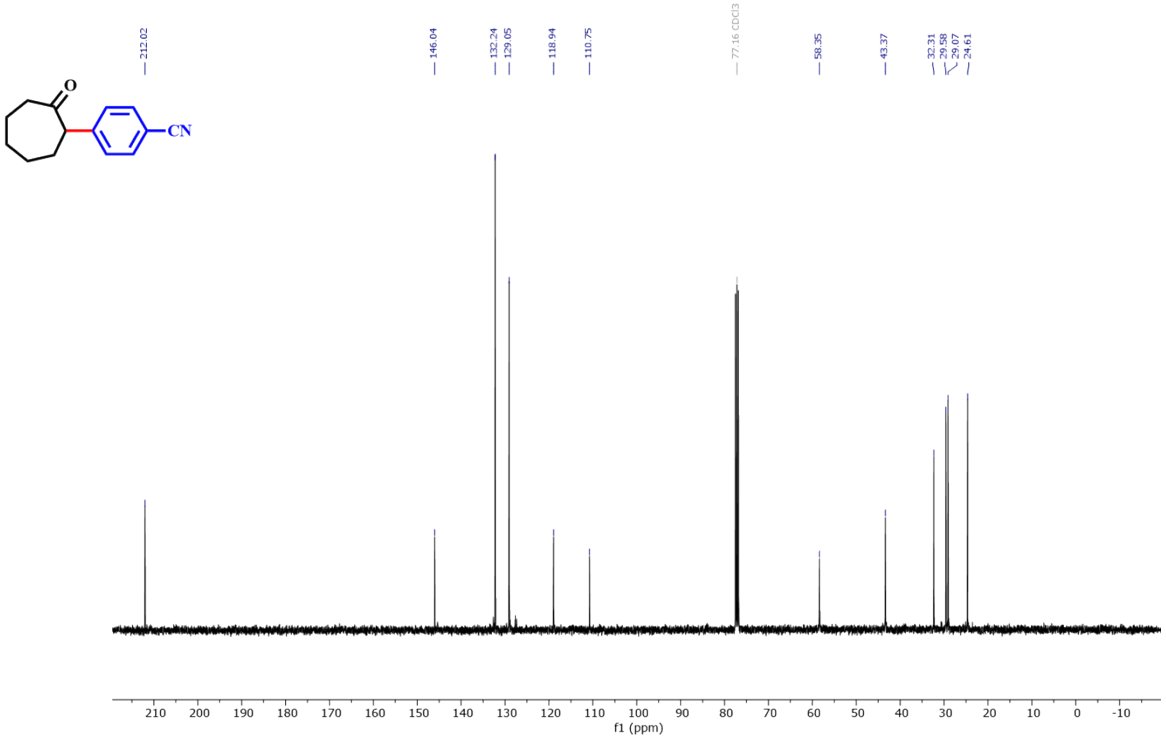
^{1}H NMR spectrum (400 MHz) of **5i** in $CDCl_3$



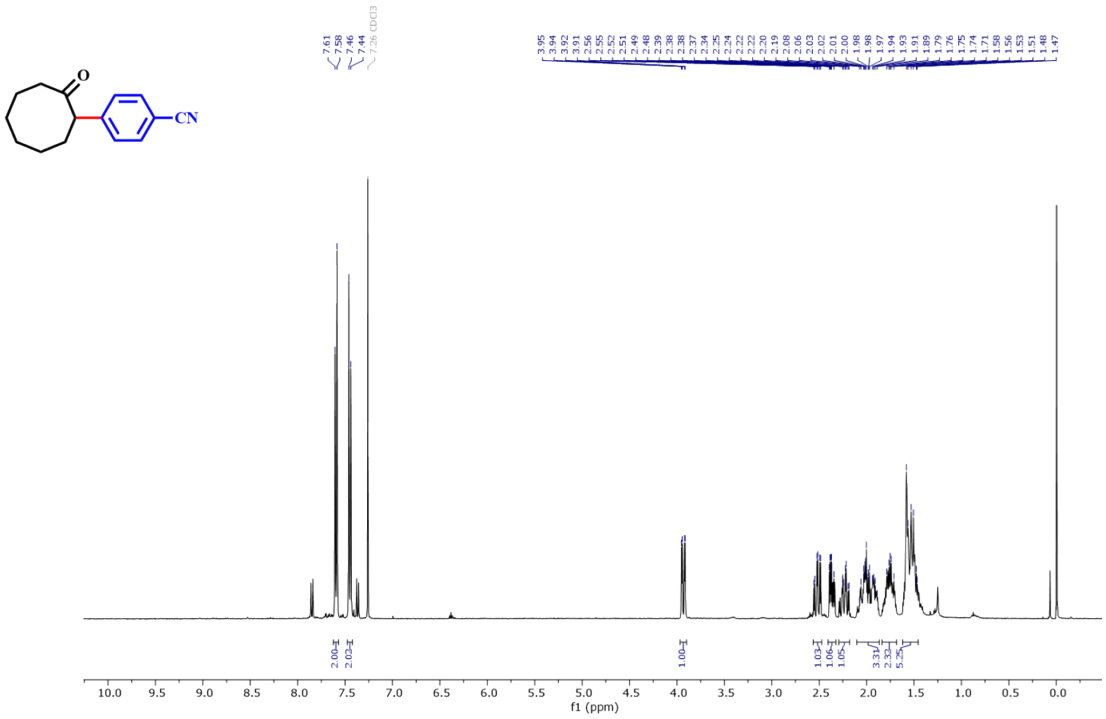
^{13}C NMR spectrum (101 MHz) of **5i** in $CDCl_3$



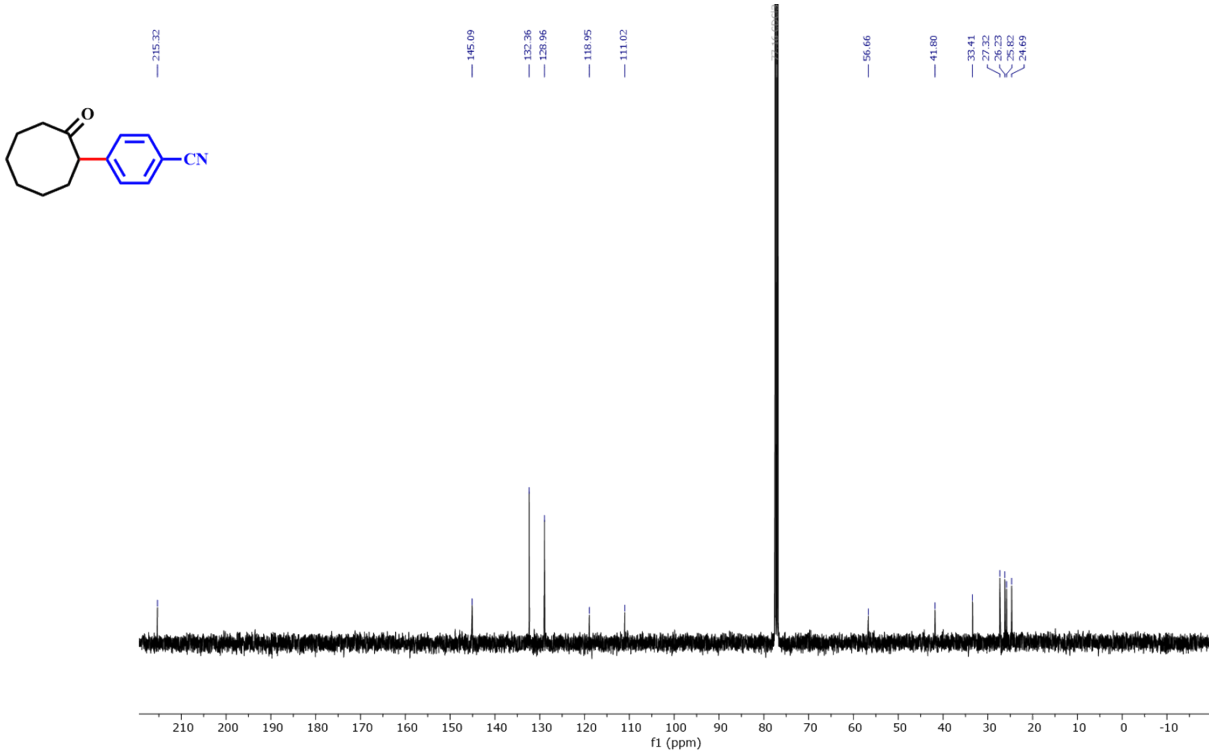
^{1}H NMR spectrum (400 MHz) of **5j** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of **5j** in $CDCl_3$



^{1}H NMR spectrum (400 MHz) of **5k** in $CDCl_3$



^{13}C NMR spectrum (101 MHz) of **5k** in $CDCl_3$

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