Electronic Supplementary Material (ESI) for ChemComm.

Monotrifluoroacetoxyborane-amines: Chemoselective reagents for challenging reductive aminations

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Supporting Information

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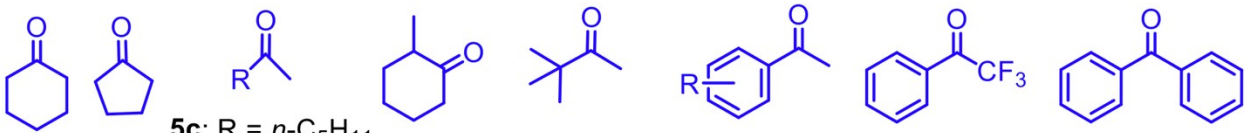
Numbering Legend for carbonyls

Aldehydes

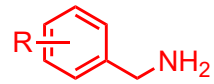
3a: R = Ph
3b: R = Chx
3c: R = *n*-pent
3d: R = 4-Cl-Ph
3e: R = 4-MeO-Ph

Ketones



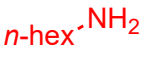
5a **5b** **5c**: R = *n*-C_5H_{11} **5d**: R = Me **5e** **5f** **5g**: R = H **5h**: R = *p*-OMe **5i**: R = *p*-CF_3 **5j** **5k**

Numbering Legend for Amines

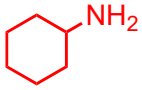


4a: R = *H*
4b: R = *o*-OMe
4c: R = *m*-OMe
4d: R = *p*-OMe
4e: R = *p*-F
4f: R = *p*-Cl

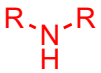
4g



4h

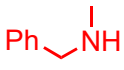


4i

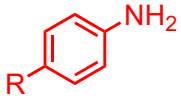


4j: R = *n*-Pr
4r: R = *n*-Bu

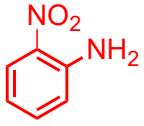
4k



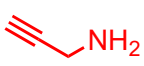
4l



4m: R = H
4n: R= CN
4o: R= NO2
4q: R = OMe

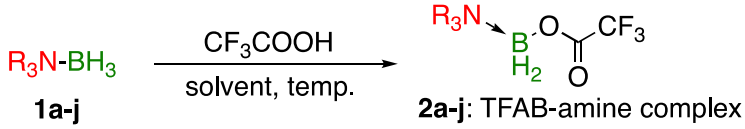


4p



4s

Table SI-1. Optimization of the synthesis of TFAB-amine complexes



1a-j **2a-j**: TFAB-amine complex

$R_3N•BH_3$ / R_3N		Eq. of TFA	Solvent (1 M)	Temperature	Time (h)	TFAB-amine complex	
1a	Ammonia	1 eq.	THF	RT	1	**2a**	$CF_3COO\text{-}BH_2•NH_3$
		1 eq.	DCM	RT	2		
		1 eq.	Toluene	RT	1		
		2 eq.	THF	RT	1		
		3 eq.	THF	RT	1		
1c	Benzylamine	1 eq.	THF	RT	18	**2c**	$CF_3COO\text{-}BH_2•NH_2CH_2Ph$
		1 eq.	Toluene	RT	2		
1b	Methylamine	1 eq.	THF	RT	20	**2b**	$CF_3COO\text{-}BH_2•NH_2Me$
		1 eq.	Toluene	RT	2		
1f	Piperidine	1 eq.	THF	reflux	6	**2f**	$CF_3COO\text{-}BH_2•$piperidine
		1 eq.	Toluene	RT	4		
1d	Dimethylamine	1 eq.	THF	reflux	4	**2d**	$CF_3COO\text{-}BH_2•NHMe_2$
		1 eq.	Toluene	RT	4		
1e	Diisopropylamine	1 eq.	THF	reflux	4	**2e**	$CF_3COO\text{-}BH_2•NH(i\text{-}Pr)_2$
		1 eq.	Toluene	RT	4		
1g	Trimethylamine	1.5 eq.	THF	reflux	12	**2g**	NR
		1.5 eq.	Toluene	RT	4		$CF_3COO\text{-}BH_2•NEt_3$
1h	Triethylamine	1.5 eq.	THF	reflux	12	**2h**	NR
		1.5 eq.	Toluene	RT	4		$CF_3COO\text{-}BH_2•NMe_3$
1i	Pyridine	1 eq.	THF	RT	-	**2i**	$CF_3COO\text{-}BH_2•$Pyridine
		1 eq.	Toluene	RT	2		
1j	2-Picoline	1 eq.	THF	RT	-	**2j**	$CF_3COO\text{-}BH_2•$2-Picoline
		1 eq.	Toluene	RT	4		

General Experimental Procedure

General Information: ^{11}B, ^{19}F, ^{13}C, and ^{1}H NMR spectra were recorded at room temperature, on a Varian INOVA 300 MHz NMR spectrophotometer. Chemical shifts (δ values) are reported in parts per million relative to $BF_3.Et_2O$ f{Peterson, 2002 #173} or ^{11}B NMR spectra. PMR spectral data are reported as: δ value, multiplicity (s=singlet, d=doublet, t=triplet, q=quartet, p=pentet, h=hextet, m=multiplet, br=broad) with integration. All solvents for routine isolation of products were reagent-grade. All amines and carbonyls were purchased from commercial sources and used without further purification.

Synthesis of TFAB-ammonia complex: Anhydrous THF (3 mL) was added to ammonia borane (3 mmol, 1 equiv.) in a 15mL round bottom flask. Trifluoroacetic acid (3 mmol, 1 equiv.) was added to the above solution at 0 °C and the resulting reaction mixture was stirred for 1 hour at room temperature. The completion of reaction was confirmed by ^{11}B-NMR spectroscopy as indicated by the disappearance of the δ - 22 ppm peak due to ammonia borane and the appearance of δ -5 ppm peak due to TFAB-NH_3.

Preparation of Crystal for X-ray crystallography: 1.5 mmol of 18-crown-6 was added to 1M THF solution containing 3 mmol of TFAB-NH_3. The mixture was stirred for 5 min and filtered. The clear solution was kept in a small vial inside a larger vial containing hexane at RT for a week to get colorless transparent block crystals. Details of crystal structure are described in X-Ray crystallography data (vide infra).

Synthesis of TFAB-amine complex: Anhydrous toluene (3 mL) was added to the respective amine boranes (3 mmol, 1 equiv.) in a 15mL round bottom flask. Trifluoroacetic acid (4.5 mmol, 1.5 equiv.) was added to the above solution at 0 °C and the resulting reaction mixture was stirred at room temperature for 4-6 h. The completion of reaction was confirmed by ^{11}B-NMR spectroscopy as indicated by the disappearance of the amine borane peak (δ -13 to -19 ppm) and appearance of the TFAB-amine peak (δ 0 to -5 ppm). Excess trifluoroacetic acid was then removed in vacuo to obtain the TFAB-amine complex.

Reductive amination of ketones with TFAB-ammonia:

Procedure A: To a 1M solution of TFAB-ammonia (3.3 mmol, 1.1 equiv.) in THF, ketone (6 mmol, 2 equiv.) and the amine (6 mmol, 2 equiv.) were added. The reaction mixture was stirred for 12-24 hrs at room temperature. Upon completion of the reaction, as revealed by TLC, the resulting mixture was quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4

and concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

Procedure B: To a 1M solution of TFAB-ammonia (3.3 mmol, 1.1 equiv.) in THF, ketone (9 mmol, 3 equiv.) and the amine (6 mmol, 2 equiv.) were added. The reaction mixture was refluxed for 6-12 hrs. Upon completion of the reaction, as revealed by TLC, the resulting mixture was cooled to RT and quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

Reductive amination of aldehydes with TFAB-NEt_3

Procedure C: To a 1M solution of TFAB-NEt_3 (3.3 mmol, 1.1 equiv.) in Toluene, aldehyde (3 mmol, 1 equiv.) and the amine (3.3 mmol, 1.1 equiv.) were added. The reaction mixture was stirred at 60 ºC for 12 h. Upon completion of the reaction, as revealed by TLC, the resulting mixture was cooled to RT and quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

Reductive amination of aromatic ketones with TFAB-NEt_3

Procedure D: To a 1M solution of TFAB-NEt_3 (3.3 mmol, 1.1 equiv.) in Toluene, ketone (3 mmol, 1 equiv.) and the amine (3 mmol, 1 equiv.) were added. The reaction mixture was stirred at 80 ºC for 12-18 h. Upon completion of the reaction, as revealed by TLC, the resulting mixture was cooled to RT and quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the crude product, which was purified by silica gel flash chromatography to obtain the desired product.

Reduction of Imines with **TFAB-NH_3** and **TFAB-Et_3N**

Reduction of N-benzylidenebenzylamine with TFAB-NH_3 (2a):

N-benzylidenebenzylamine (3 mmol) was synthesised by stirring benzaldehyde (3 mmol) and benzylamine (1 equiv.) for 12h in presence of molecular sieves. A 1M solution of TFAB-ammonia (3 mmol, 1 equiv.) in THF was added to this mixture and the reaction was stirred for 6 hr at room temperature. The reaction was quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the product. ^{1}HNMR analysis shows a complete reduction of starting imine to corresponding amine (dibenzyl amine). This shows the efficacy of TFAB-NH_3 (**2a**) as a reagent for imine reduction.

Reduction of N-benzylidenebenzylamine with TFAB-Et_3N (2h):

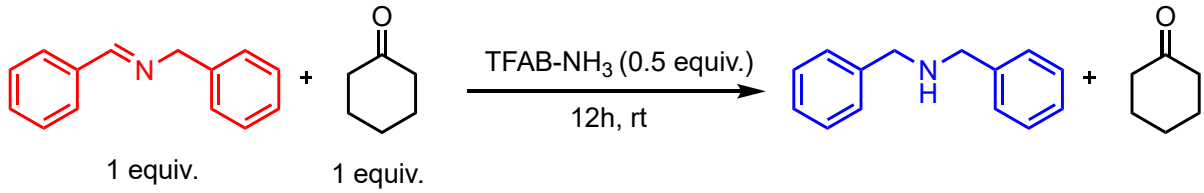
N-benzylidenebenzylamine (3 mmol) was synthesised by stirring benzaldehyde (3 mmol) and benzylamine (3 mmol) for 12h in presence of molecular sieves. A 1M solution of TFAB-NEt_3 (3 mmol, 1 equiv.) in toluene was added to this mixture and the reaction was stirred at 60 ºC for 6 h. Upon completion the resulting mixture was cooled to RT and quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the product. ^{1}HNMR analysis shows a complete reduction of starting imine to corresponding amine (dibenzyl amine). This shows the capability of TFAB-Et_3N (**2h**) as an efficient reagent for imine reduction.

Hydride analysis* of TFAB-NH_3 + Benzylamine polymer

A equimolar mixture of TFAB-NH_3 and benzylamine in 1M THF is prepared in a 3-dram vial with a septum inlet fitted with a connecting tube. The connecting tube was attached to an analytical gas burette filled with CuSO4 solution. 3 M HCl was syringed into the vial, dropwise, till no further gas evolution was observed. The gas evolved was measured using the analytical gas burette. The temperature of the reaction was maintained at 25 °C.

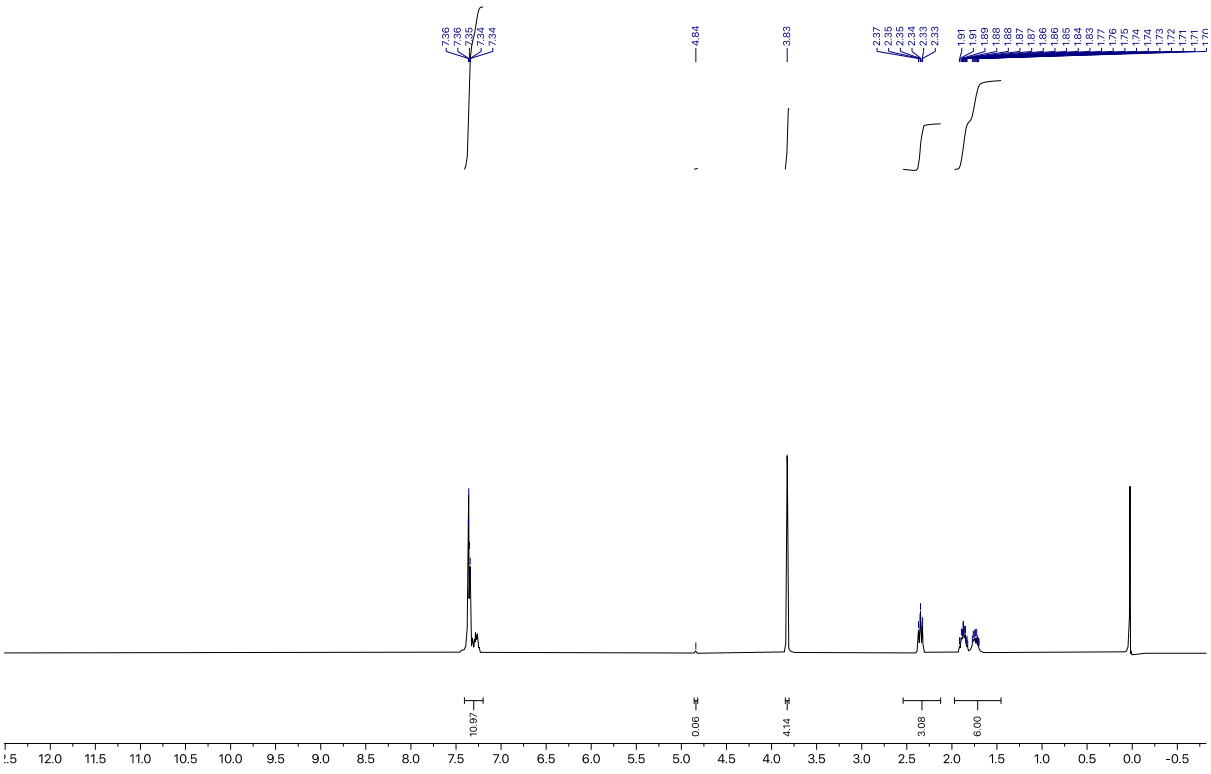
*(H. C. Brown, Organic Syntheses Via Boranes, Wiley, New York, 1975, ch. 9.)

Competitive reduction of ketone vs imine with TFAB-NH_3 (2a):



1 equiv. 1 equiv.

N-benzylidenebenzylamine was as mentioned in the above procedure. N-benzylidenebenzylamine (3 mmol, 1 equiv.) and cyclohexanone (3 mmol, 1 equiv.) were added to a 1M solution of TFAB-NH_3 (1.5 mmol, 0.5 equiv.) in THF. The reaction mixture was stirred for 12 hrs at room temperature. Upon completion of the reaction, the mixture was quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the crude product. ^{1}HNMR analysis shows that N-benzylidenebenzylamine (δ 4.84 ppm) is selectively reduced to dibenzyl amine (δ 3.83 ppm) and cyclohexanone remains unreacted with no reduction to cyclohexanol observed. Hence, TFAB-NH_3 exclusively reduces imines in presence of ketones.



SI Figure 1: ^{1}HNMR analysis of competitive reduction of ketone vs imine with **2a**.

Reductive amination with polyaminoboranes: To a 1M solution of TFAB-amine (3mmol) in toluene, amine (6 mmol, 2 equiv.) was added. The reaction was stirred for 15 mins to obtain the corresponding aminoborane polymer. To this 1 equiv. of benzylamine and cyclohexanone were added and stirred for 24h at 60 °C. Upon completion the resulting mixture was cooled to RT and quenched with NaOH (5 mL, 3 M), diluted with water (10 mL), and extracted with diethyl ether (3 × 15 mL). The combined organic extracts were washed with brine (1 × 45 mL), dried over anhydrous Na_2SO_4 and concentrated in vacuo to obtain the product.

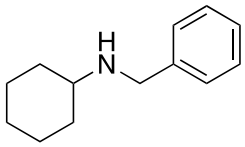
Comparison of reductive amination of acetophenone with benzylamine with reagents from literature.

No.	Reagent	Reaction condition	Yield
1	TFAB-NEt_3	24h, 80 °C	99%
2	pyridine-BH_3	-	10% [29]
3	2-picoline-BH_3	72h, RT	87% [30]
4	5-ethyl-2-methylpyridine-BH_3	4 h, 50 °C	70% [29]
5	$PhMe_2SiH$ + $B(C_6F_5)_3$ catalyst	48h, 100 °C	75% [31]
6	Hydrosilatrane	16h, 70 °C	50% [32]
7	1,2,3-triazole-boranes	5h, 60 °C	87% [33]

Green Matrix calculation - Process mass intensity (PMI)

(A) Reductive amination with TFAB-NH_3

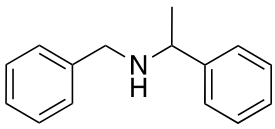
N-benzylcyclohexanamine (**6aa**)



		Equivalents used	mmol	MW (g/mol)	g
Reactants	Cyclohexanone	2	6	98.14	0.589
	Benzylamine	2	6	107.15	0.643
	TFAB-NH_3	1.1	3.3	142.87	0.471
Auxiliary	THF				2.66
	NaOH			38	7.2
	H_2O			18	10
	diethyl ether				35
	Na_2SO_4				10
Product	N-benzylcyclohexanamine	0.89	5.34	189.30	1.010
Calculation	Total mass in process				67.573
	Mass of product				1.010
	%Yield				89
	PMI				**66.9**

(B) Reductive amination with TFAB-NEt_3

N-benzyl-1-phenylethan-1-amine (**6ga**)



		Equivalents used	mmol	MW (g/mol)	g
Reactants	Acetophenone	1	3	120.15	0.360
	Benzylamine	1	3	107.15	0.643
	TFAB-NH_3	1.1	3.3	227.03	0.749
Auxiliary	Toluene				2.59
	NaOH			38	7.2
	H_2O			18	10
	diethyl ether				35
	Na_2SO_4				10
Product	N-benzyl-1-phenylethan-1-amine	0.99	2.97	211.31	0.628
Calculation	Total mass in process				67.17
	Mass of product				0.628
	%Yield				99
	PMI				**106.96**

X-Ray crystallography:

Data were collected, reflections were indexed and processed, and the files scaled and corrected for absorption using APEX3 [1] and SADABS [2]. The space groups were assigned using XPREP within the SHELXTL suite of programs [3,4] and solved by direct methods using ShelXS [4] or dual methods using ShelXT [5] and refined by full matrix least squares against F^2 with all reflections using Shelxl2018 [6] using the graphical interface Shelxle [7]. If not specified otherwise H atoms attached to carbon, boron and nitrogen atoms as well as hydroxyl hydrogens were positioned geometrically and constrained to ride on their parent atoms. C-H bond distances were constrained to 0.95 Å for aromatic and alkene C-H and CH_2 and alkyne C-H moieties, and to 1.00, 0.99 and 0.98 Å for aliphatic C-H, CH_2 and CH_3 moieties, respectively. B-H bond distances were constrained to 1.00 Å for pyramidal (sp^3 hybridized) R_3B-H moieties. N-H bond distances were constrained to 0.88 Å for planar (sp^2 hybridized) N-H, $N\text{-}H^+$ and NH_2 groups. N-H bond distances were constrained to 0.91 Å for pyramidal (sp^3 hybridized) ammonium NH_2^+ and NH_3^+ groups. O-H distances of alcohols were constrained to 0.84 Å. Methyl CH_3, ammonium NH_3^+ and hydroxyl H atoms were allowed to rotate but not to tip to best fit the experimental electron density. H atoms of pyramidalized R_2NH and RNH_2 units were refined and N-H distances were restrained to 0.88(2) Å. Water H atom positions were refined and O-H distances were restrained to 0.84(2) Å. Where necessary, water H···H distances were restrained to 1.36(2) Å, and H atom positions were further restrained based on hydrogen bonding considerations. $U_{iso}(H)$ values were set to a multiple of $U_{eq}(C)$ with 1.5 for CH_3, NH_3^+ and OH, and 1.2 for C-H, CH_2, B-H, N-H and NH_2 units, respectively.

Complete crystallographic data, in CIF format, have been deposited with the Cambridge Crystallographic Data Centre. CCDC-2175383 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

[1] Bruker (2019). Apex3 v2019.1-0, SAINT V8.40A, Bruker AXS Inc.: Madison (WI), USA.
[2] Krause, L., Herbst-Irmer, R., Sheldrick, G.M. & Stalke, D. (2015). J. Appl. Cryst. 48, 3-10.
[3] SHELXTL suite of programs, Version 6.14, 2000-2003, Bruker Advanced X-ray Solutions, Bruker AXS Inc., Madison, Wisconsin: USA
[4] Sheldrick, G.M. A short history of SHELX. Acta Crystallogr A. 2008, 64(1), 112–122.
[5] Sheldrick, G. M., "SHELXT--Integrated space-group and crystal-structure determination", Acta Crystallogr A. 2015, A71, 3-8.
[6] a) Sheldrick, G.M. University of Göttingen, Germany, 2018. b) Sheldrick, G.M. Crystal structure refinement with SHELXL. Acta Crystallogr Sect C Struct Chem. 2015, 71(1), 3–8.
[7] Hübschle, C.B., Sheldrick, G.M. & Dittrich, B. ShelXle: a Qt graphical user interface for SHELXL. J. Appl. Crystallogr. 2011, 44(6), 1281–1284.

Table SI-2. Experimental details

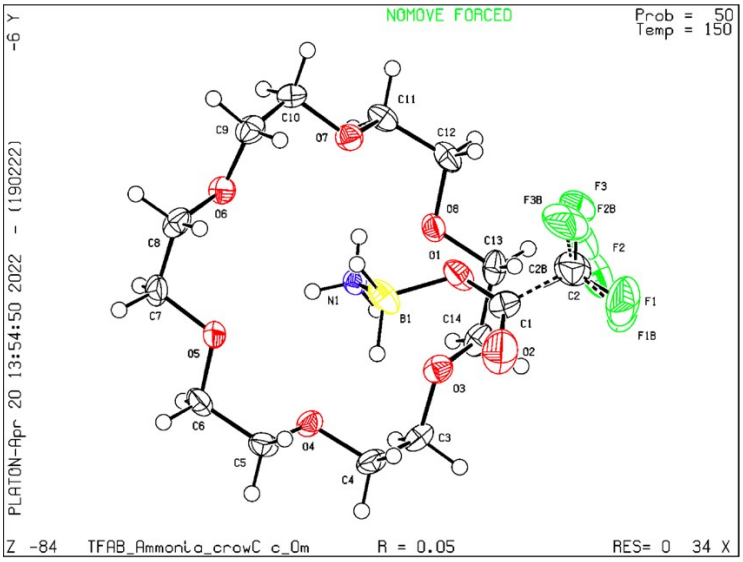
For all structures: monoclinic, $P2_1/n$, $Z = 4$. Experiments were carried out with Mo $K\alpha$ radiation using a CCD area detector.

	(1957962)	(I)
Crystal data		
Chemical formula	$C_8H_{19}BClNO_5$	$C_8H_{18}BCl_2NO_5$
M_r	255.50	289.94
Temperature (K)	103	153
a, b, c (Å)	10.312 (2), 12.187 (2), 10.6044 (19)	8.6163 (11), 11.9140 (15), 13.6933 (16)
β (°)	100.686 (6)	99.807 (3)
V (Å^3)	1309.5 (4)	1385.1 (3)
μ (mm^{-1})	0.30	0.48
Crystal size (mm)	0.42 × 0.40 × 0.38	0.36 × 0.24 × 0.22
Data collection		
Absorption correction	Multi-scan *SADABS* V2014/4 (Bruker AXS Inc.)	Multi-scan *SADABS* V2014/5 (Bruker AXS Inc.)
T_{min}, T_{max}	0.53, 0.90	0.78, 0.90
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14073, 2370, 1779	13711, 3186, 1698
R_{int}	0.134	0.070
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.600	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.075, 0.203, 1.02	0.056, 0.148, 0.91
No. of reflections	2370	3186
No. of parameters	145	313
No. of restraints	0	702
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.52, -0.53	0.45, -0.46

Computer programs: *APEX2* (Bruker AXS Inc., 2014), *SAINT* V8.34A (Bruker AXS Inc., 2013), *SHELXT* (Bruker AXS Inc., 2014), *SHELXL2018*/3 (Sheldrick, 2018), *SHELXL2014*/7 (Sheldrick, 2014), *XP* (Bruker AXS Inc., 2014).

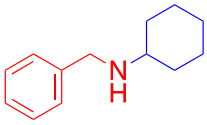
Table SI-3. Selected geometric parameters: bond lengths (Å), bond angles (º)

B1—O1	1.505 (4)	C4—O3	1.428 (4)
B1—N1	1.579 (4)	C4—C5	1.497 (5)
C1—C2	1.504 (5)	C5—O4	1.431 (4)
C1—Cl1	1.778 (3)	C6—O4	1.433 (4)
C2—O2	1.217 (4)	C6—C7	1.492 (5)
C2—O1	1.303 (4)	C7—O5	1.430 (4)
C3—O3	1.426 (4)	C8—O5	1.430 (4)
C3—C8^{i}	1.490 (4)		
O1—B1—N1	102.7 (2)	O4—C6—C7	109.6 (3)
C2—C1—Cl1	117.6 (2)	O5—C7—C6	107.8 (2)
O2—C2—O1	125.5 (3)	O5—C8—C3^{i}	109.0 (2)
O2—C2—C1	116.9 (3)	C2—O1—B1	118.2 (2)
O1—C2—C1	117.5 (3)	C3—O3—C4	110.8 (2)
O3—C3—C8^{i}	109.0 (3)	C5—O4—C6	110.3 (2)
O3—C4—C5	108.9 (2)	C8—O5—C7	110.8 (2)
O4—C5—C4	108.2 (3)		



SI Figure 2: Crystal structure of TFAB-NH_3 (**2a**) co-crystalized with 18-crown ether

Experimental: NMR Data

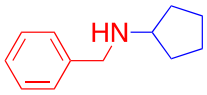


N-benzylcyclohexanamine (**6aa**)

The product was prepared using *procedure A* and isolated in 89% yield (1.0 g, 5.34 mmol) as a pale yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[1].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.05 (m, 5H), 3.77 (d, *J* = 1.2 Hz, 2H), 2.45 (tt, *J* = 10.0, 3.6 Hz, 1H), 1.96 – 1.83 (m, 2H), 1.80 – 1.55 (m, 3H), 1.35 – 1.00 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 139.4, 132.3, 129.3, 128.4, 56.2, 50.3, 33.7, 26.3, 25.1.

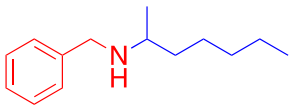


N-benzylcyclopentanamine (**6ba**)

The product was prepared using *procedure A* and isolated in 83% yield (0.871 g, 4.98 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[1].

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.25 (d, *J* = 8.9 Hz, 5H), 3.72 (d, *J* = 9.4 Hz, 2H), 3.06 (p, *J* = 6.6 Hz, 1H), 1.95 – 1.20 (m, 8H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 138.9, 132.4, 129.4, 128.4, 59.2, 52.0, 33.2, 24.2.

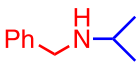


N-benzyl-3,3-dimethylbutan-2-amine (**6ca**)

The product was prepared using *procedure A* and isolated in 93% yield (1.148 g, 5.58 mmol) as a pale yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[3].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 (d, *J* = 4.3 Hz, 4H), 7.28 – 7.17 (m, 1H), 4.18 – 3.40 (m, 2H), 2.89 – 2.29 (m, 1H), 1.56 – 1.38 (m, 1H), 1.42 – 1.18 (m, 7H), 1.08 (d, *J* = 6.2 Hz, 3H), 0.94 – 0.81 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 140.8, 128.3, 128.0, 126.7, 52.6, 51.5, 37.2, 32.2, 25.8, 22.8, 20.5, 14.2.

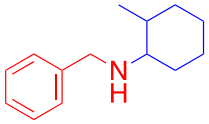


N-benzylpropan-2-amine (**6da**)

The product was prepared using *procedure A* and isolated in 88% yield (0.787 g, 5.3 mmol) as a yellow liquid after purification via column chromatography on silica gel using hexane as an eluent. The spectral data matched with those reported in the literature[5].

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.38 – 7.20 (m, 5H), 3.79 (s, 2H), 2.86 (p, *J* = 6.2 Hz, 1H), 1.69 (s, 1H), 1.11 (d, *J* = 6.2 Hz, 6H).

^{13}C{1H} NMR (75 MHz, $CDCl_3$) δ 134.9, 129.3, 128.6, 128.0, 49.3, 48.1, 20.6.

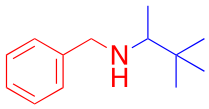


N-benzyl-2-methylcyclohexan-1-amine (**6ea**)

The product was prepared using *procedure B* and isolated in 91% yield as a trans/cis (91:9) mixture (1.108 g, 5.5 mmol) as a yellow liquid after purification via column chromatography on silica gel using hexane as an eluent. The spectral data matched with those reported in the literature.[4]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 – 6.98 (m, 5H), 3.96 – 3.64 (m, 2H), 2.65 (dt, *J* = 8.0, 4.0 Hz, 1H), 1.96 (dq, *J* = 6.7, 3.4 Hz, 1H), 1.81 – 1.17 (m, 8H), 0.95 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 141.1, 128.2, 128.0, 126.6, 57.7, 51.2 33.0, 31.0, 28.4, 23.6, 22.5, 14.1.

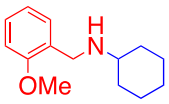


N-benzyl-3,3-dimethylbutan-2-amine (**6fa**)

The product was prepared using *procedure A* and isolated in 82% yield (0.935 g, 4.92 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[2].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.18 (m, 5H), 3.94 (d, *J* = 13.2 Hz, 1H), 3.66 (d, *J* = 13.2 Hz, 1H), 2.36 – 2.24 (m, 1H), 1.09 (d, *J* = 6.4 Hz, 3H), 0.87 (s, 9H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 141.2, 128.2, 128.1, 126.6, 61.2, 52.7, 34.5, 26.6, 14.8.

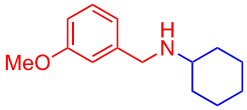


N-(2-methoxybenzyl)cyclohexanamine (**6ab**)

The product was prepared using *procedure A* and isolated in 75% yield (0.977 g, 4.5 mmol) as a orange-yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[15].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.16 (m, 2H), 6.96 – 6.81 (m, 2H), 3.82 (d, *J* = 6.6 Hz, 5H), 2.43 (tt, *J* = 9.7, 3.6 Hz, 1H), 1.98 – 1.84 (m, 2H), 1.80 – 1.54 (m, 4H), 1.35 – 1.03 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 157.5, 129.6, 128.8, 127.9, 120.3, 110.1, 55.9, 55.3, 46.3, 33.6, 26.3, 25.1.

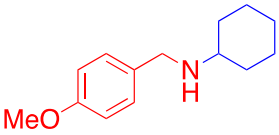


N-(3-methoxybenzyl)cyclohexanamine (**6ac**)

The product was prepared using *procedure A* and isolated in 86% yield (1.130 g, 5.2 mmol) as a yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[6].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 – 7.67 (m, 4H), 3.8 (m, 5H), 2.4-2.5 (m, 1H), 1.96 – 1.83 (m, 2H), 1.80 – 1.55 (m, 3H), 1.35 – 1.00 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 159.6, 142.6, 129.3, 120.3, 56.2, 55.2, 51.1, 33.7, 26.3, 25.1.

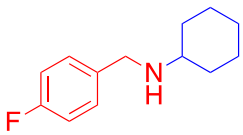


N-(4-methoxybenzyl)cyclohexanamine (**6ad**)

The product was prepared using *procedure A* and isolated in 92% yield (1.314 g, 5.5 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[6].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 – 7.18 (m, 2H), 6.94 – 6.77 (m, 2H), 3.79 (s, 3H), 3.74 (s, 2H), 2.47 (tt, *J* = 10.0, 3.7 Hz, 1H), 1.98 – 1.84 (m, 2H), 1.80 – 1.67 (m, 2H), 1.68 – 1.54 (m, 1H), 1.33 – 0.92 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 158.3, 133.0, 129.2, 113.7, 56.1, 55.3, 50.5, 33.6, 26.3, 25.1.

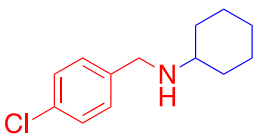


N-(4-fluoro)cyclohexanamine (**6ae**)

The product was prepared using *procedure A* and isolated in 79% yield (0.973 g, 4.7 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[6].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.20 (m, 2H), 7.08 – 6.91 (m, 2H), 3.76 (s, 2H), 2.60 – 2.38 (m, 1H), 1.96 – 1.53 (m, 5H), 1.28 – 1.11 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 163.3, 160.0, 136.6, 129.5, 129.4, 115.2, 114.9, 56.2, 50.4, 33.7, 26.3, 25.1.

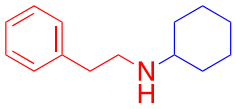


N-(4-chloro)cyclohexanamine (**6af**)

The product was prepared using *procedure A* and isolated in 84% yield (1.124 g, 5 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[7].

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.52 – 7.09 (m, 4H), 3.77 (s, 2H), 2.45 (tt, *J* = 10.1, 3.7 Hz, 1H), 1.89 (dd, *J* = 11.8, 3.8 Hz, 2H), 1.81 – 1.69 (m, 2H), 1.66 – 1.54 (m, 1H), 1.38 – 0.87 (m, 5H).

^{13}C{1H} NMR (75 MHz, $CDCl_3$) δ 139.4, 132.3, 129.3, 128.3, 56.1, 50.3, 33.6, 26.2, 25.1.

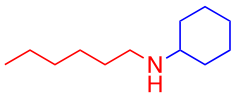


N-phenethylcyclohexanamine (**6ag**)

The product was prepared using *procedure A* and isolated in 81% yield (0.987 g, 4.9 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as an eluent. The spectral data matched with those reported in the literature[8].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 – 7.10 (m, 5H), 3.03 – 2.70 (m, 4H), 2.42 (tt, *J* = 10.3, 3.7 Hz, 1H), 2.01 – 1.50 (m, 5H), 1.47 – 0.90 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 140.1, 128.6, 128.4, 126.0, 56.8, 48.4, 36.8, 33.7, 26.3, 25.2.

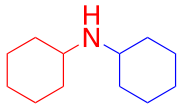


N-hexylcyclohexanamine (**6ah**)

The product was prepared using *procedure B* and isolated in 86% yield (0.952 g, 5.2 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5) as an eluent. The spectral data matched with those reported in the literature[9].

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.57 (t, *J* = 7.2 Hz, 2H), 2.36 (tt, *J* = 10.3, 3.7 Hz, 1H), 1.92 – 1.52 (m, 2H), 1.42 (q, *J* = 7.5 Hz, 2H), 1.36 – 1.20 (m, 3H), 1.25 – 0.90 (m, 12H), 0.90 – 0.77 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 57.0, 47.2, 33.8, 31.9, 30.6, 27.3, 26.3, 25.2, 22.7, 14.2.

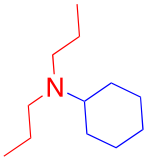


N-hexylcyclohexanamine (**6ai**)

The product was prepared using *procedure B* and isolated in 71% yield (0.771 g, 4.26 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[11].

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.53 (ttd, *J* = 9.9, 3.8, 1.7 Hz, 2H), 1.92 – 1.52 (m, 10H), 1.34 – 0.91 (m, 10H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 53.1, 34.4, 26.3, 25.4.

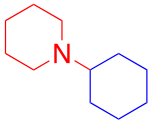


N-hexylcyclohexanamine (**6aj**)

The product was prepared using *procedure B* and isolated in 78% yield (0.721 g, 4.69 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[11].

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.42 (tt, *J* = 10.6, 3.9 Hz, 1H), 2.07 – 1.07 (m, 24H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 60.3, 36.9, 32.1, 29.3, 27.9, 25.8, 24.3.

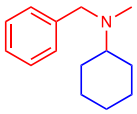


N-hexylcyclohexanamine (**6ak**)

The product was prepared using *procedure D* for 18h and isolated in 72% yield (0.856 g, 4.32 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[12].

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.61 – 2.43 (m, 4H), 2.22 (dt, *J* = 5.8, 2.6 Hz, 1H), 1.90 – 0.81 (m, 16H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 64.4, 53.1, 50.1, 34.5, 28.9, 26.6, 26.6, 26.3, 25.4, 25.0.

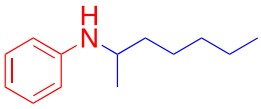


N-benzyl-N-methylcyclohexanamine (**6al**)

The product was prepared using *procedure B* and isolated in 82% yield (0.998 g, 4.92 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[11].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 – 6.93 (m, 5H), 3.57 (s, 2H), 2.09 – 1.75 (m, 1H), 2.19 (s, 3H), 1.64 (dtd, *J* = 11.6, 3.5, 2.1 Hz, 4H), 1.43 – 1.02 (m, 6H).

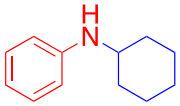
^{13}C NMR (75 MHz, $CDCl_3$) δ 140.3, 128.7, 128.1, 126.6, 62.6, 57.9, 37.8, 28.8, 26.6, 26.2.



N-(heptan-2-yl)aniline (**6cm**)

The product was prepared using *procedure A* and isolated in 91% yield (1.050 g, 5.46 mmol) as a brown oil after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[13].

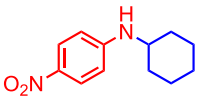
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.10 (m, 2H), 6.73 – 6.53 (m, 3H), 3.47 (m, 2H), 1.64 – 1.38 (m, 1H), 1.44 – 1.30 (m, 3H), 1.35 – 1.23 (m, 4H), 1.19 (dd, *J* = 6.2, 1.4 Hz, 3H), 0.96 – 0.85 (m, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 147.6, 129.2, 116.7, 113.0, 48.5, 37.3, 32.0, 26.0, 22.8, 20.9, 14.2.



N-cyclohexylaniline (**6am**)

The product was prepared using *procedure A* and isolated in 89% yield (0.934 g, 5.34 mmol) as a yellow oil after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[11].

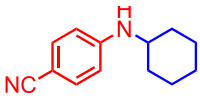
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.10 (m, 2H), 6.75 – 6.62 (m, 1H), 6.67 – 6.52 (m, 2H), 3.56 – 3.50 (m, 1H), 3.28 (tt, *J* = 10.1, 3.8 Hz, 1H), 2.16 – 2.01 (m, 2H), 1.87 – 1.60 (m, 3H), 1.50 – 1.08 (m, 5H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 147.3, 129.2, 116.8, 113.1, 51.7, 33.6, 26.1, 25.2.



N-cyclohexyl-4-nitroaniline (**6ao**)

The product was prepared using *procedure B* and isolated in 71% yield (0.937 g, 4.26 mmol) as a yellow solid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as the eluent. The spectral data matched with those reported in the literature.[15]

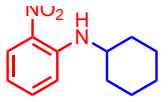
^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 (d, *J* = 9.2 Hz, 2H), 6.49 (d, *J* = 9.2 Hz, 2H), 4.43 (s, 2H), 2.12 – 1.98 (m, 3H), 1.86 – 1.60 (m, 5H), 1.50 – 1.13 (m, 9H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 152.4, 137.2, 126.5, 111.1, 51.6, 32.9, 25.7, 24.9.



N-cyclohexyl-4-cyanoaniline (**6an**)

The product was prepared using *procedure B* and isolated in 78% yield (0.936 g, 4.68 mmol) as a yellow solid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as the eluent. The spectral data matched with those reported in the literature.[15]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.17 (tdd, *J* = 7.5, 1.2, 0.6 Hz, 1H), 6.92 – 6.82 (m, 1H), 6.80 – 6.69 (m, 2H), 3.84 (s, 1H), 3.21 (tt, *J* = 10.1, 3.8 Hz, 1H), 2.09 – 1.94 (m, 2H), 1.88 – 1.59 (m, 3H), 1.47 – 1.06 (m, 5H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 147.5, 129.8, 119.9, 119.6, 117.4, 114.9, 112.8, 51.5, 33.1, 25.9, 25.0.

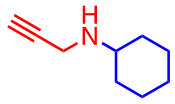


N-cyclohexyl-2-nitroaniline (**6ap**)

The product was prepared using *procedure C* for 18h and isolated in 82% yield (0.541 g, 2.46 mmol) as a pale yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc (1:1) as an eluent. The spectral data matched with those reported in the literature.[15]

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (td, *J* = 7.1, 3.3 Hz, 2H), 7.39 (ddd, *J* = 8.3, 6.6, 1.4 Hz, 1H), 6.59 (ddt, *J* = 8.8, 6.9, 1.0 Hz, 1H), 3.51 (d, *J* = 8.9 Hz, 1H), 2.12 – 2.01 (m, 2H), 1.71 – 1.61 (m, 1H), 1.53 – 1.22 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 144.7, 135.9, 131.4, 127.0, 114.7, 114.1, 51.0, 32.8, 25.7, 24.7.

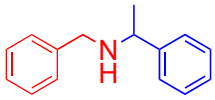


N-(prop-2-yn-1-yl)cyclohexanamine (6as)

The product was prepared using *procedure A* and isolated in 92% yield (0.756 g, 5.52 mmol) as a brown liquid after purification via column chromatography on silica gel using hexane/EtOAc 95:5 as the eluent. The spectral data matched with those reported in the literature.[15]

^{1}H NMR (300 MHz, $CDCl_3$) δ 3.41 (dd, *J* = 2.4, 0.8 Hz, 2H), 2.69 – 2.43 (m, 1H), 2.16 (td, *J* = 2.4, 0.8 Hz, 1H), 1.94 – 1.49 (m, 4H), 1.35 – 0.92 (m, 5H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 82.5, 70.9, 54.9, 35.2, 33.1, 26.2, 24.9.

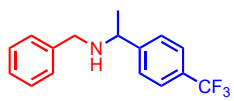


***N*-benzyl-1-phenylethan-1-amine** (**6ga**)

The product was prepared using *procedure D* and isolated in 99% yield (0.626 g, 2.97 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[3].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 – 7.21 (m, 10H), 3.86 (q, *J* = 6.6 Hz, 1H), 3.76 – 3.58 (m, 2H), 1.61 (s, 1H), 1.41 (dd, *J* = 6.6, 0.5 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 145.5, 140.6, 128.4, 128.3, 128.1, 126.9, 126.8, 126.7, 57.6, 51.8, 24.7.

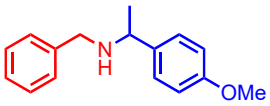


1-phenyl-N-(4-(trifluoromethyl)benzyl)ethan-1-amine (**6ia**)

The product was prepared using *procedure D* and isolated in 82% yield (0.686 g, 2.46 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[23].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.1 Hz, 2H), 7.51 (d, *J* = 8.1 Hz, 2H), 7.42 – 7.20 (m, 5H), 3.90 (q, *J* = 6.5 Hz, 1H), 3.72 – 3.55 (m, 2H), 1.64 (s, 1H), 1.38 (dd, *J* = 6.6, 1.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 149.7, 140.2, 128.4, 128.0, 127.0, 126.9, 125.4, 125.4, 57.3, 51.8, 24.7.

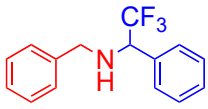


N-(4-methoxybenzyl)-1-phenylethan-1-amine (**6ha**)

The product was prepared using *procedure D* and isolated in 99% yield (0.715 g, 2.97 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[24].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 – 7.12 (m, 7H), 7.02 – 6.87 (m, 2H), 3.93 – 3.76 (m, 2H), 3.84 (s, 3H), 3.75 – 3.56 (m, 2H), 1.40 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 158.5, 140.7, 137.6, 128.3, 128.1, 127.7, 126.8, 113.8, 56.9, 55.3, 51.7, 24.7.

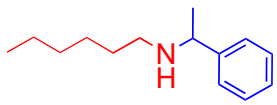


N-benzyl-2,2,2-trifluoro-1-phenylethan-1-amine (**6ja**)

The product was prepared using *procedure D* and isolated in 72% yield (0.552 g, 2.16 mmol) as a pale yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc (1:1) as an eluent. The spectral data matched with those reported in the literature.[6]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 – 7.35 (m, 1H), 4.99 (q, *J* = 6.8 Hz, 0H), 3.43 (s, 0H), 3.14 (s, 0H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 193.10, 166.10, 134.45, 129.21, 128.61, 128.55, 128.40, 127.44, 73.12, 72.70, 72.28, 71.86, 50.30.

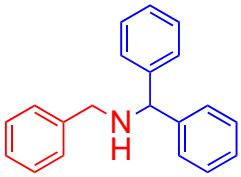


N-(1-phenylethyl)hexan-1-amine (**6gh**)

The product was prepared using *procedure D* and isolated in 87% yield (0.535 g, 2.61 mmol) as a pale blue liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[9].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 – 7.01 (m, 5H), 3.75 (q, *J* = 6.6 Hz, 1H), 2.59 – 2.34 (m, 2H), 1.57 – 1.17 (m, 14H), 0.88 (dt, *J* = 9.2, 4.5 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 145.8, 128.3, 126.7, 126.5, 58.5, 48.0, 31.9, 30.4, 27.2, 24.5, 22.7, 14.2.

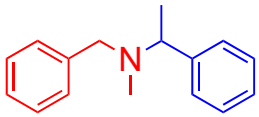


N-benzyl-1,1-diphenylmethanamine (**6ka**)

The product was prepared using *procedure D* and isolated in 90% yield (0.737 g, 2.7 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[25].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 – 6.87 (m, 15H), 4.94 (s, 1H), 3.83 (s, 2H), 1.94 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 140.4, 128.5, 128.4, 128.2, 127.4, 127.0, 126.9, 66.6, 52.0.

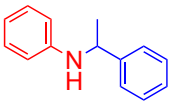


N-benzyl-N-methyl-1-phenylethan-1-amine (**6gl**)

The product was prepared using *procedure D* and isolated in 96% yield (0.648 g, 2.88 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[26].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 – 7.20 (m, 10H), 3.82 – 3.51 (m, 2H), 3.35 (d, *J* = 13.3 Hz, 1H), 2.19 (s, 3H), 1.47 (dt, *J* = 6.7, 0.8 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 144.1, 140.0, 128.7, 128.1, 127.6, 126.8, 126.7, 63.3, 59.0, 38.5, 18.6.

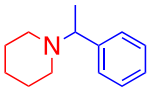


N-(1-phenylethyl)aniline (**6gm**)

The product was prepared using *procedure D* and isolated in 95% yield (0.561 g, 2.85 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (9:1) as an eluent. The spectral data matched with those reported in the literature[27].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 – 7.33 (m, 4H), 7.33 – 7.23 (m, 1H), 7.15 (t, *J* = 7.7 Hz, 2H), 6.70 (t, *J* = 7.3 Hz, 1H), 6.57 (d, *J* = 7.9 Hz, 2H), 4.54 (q, *J* = 6.7 Hz, 1H), 4.07 (s, 1H), 1.56 (d, *J* = 6.9 Hz, 3H)

^{13}C NMR (75 MHz, $CDCl_3$) δ 147.2, 145.2, 129.1, 128.6, 126.8, 125.8, 117.2, 113.3, 53.5, 25.2.

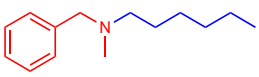


1-(1-phenylethyl)piperidine (**6gk**)

The product was prepared using *procedure D* and isolated in 86% yield (0.487 g, 2.58 mmol) as a pale yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc (8:2) as an eluent. The spectral data matched with those reported in the literature[28].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.17 (m, 5H), 3.47 – 3.34 (m, 1H), 2.39 (tp, *J* = 11.5, 5.2 Hz, 4H), 1.57 (dq, *J* = 10.7, 5.5 Hz, 6H), 1.49 – 1.35 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 143.8, 127.9, 127.7, 126.6, 65.3, 51.6, 26.4, 24.8, 19.6.

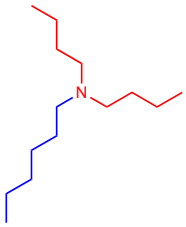


N-benzyl-N-methylhexan-1-amine (**7bl**)

The product was prepared using *procedure C* and isolated in 88% yield (0.541 g, 2.64 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[16].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.18 (m, 5H), 3.48 (s, 2H), 2.42 – 2.31 (m, 2H), 2.19 (s, 3H), 1.51 (qd, *J* = 6.7, 3.6 Hz, 2H), 1.41 – 1.20 (m, 6H), 0.95 – 0.84 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 139.2, 129.0, 128.1, 126.7, 62.4, 57.7, 42.4, 31.9, 27.5, 27.3, 22.8, 14.2.

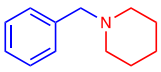


N,N-dibutylhexan-1-amine (**7br**)

The product was prepared using *procedure C* and isolated in 76% yield (0.485 g, 2.28 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. HRMS (CI) m/z: [M+H]+ calcd. for $C_{14}H_{32}N$ 214.2535, found 214.2535.

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.36 (tt, *J* = 7.9, 2.3 Hz, 6H), 1.68 – 1.11 (m, 16H), 0.89 (t, *J* = 7.1 Hz, 9H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 54.3, 54.0, 32.0, 29.3, 27.4, 27.1, 22.8, 20.9, 14.2.

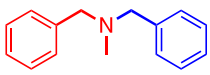


Benzylpiperidine (**7ak**)

The product was prepared using *procedure C* and isolated in 97% yield (0.509 g, 2.91 mmol) as a clear yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc (95:5) as an eluent. The spectral data matched with those reported in the literature[18].

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.40 – 7.23 (m, 5H), 3.51 (s, 2H), 2.42 (t, *J* = 5.3 Hz, 4H), 1.71 – 1.58 (m, 4H), 1.48 (td, *J* = 6.1, 3.2 Hz, 2H).

$^{13}C\{1H\}$ NMR (75 MHz, $CDCl_3$) δ 138.6, 129.2, 128.0, 126.8, 64.0, 54.6, 26.2, 24.6.

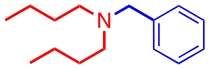


N-benzyl-N-methyl-1-phenylmethanamine (**7al**)

The product was prepared using *procedure C* and isolated in 98% yield (0.620 g, 2.94 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[19].

^1H NMR (300 MHz, $CDCl_3$) δ 7.67 – 7.00 (m, 10H), 3.58 (s, 4H), 2.27 – 2.21 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 139.3, 128.9, 128.2, 126.9, 61.9, 42.4.

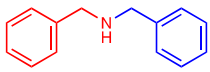


N-benzyl-N-butylbutan-1-amine (**7ar**)

The product was prepared using *procedure C* and isolated in 99% yield (0.620 g, 2.94 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[17].

^1H NMR (300 MHz, $CDCl_3$) δ 7.67 – 7.00 (m, 5H), 3.58 (s, 2H), 2.27 – 2.21 (m, 4H), 1.5 – 1.3 (m, 8H), 0.9 (t, J = 6.5 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 139.3, 128.7, 127.9, 126.5, 58.7, 53.6, 29.4, 20.8, 14.2.

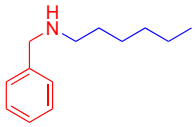


Dibenzyl amine (**7aa**)

The product was prepared using *procedure C* and isolated in 54% yield (0.319 g, 1.54 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (95:5) as an eluent. The spectral data matched with those reported in the literature[7].

^1H NMR (300 MHz, Chloroform-*d*) δ 7.42 – 7.25 (m, 10H), 3.85 (s, 4H), 2.09 (s, 1H).

$^{13}C\{1H\}$ NMR (75 MHz, $CDCl_3$) δ 140.0, 128.4, 128.2, 127.0, 53.1.

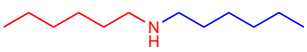


N-benzylhexan-1-amine (**7ca**)

The product was prepared using *procedure C* and isolated in 70% yield (0.401 g, 2.1 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (95:5) as an eluent. The spectral data matched with those reported in the literature[20].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 – 7.17 (m, 5H), 3.8 (s, 2H), 2.63 (t, *J* = 7.2 Hz, 2H), 1.62 – 1.39 (m, 2H), 1.36 – 1.28 (m, 6H), 0.92 – 0.83 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 140.5, 128.3, 128.0, 126.8, 54.2, 49.6, 31.9, 30.2, 27.2, 22.8, 14.2.

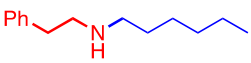


N-benzylhexan-1-amine (**7ch**)

The product was prepared using *procedure C* and isolated in 77% yield (0.427 g, 2.31 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (95:5) as an eluent. The spectral data matched with those reported in the literature[21].

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.55 (t, *J* = 6.9 Hz, 4H), 1.53 – 1.36 (m, 4H), 1.28 (ddt, *J* = 10.0, 3.9, 2.0 Hz, 12H), 0.95 – 0.78 (m, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 50.3, 31.9, 30.3, 27.2, 22.7, 14.1.

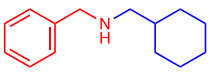


N-phenethylhexan-1-amine (**7cg**)

The product was prepared using *procedure C* and isolated in 72% yield (0.442 g, 2.16 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane/EtOAc (95:5) as an eluent. The spectral data matched with those reported in the literature[17].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 – 6.98 (m, 5H), 2.97 – 2.73 (m, 4H), 2.69 – 2.55 (m, 2H), 1.37 – 1.18 (m, 8H), 0.95 – 0.82 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 140.1, 128.6, 128.4, 126.0, 50.0, 36.5, 31.9, 30.2, 27.2, 22.7, 14.2, 1.2.

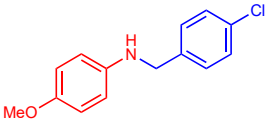


N-benzyl-1-cyclohexylmethanamine (**7ba**)

The product was prepared using *procedure C* and isolated in 84% yield (0.511 g, 2.5 mmol) as a pale yellow liquid after purification via column chromatography on silica gel using hexane as an eluent. The spectral data matched with those reported in the literature[22].

^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.31 (dd, *J* = 23.7, 4.5 Hz, 5H), 3.80 (s, 2H), 2.50 (d, *J* = 6.7 Hz, 2H), 1.89 – 1.63 (m, 4H), 1.62 – 1.46 (m, 1H), 1.42 – 1.09 (m, 4H), 0.97 (td, *J* = 11.7, 2.9 Hz, 2H).

^{13}C{1H} NMR (75 MHz, $CDCl_3$) δ 140.6, 128.3, 128.1, 128.0, 126.8, 56.3, 54.3, 38.1, 31.9, 31.6, 26.9, 26.3.

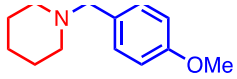


N-(4-chlorobenzyl)-4-methoxyaniline (7dq)

The product was prepared using *procedure C* and isolated in 96% yield (0.714 g, 2.88 mmol) as a yellow liquid after purification via column chromatography on silica gel using hexane/EtOAc (90:10) as an eluent. The spectral data matched with those reported in the literature[17].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (s, 4H), 6.79 (d, *J* = 8.9 Hz, 2H), 6.59 (d, *J* = 8.9 Hz, 2H), 4.27 (s, 2H), 3.75 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 152.2, 142.0, 138.2, 132.7, 128.7, 128.6, 114.9, 114.1, 55.9, 48.6.



1-(4-methoxybenzyl)piperidine (7ek)

The product was prepared using *procedure C* and isolated in 94% yield (0.578 g, 2.82 mmol) as a colourless liquid after purification via column chromatography on silica gel using hexane the eluent. The spectral data matched with those reported in the literature[14].

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.17 (m, 2H), 3.79 (d, *J* = 0.5 Hz, 3H), 3.42 (s, 2H), 2.36 (t, *J* = 5.3 Hz, 4H), 1.57 (p, *J* = 5.5 Hz, 4H), 1.42 (hept, *J* = 4.3 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 158.4, 130.3, 113.4, 63.2, 55.3, 54.4, 26.0, 24.5.

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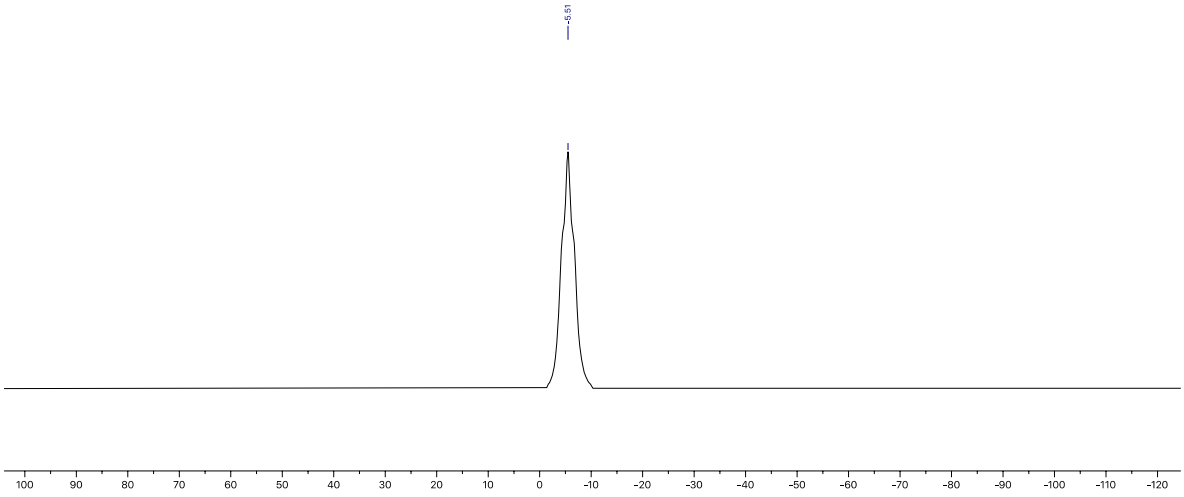
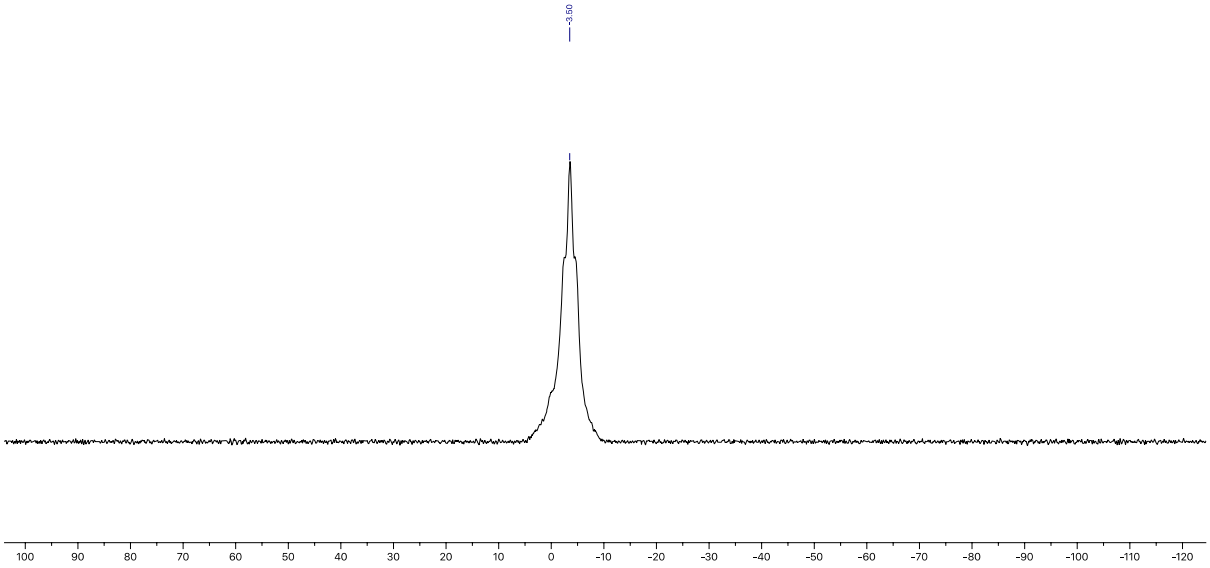
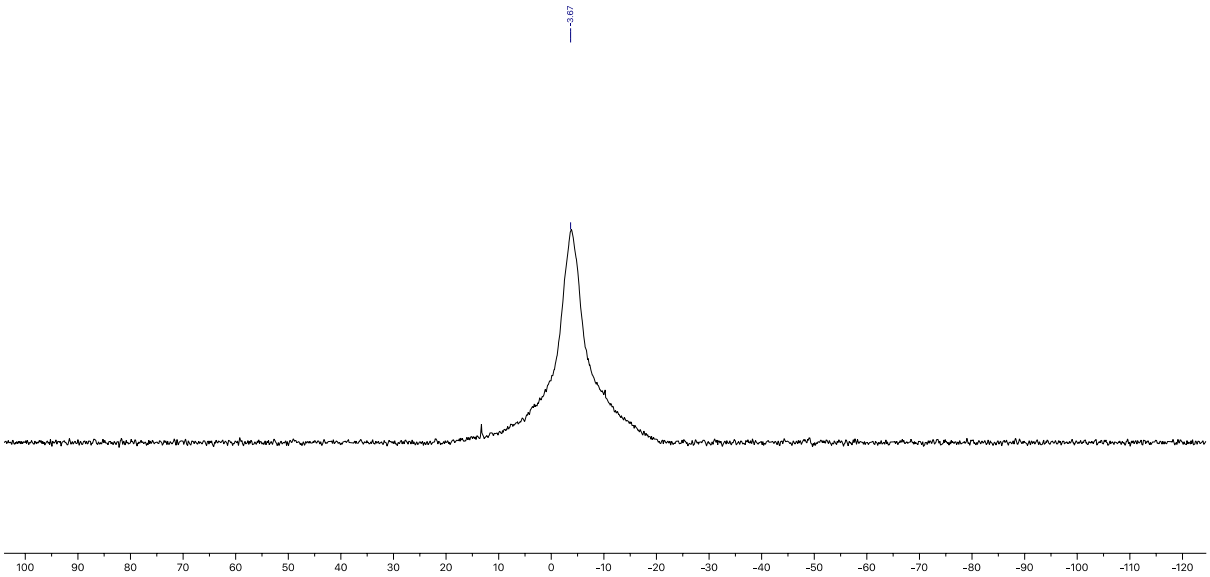


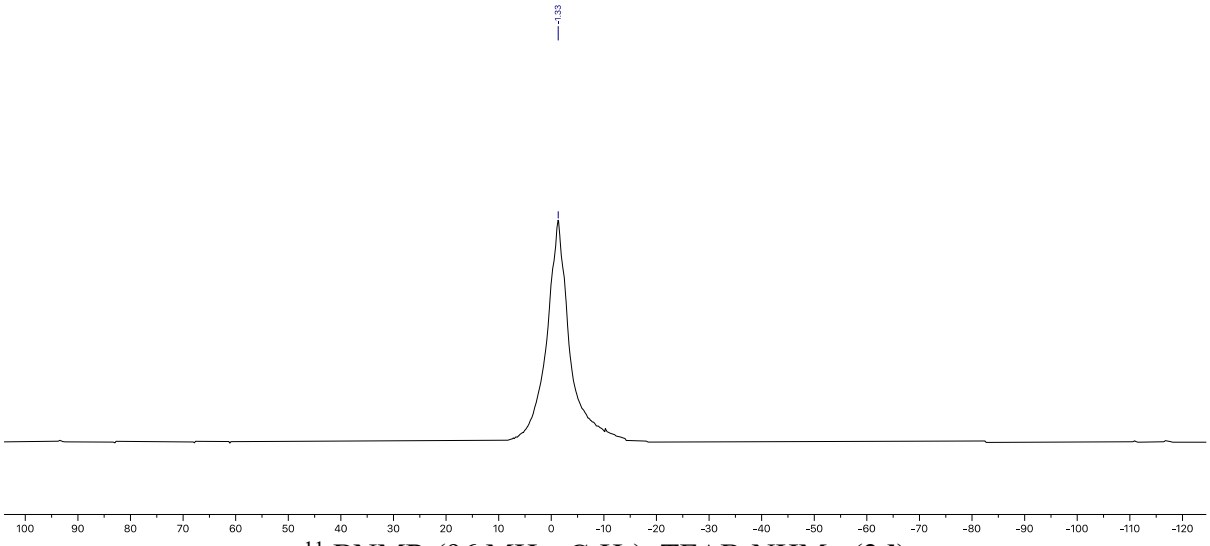
^{11} BNMR (96 MHz, THF) TFAB-NH_3(**2a**)



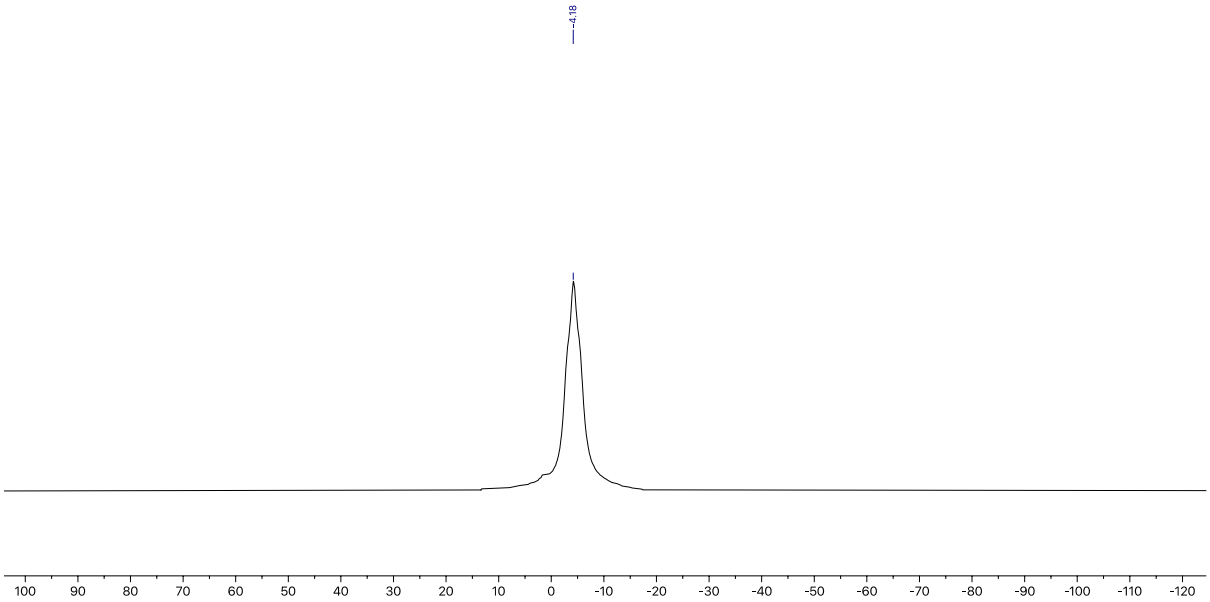
^{11} BNMR (96 MHz, C_7H_8) TFAB-NH_2Me (**2b**)



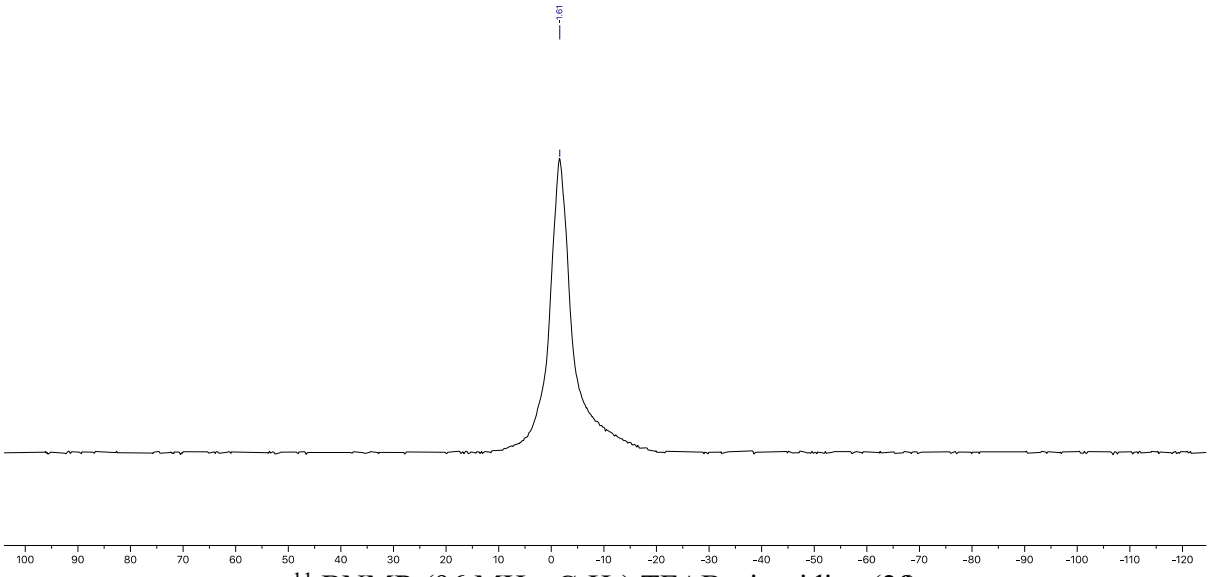
^{11} BNMR (96 MHz, C_7H_8) TFAB-NH_2Bn (**2c**)



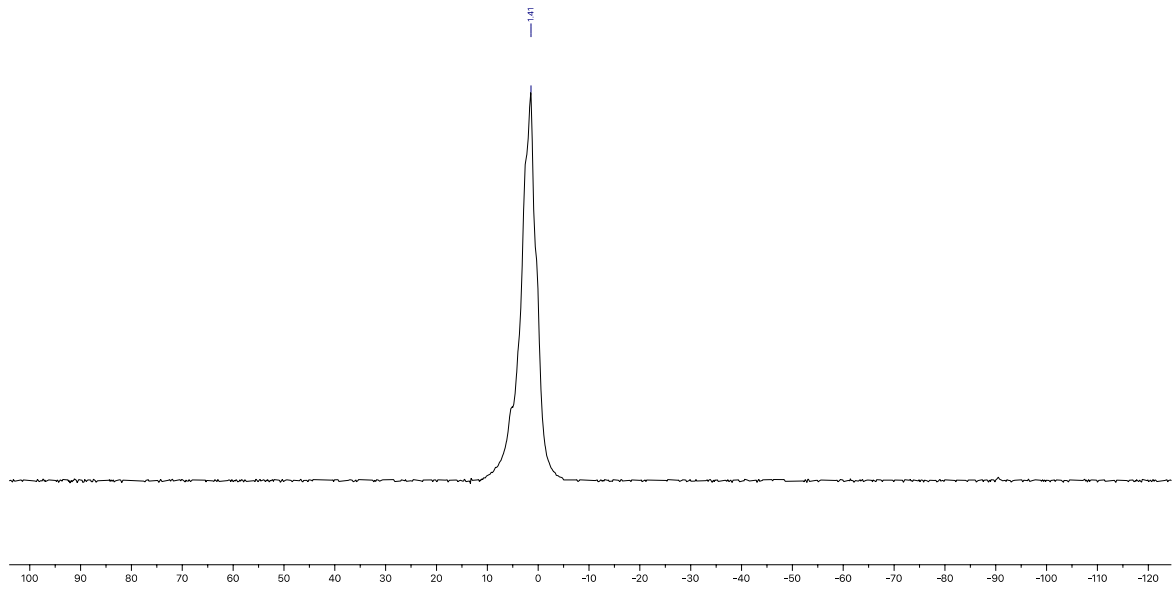
^{11} BNMR (96 MHz, C_7H_8) TFAB-$NHMe_2$(**2d**)



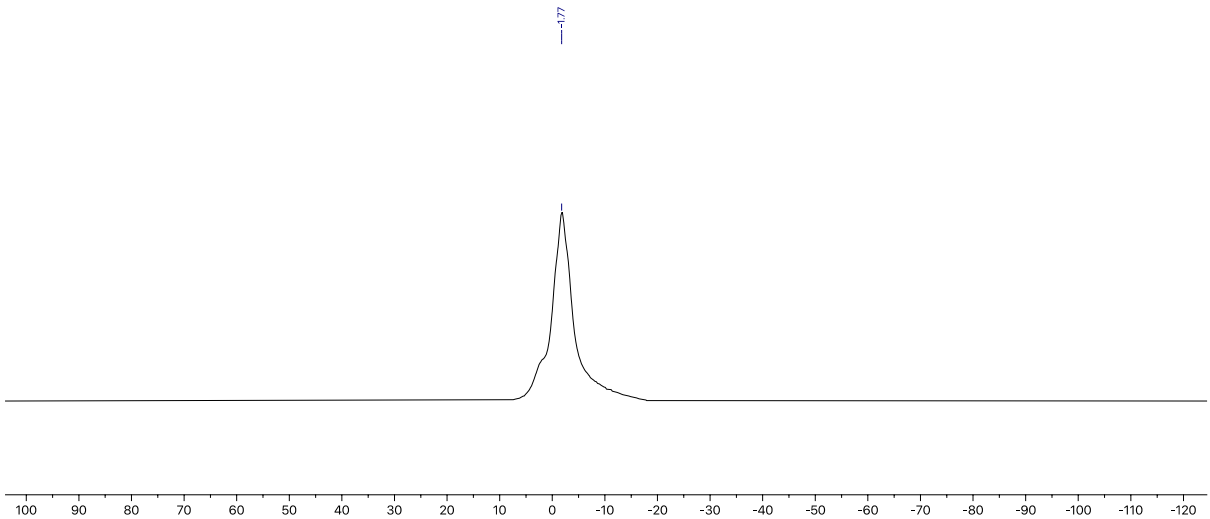
^{11} BNMR (96 MHz, C_7H_8) TFAB-NH(i-Pr)$_2$ (**2e**)



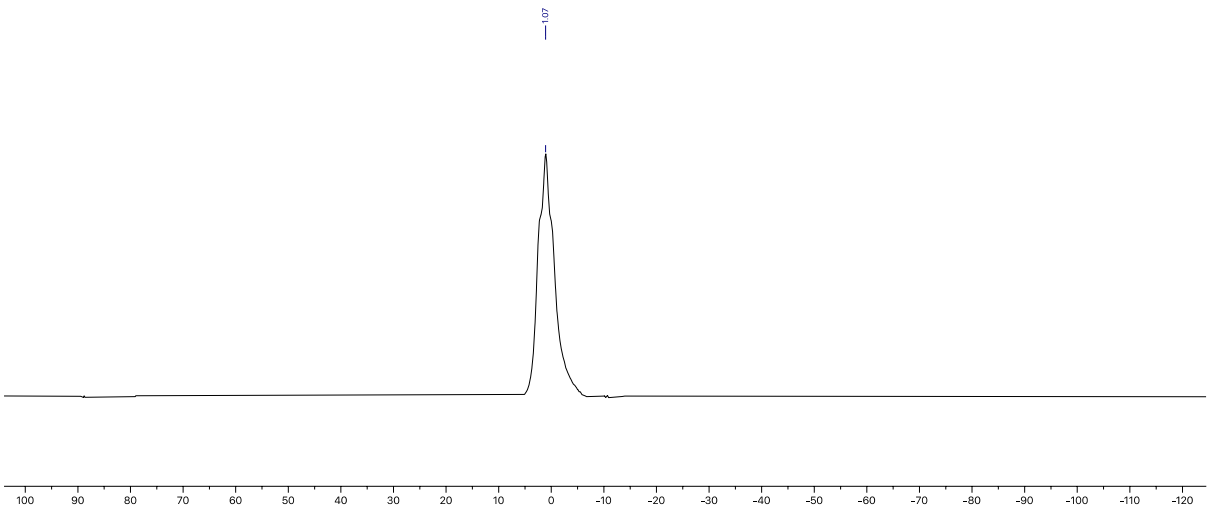
^{11} BNMR (96 MHz, C_7H_8) TFAB-piperidine (**2f**)



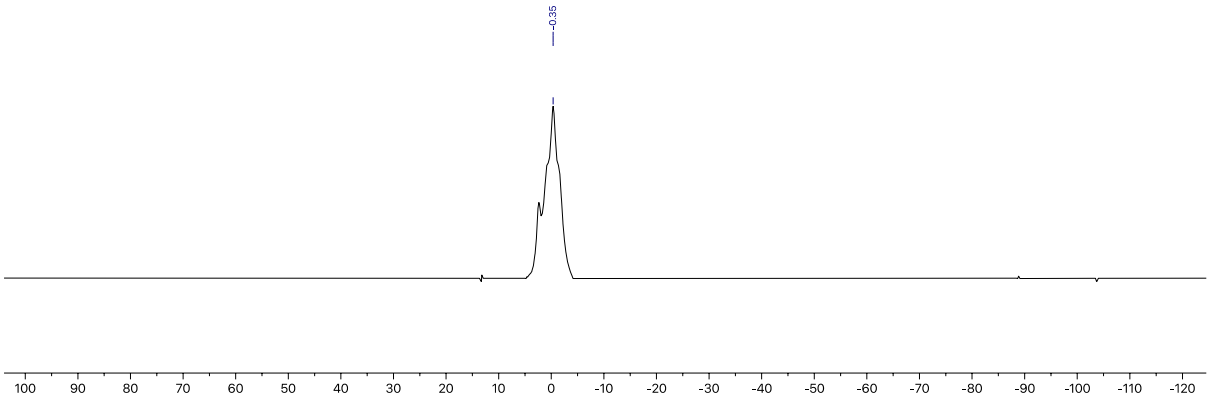
^{11} BNMR (96 MHz, C_7H_8) TFAB-NMe_3 (**2g**)



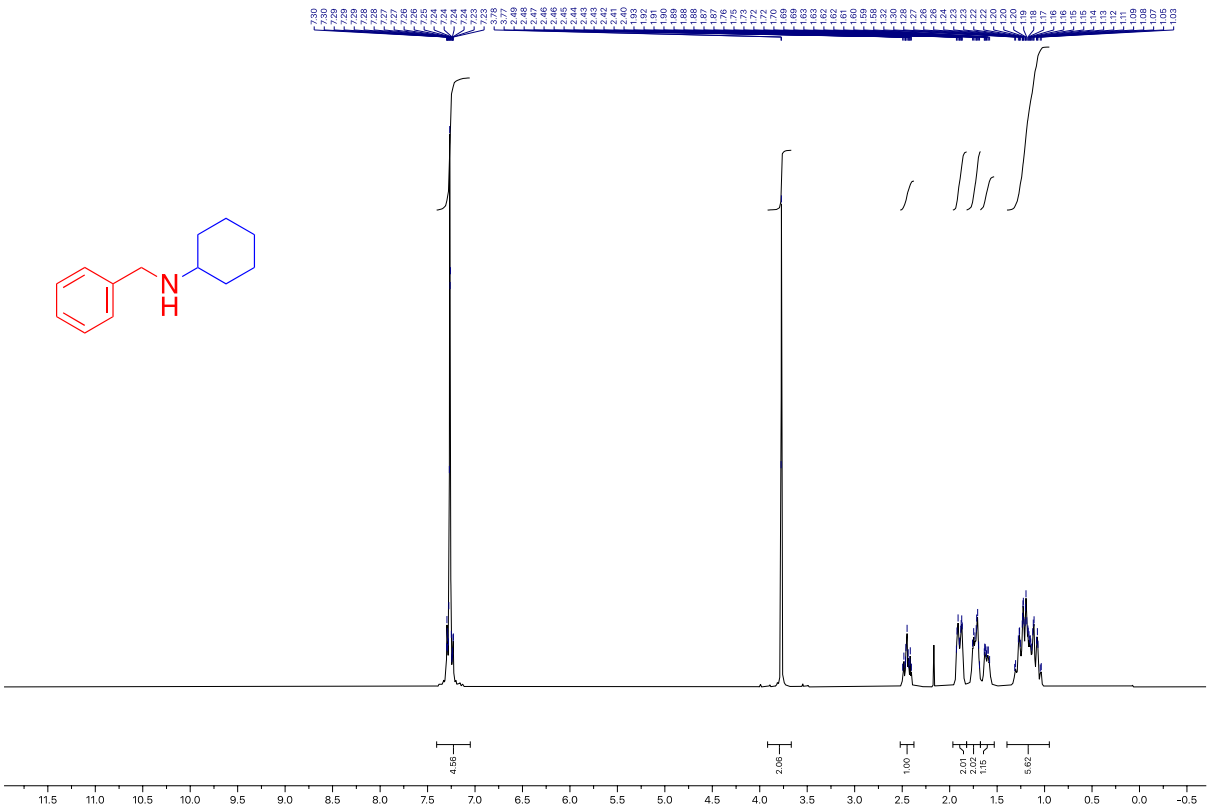
^{11}B NMR (96 MHz, C_7H_8) TFAB-NEt_3 (**2h**)



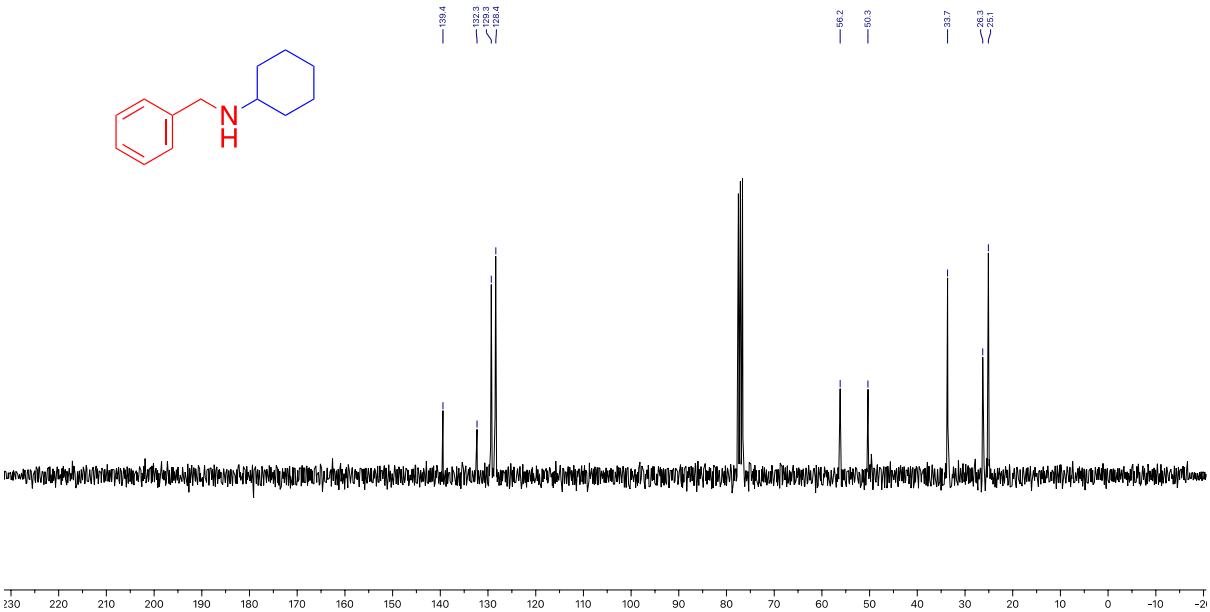
^{11} BNMR (96 MHz, C_7H_8) TFAB- Pyridine (**2i**)



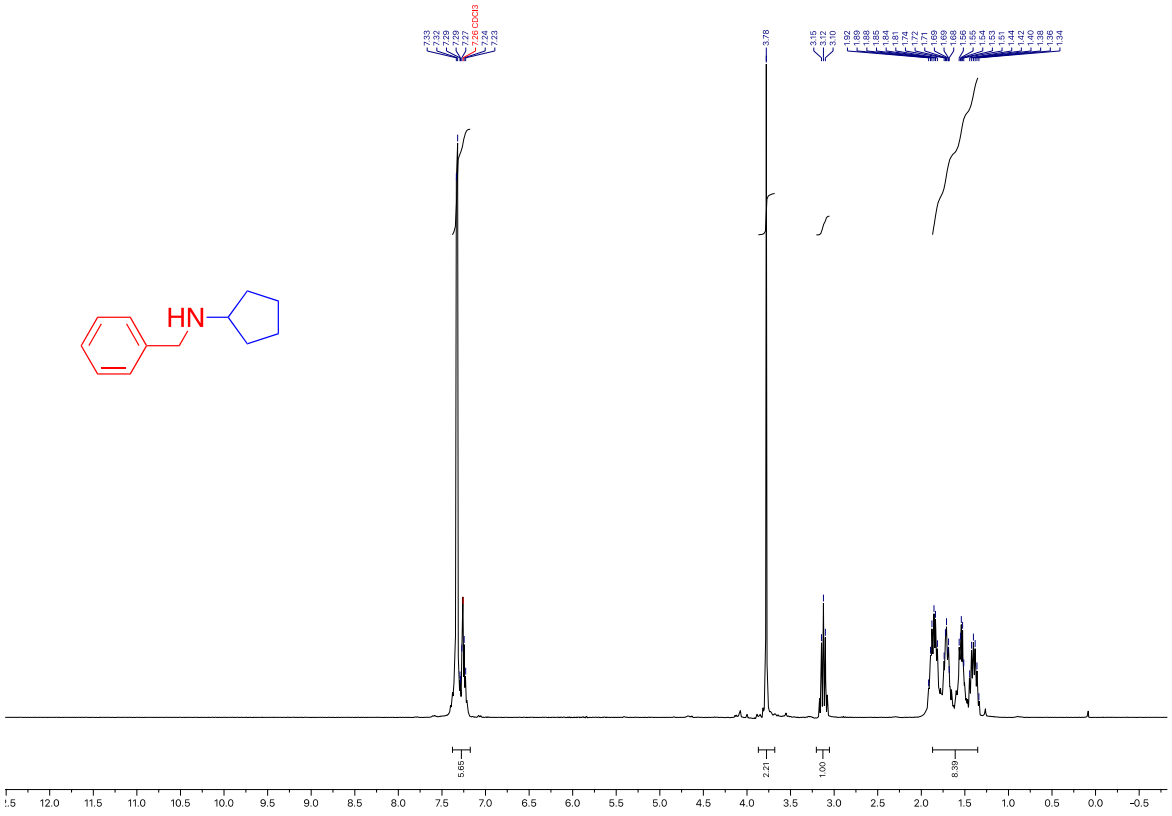
^{11} BNMR (96 MHz, C_7H_8) TFAB- Picoline (**2j**)



^{1}H NMR (300 MHz, Chloroform-*d)* of **6aa**



^{13}C NMR (75 MHz, Chloroform-*d)* of **6aa**



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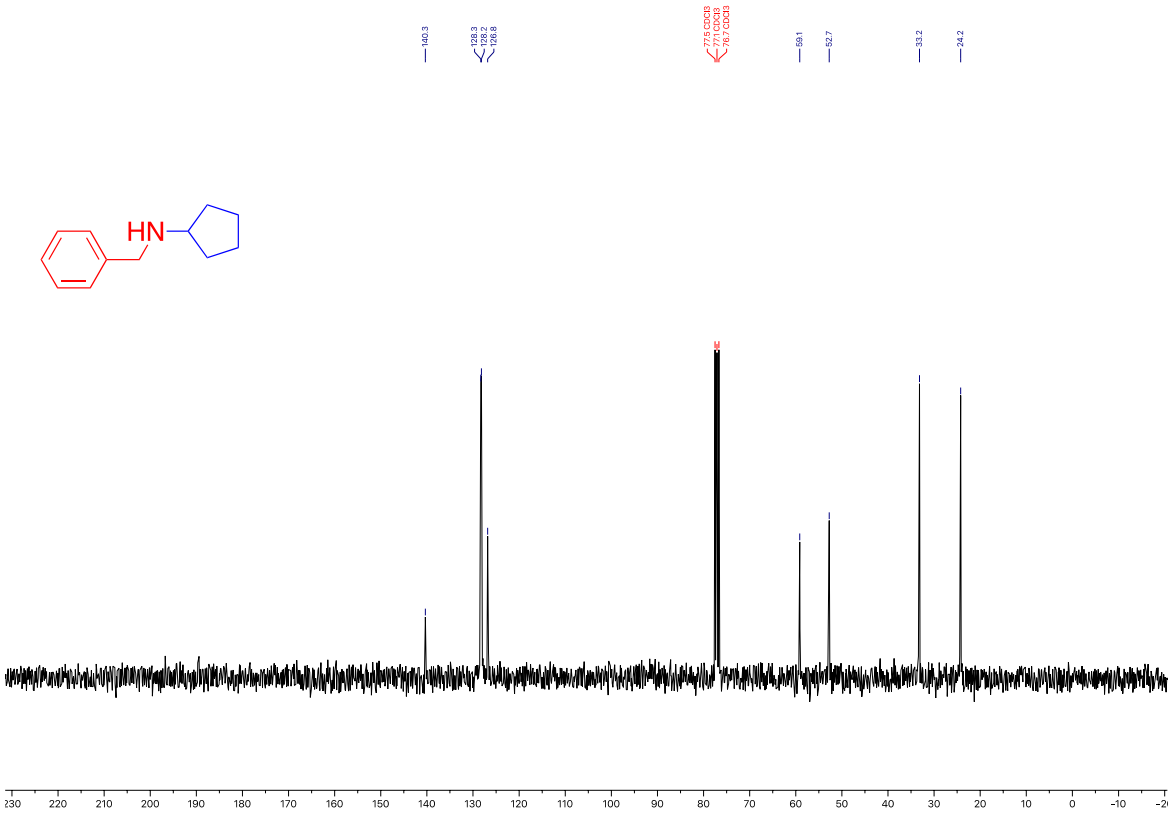
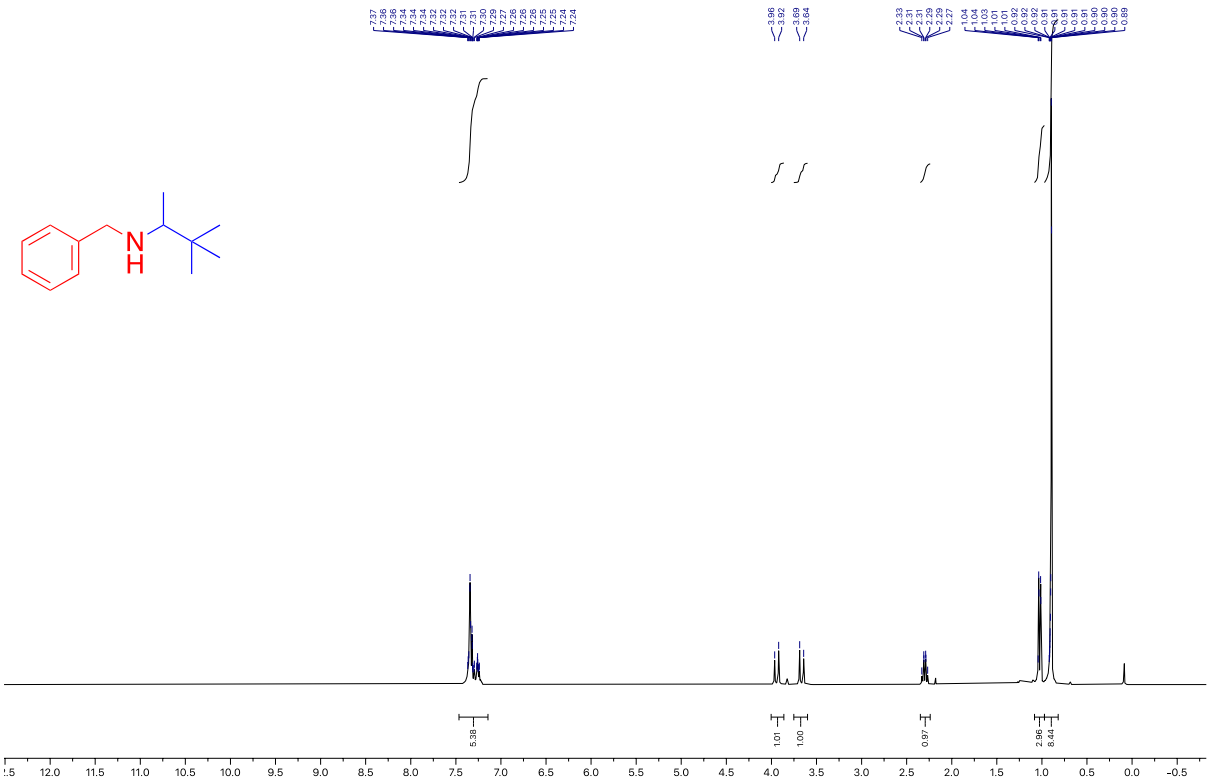
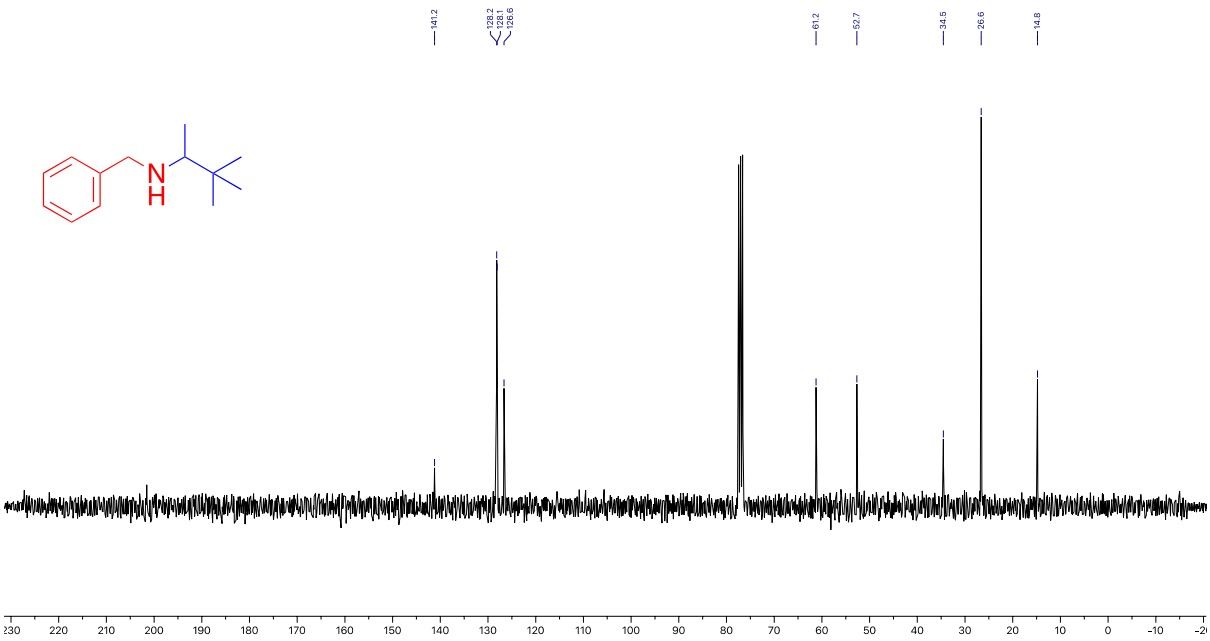


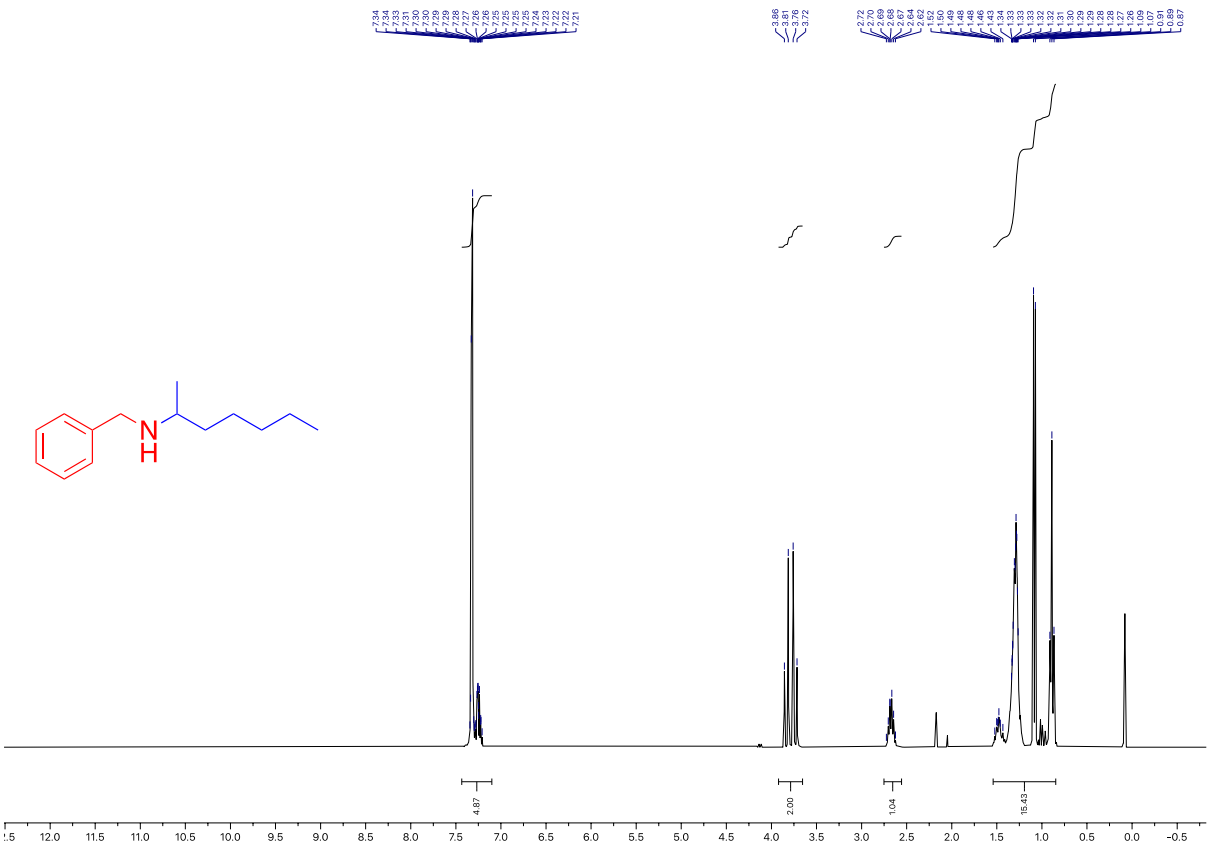
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ba**



^{1}H NMR (300 MHz, Chloroform-*d)* of **6fa**



^{13}C NMR (75 MHz, Chloroform-*d)* of **6fa**



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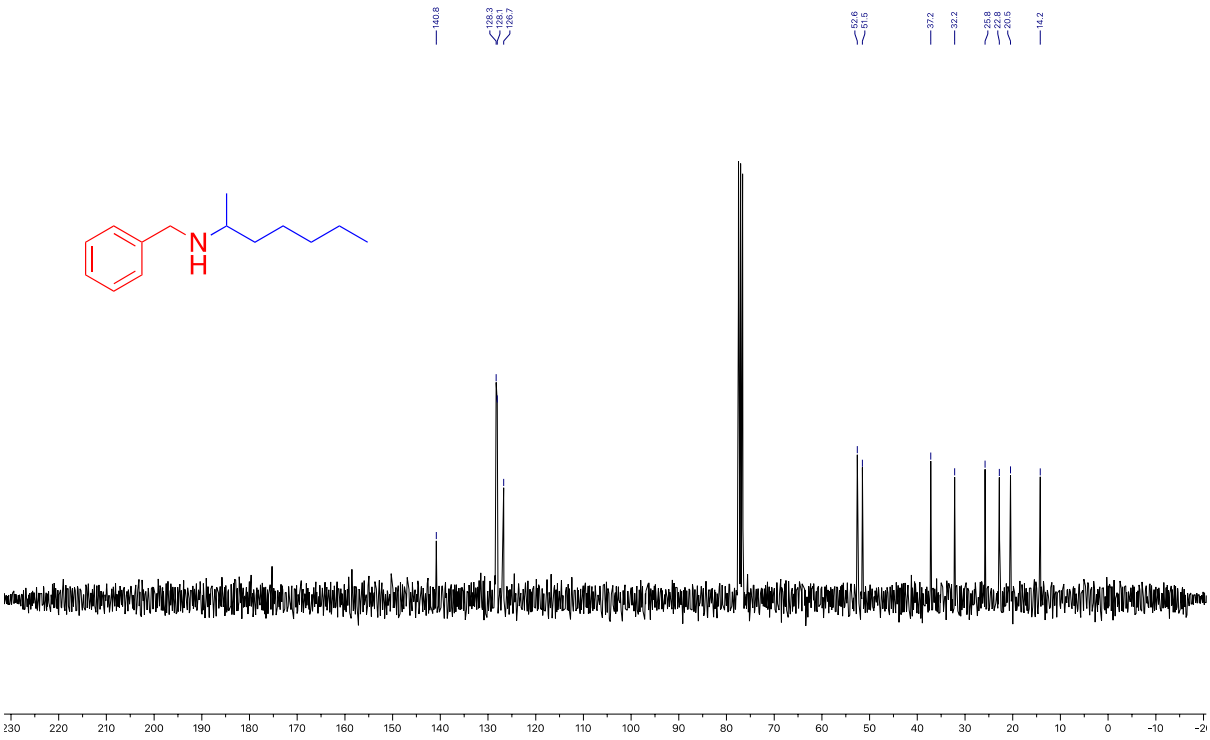

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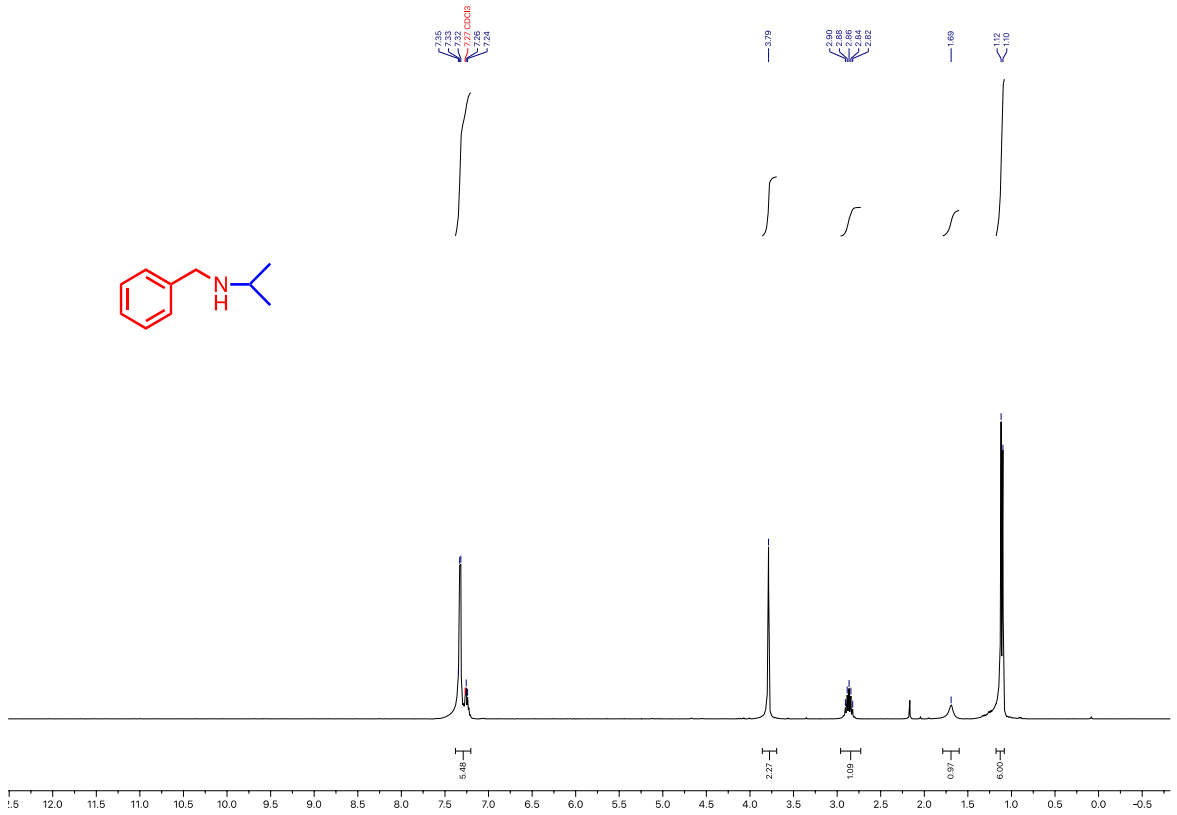

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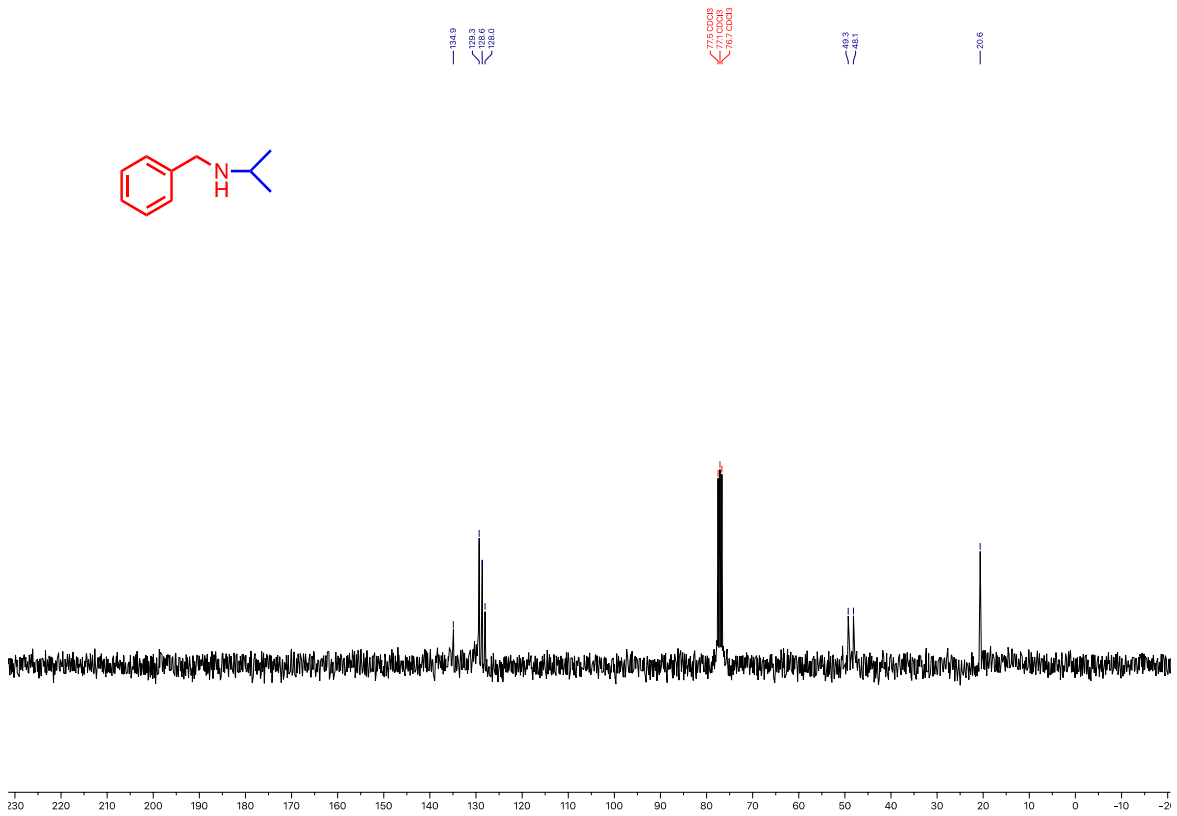
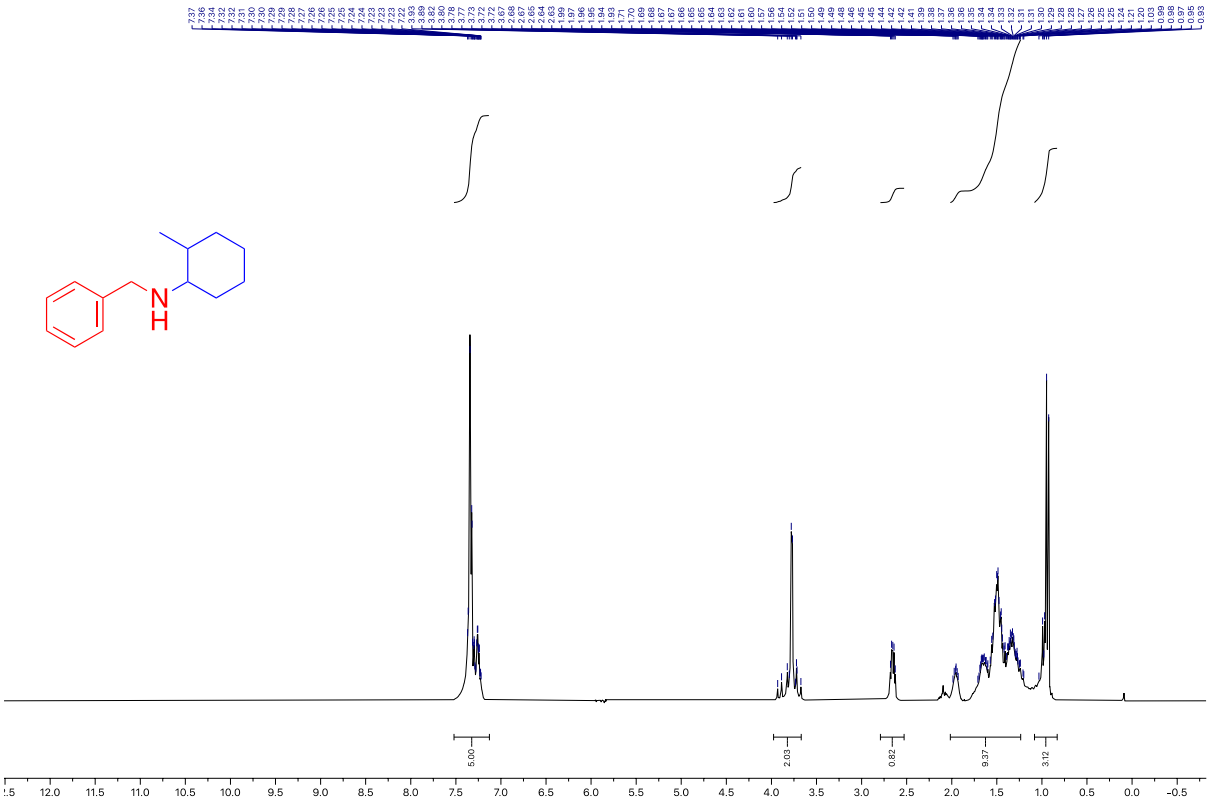


^{13}C NMR (75 MHz, Chloroform-*d)* of **6da**



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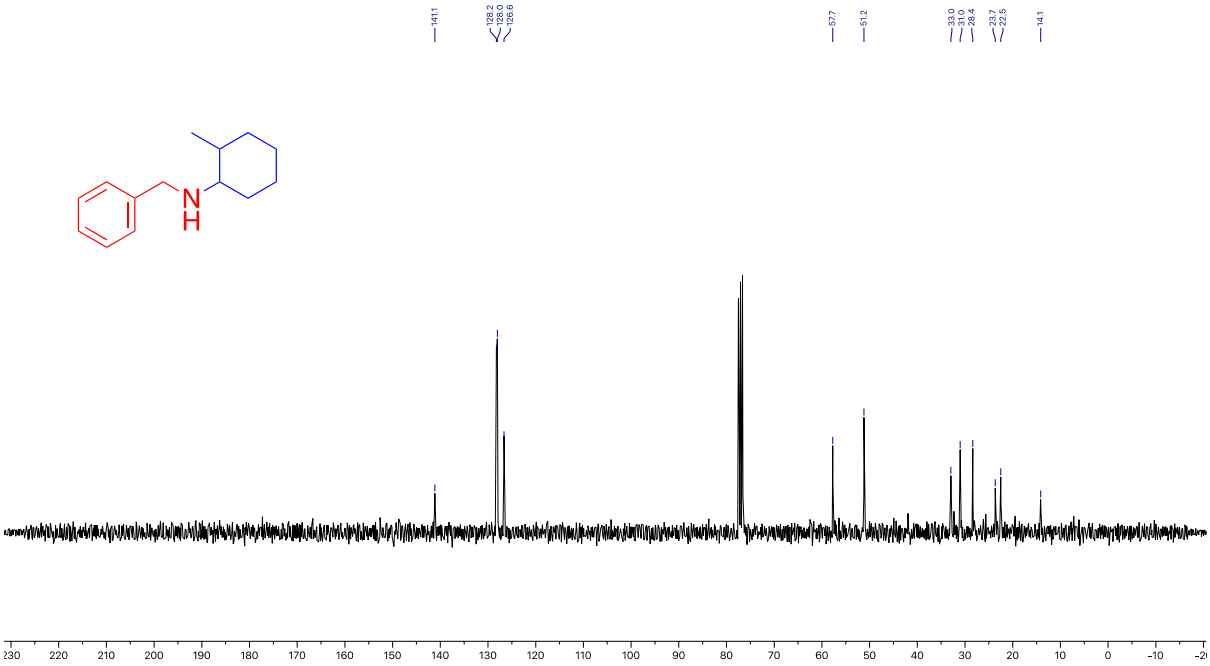
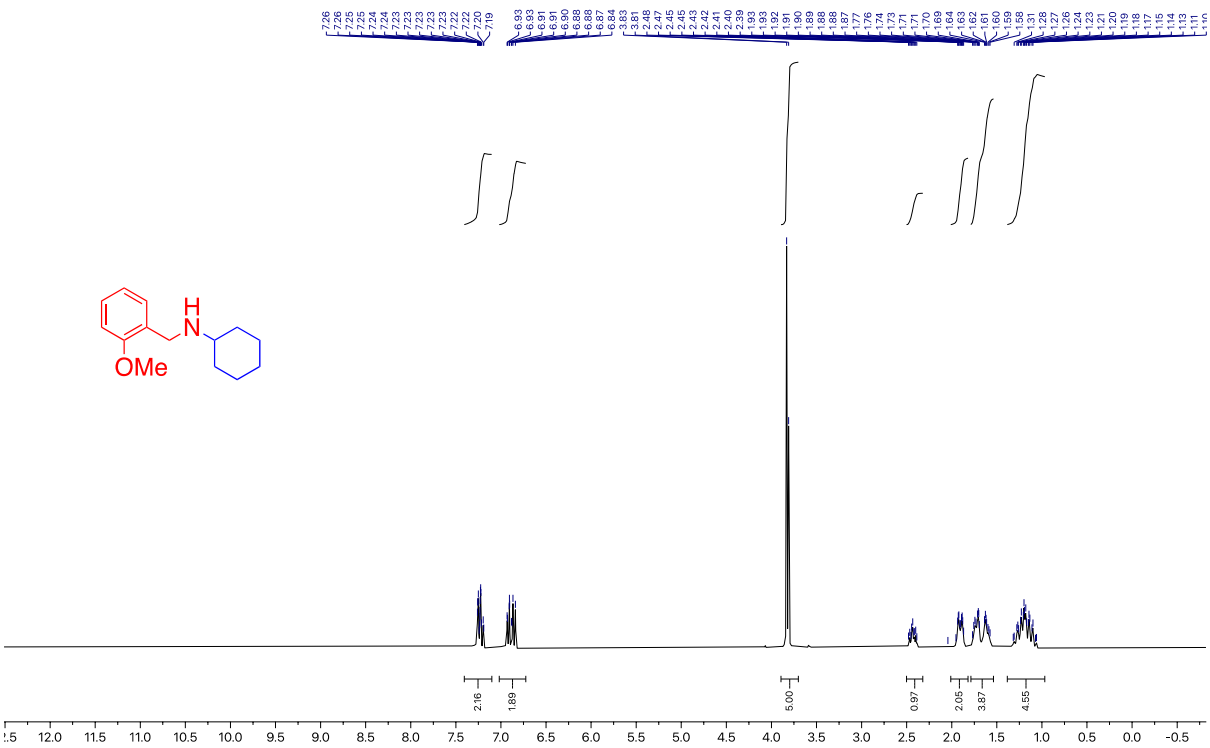
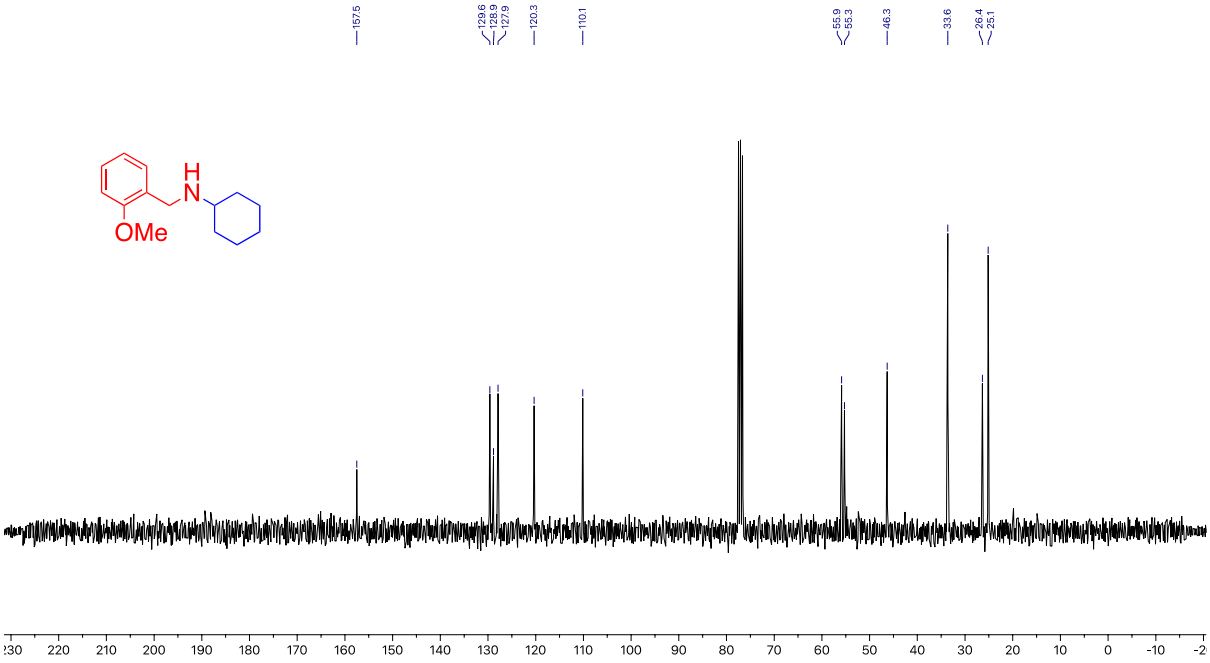


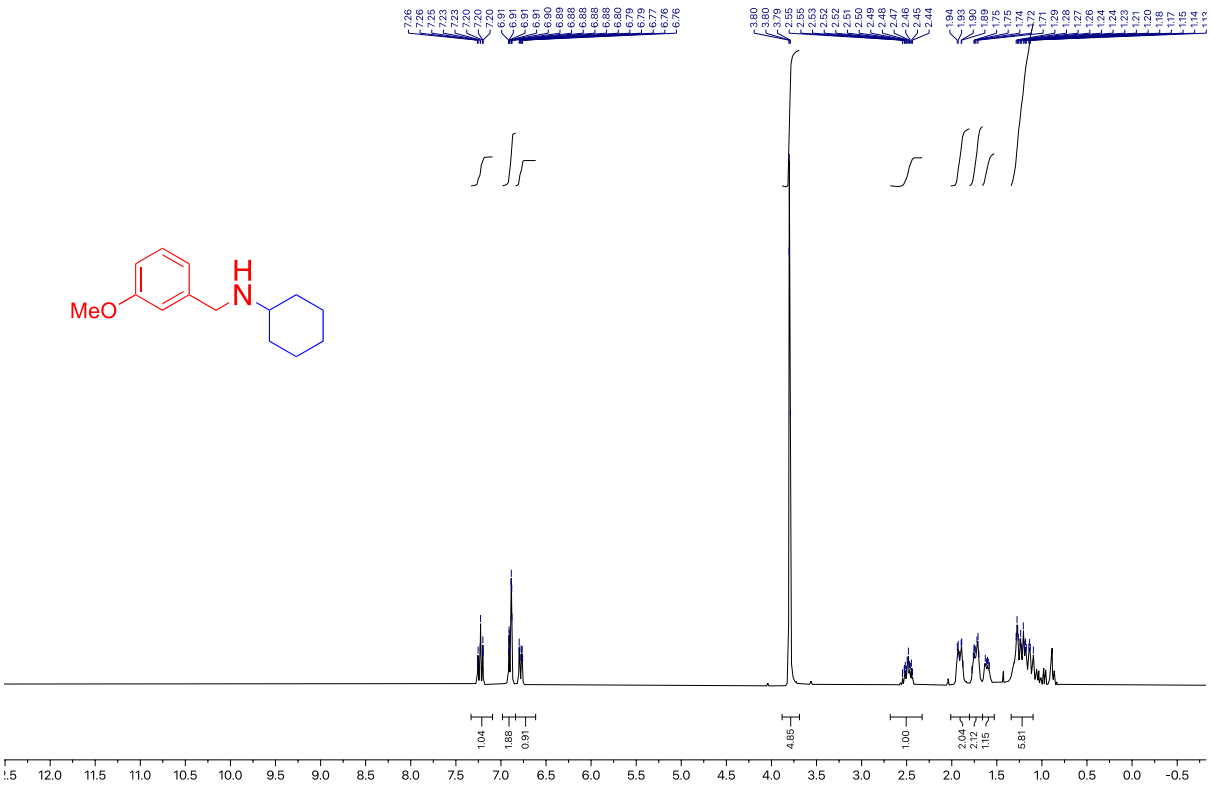
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ea**



^{1}H NMR (300 MHz, Chloroform-*d)* of **6ab**



^{13}C NMR (75 MHz, Chloroform-*d)* of **6ab**



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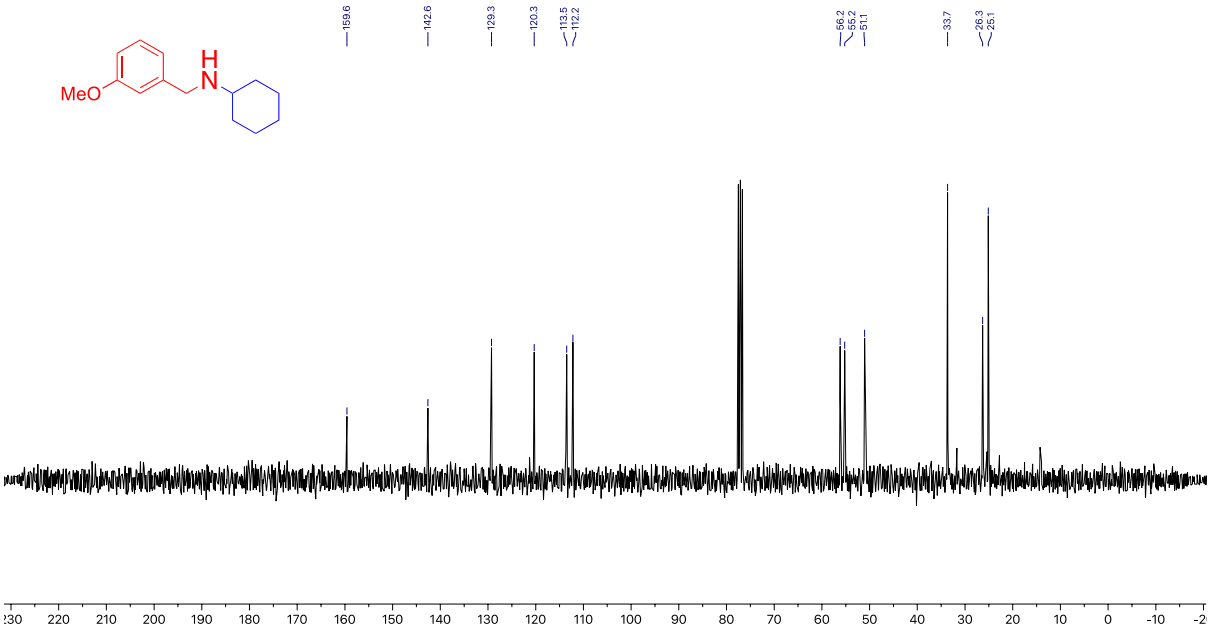

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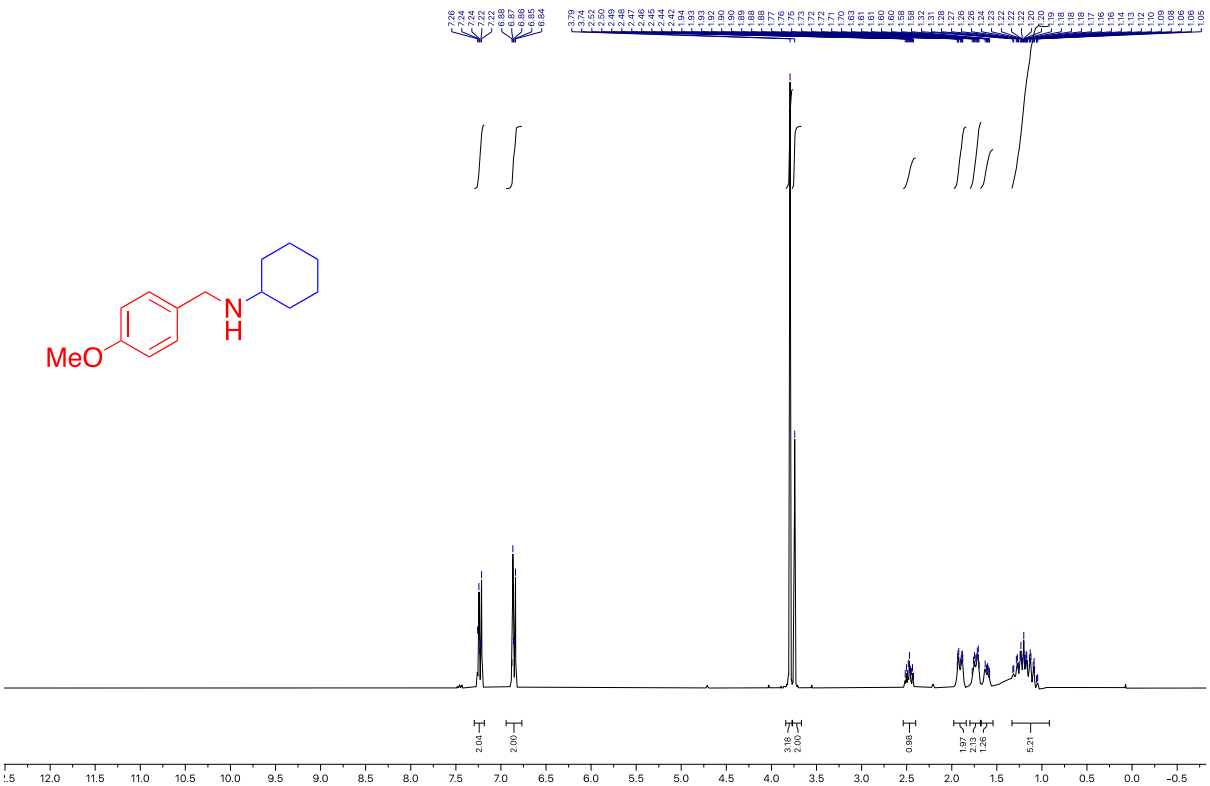

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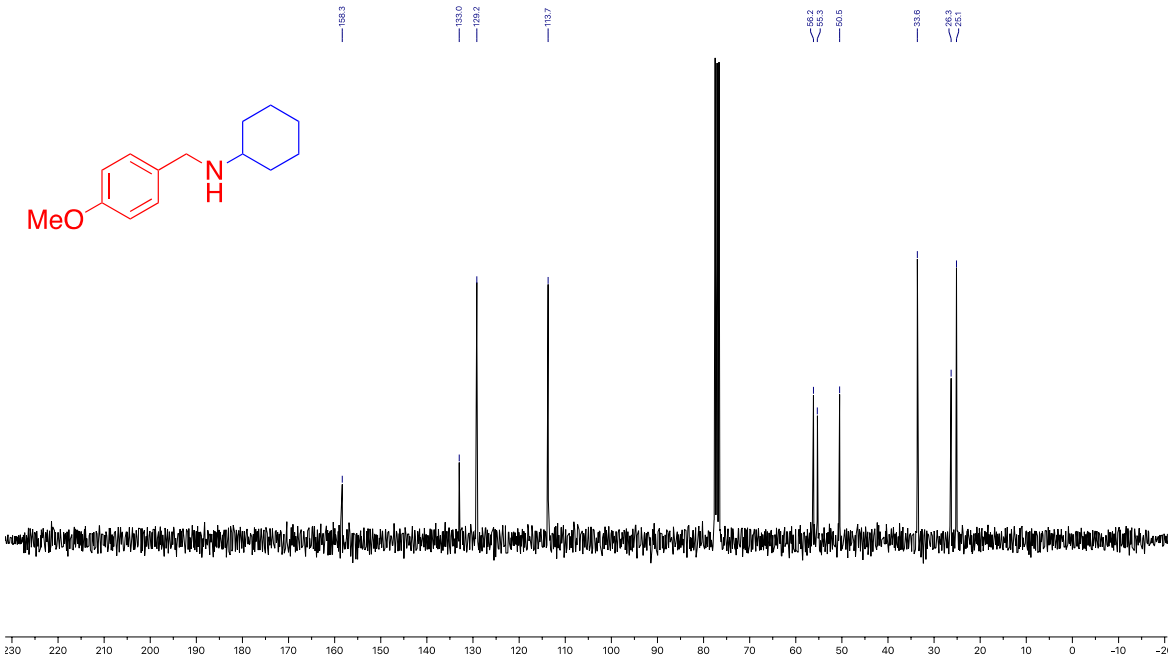

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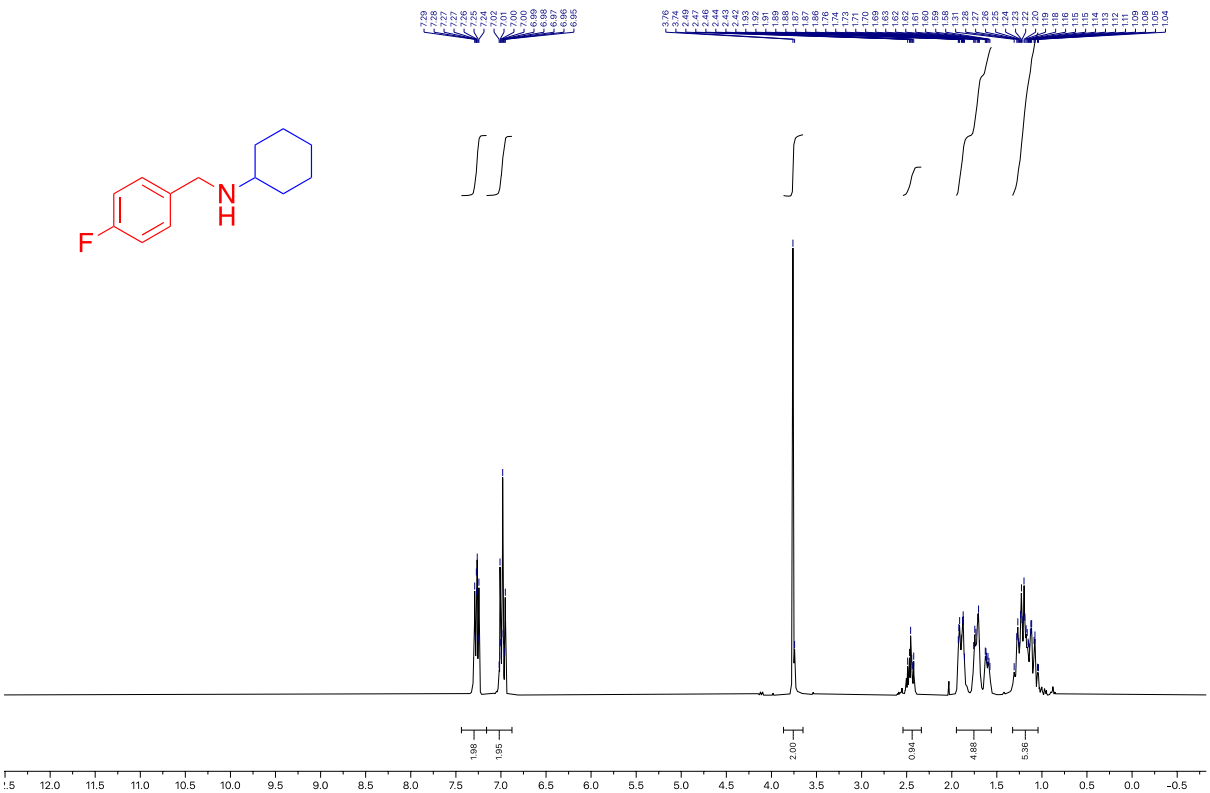

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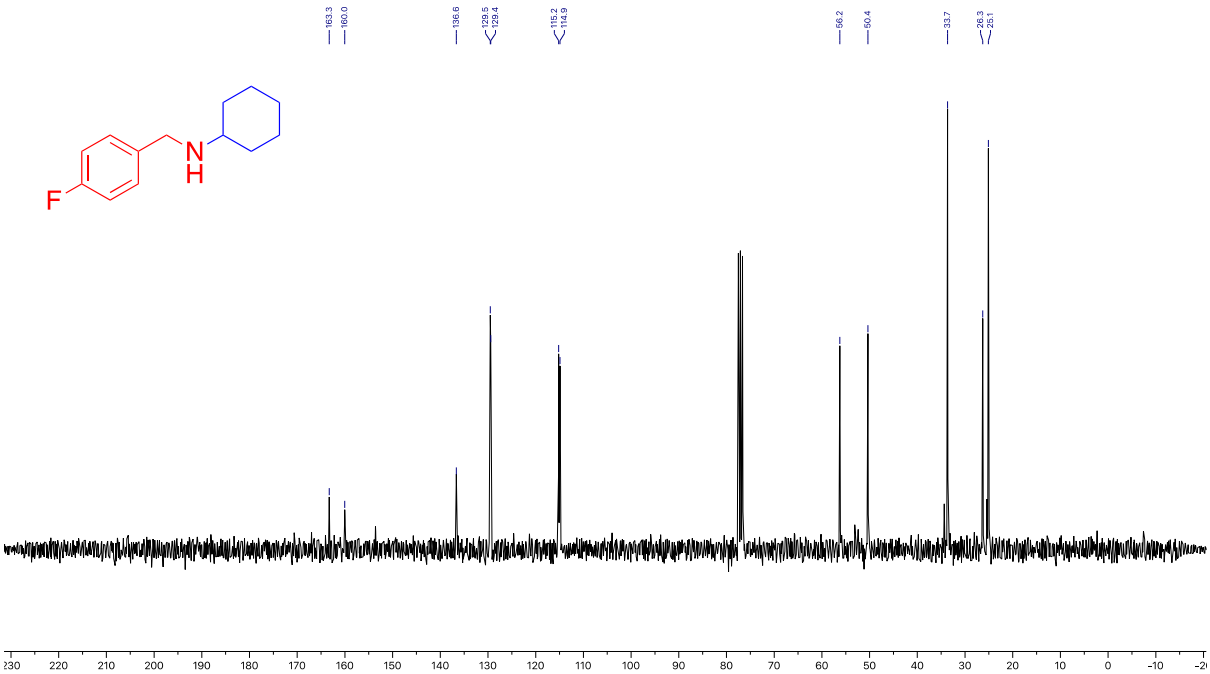

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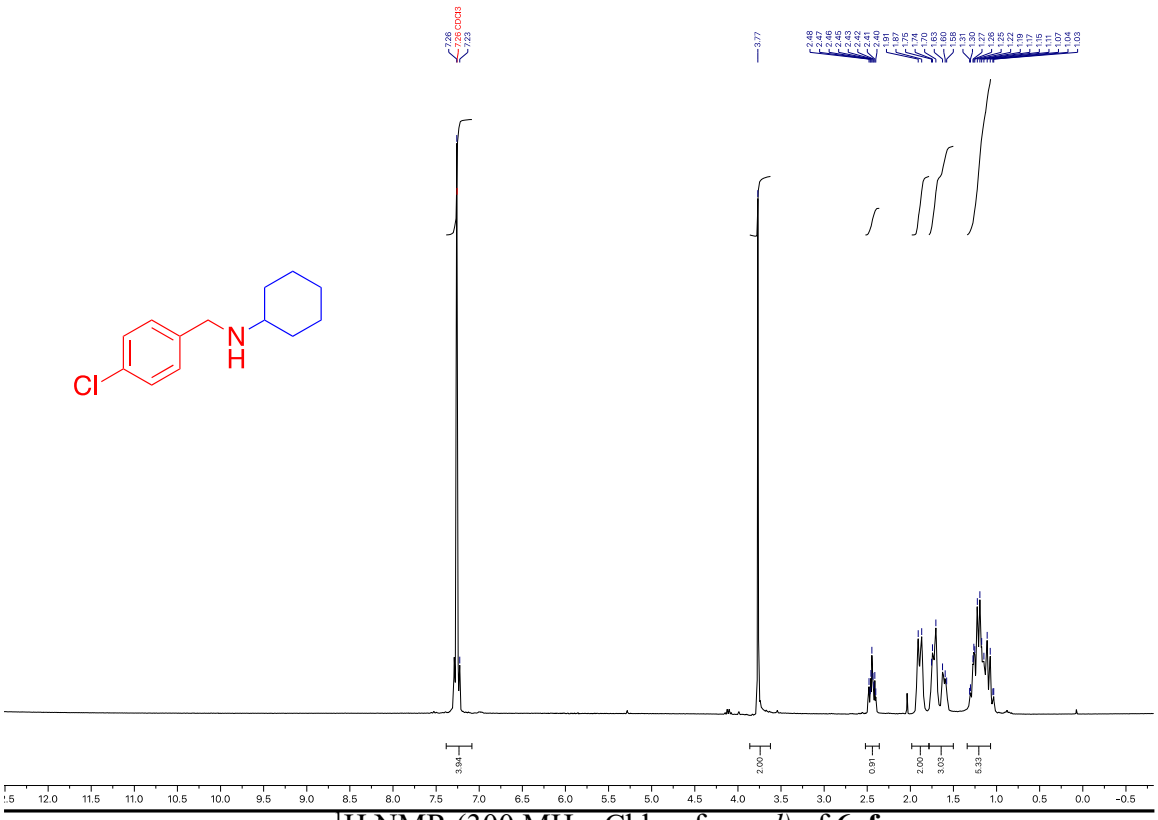

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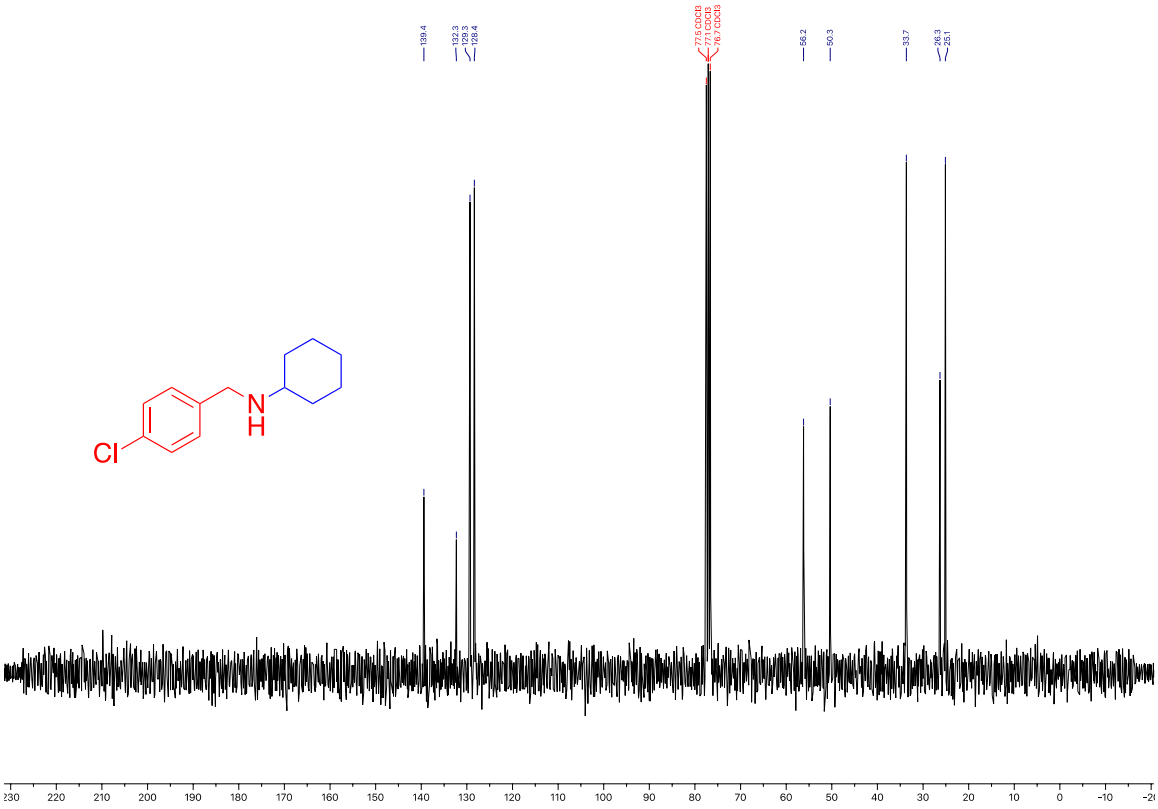
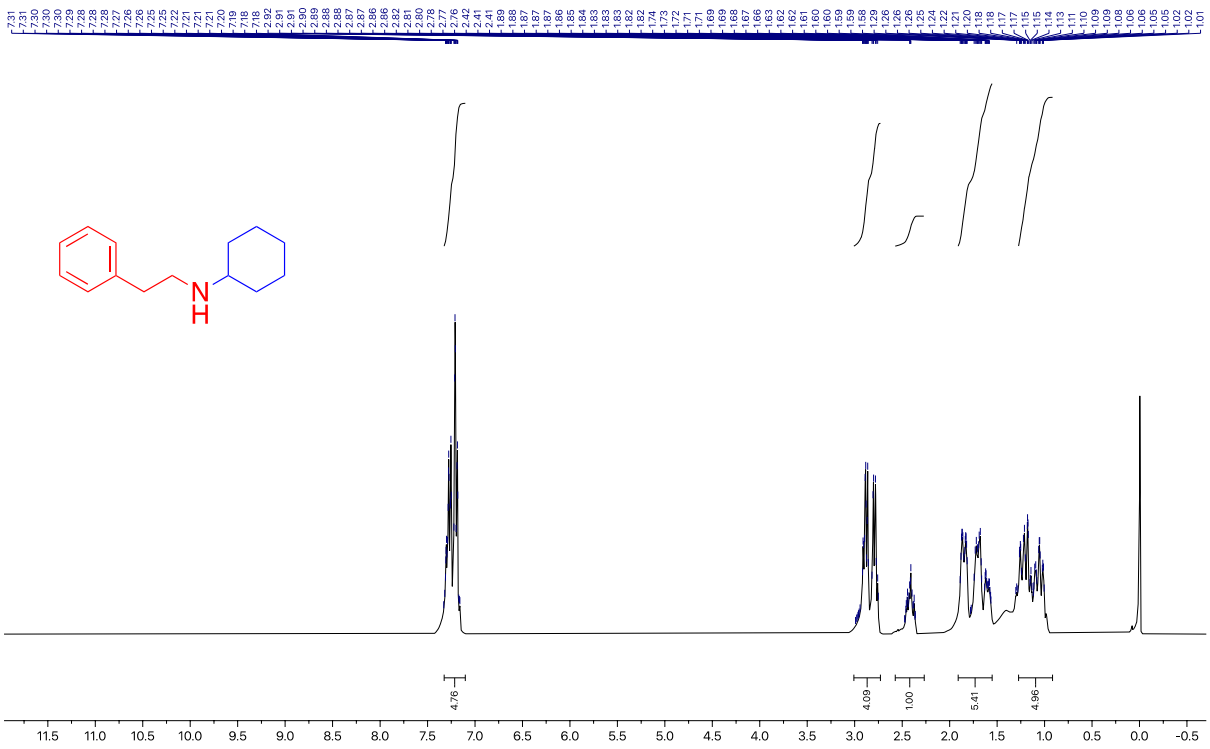
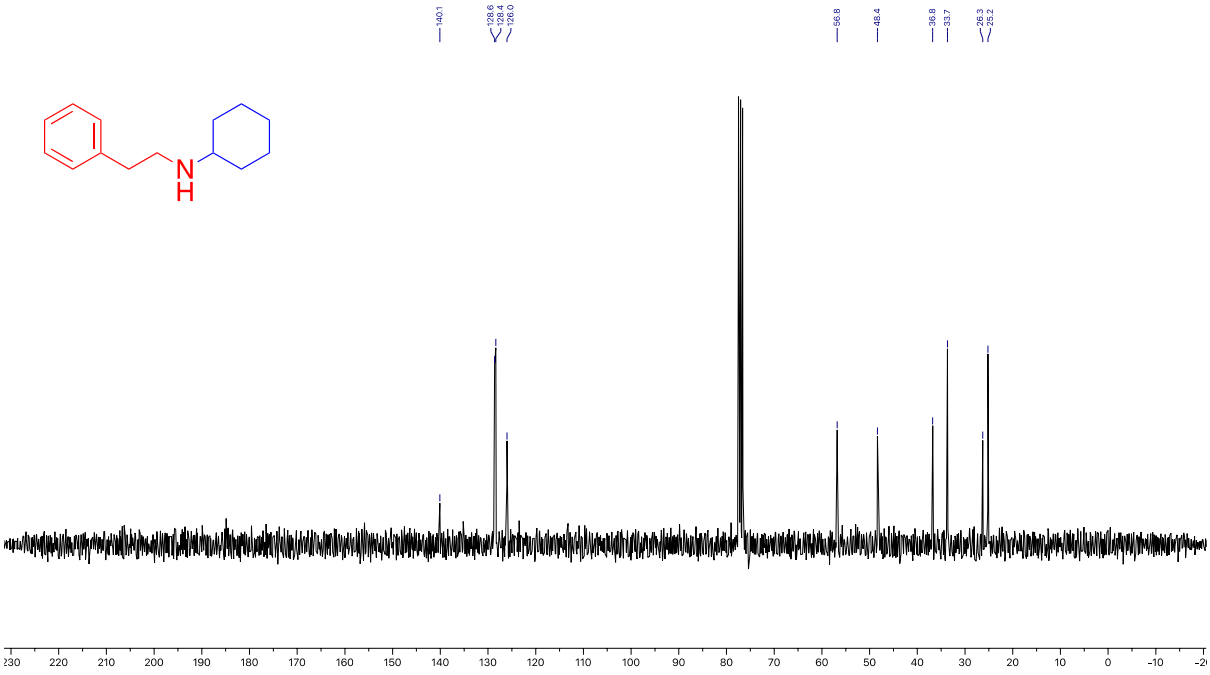


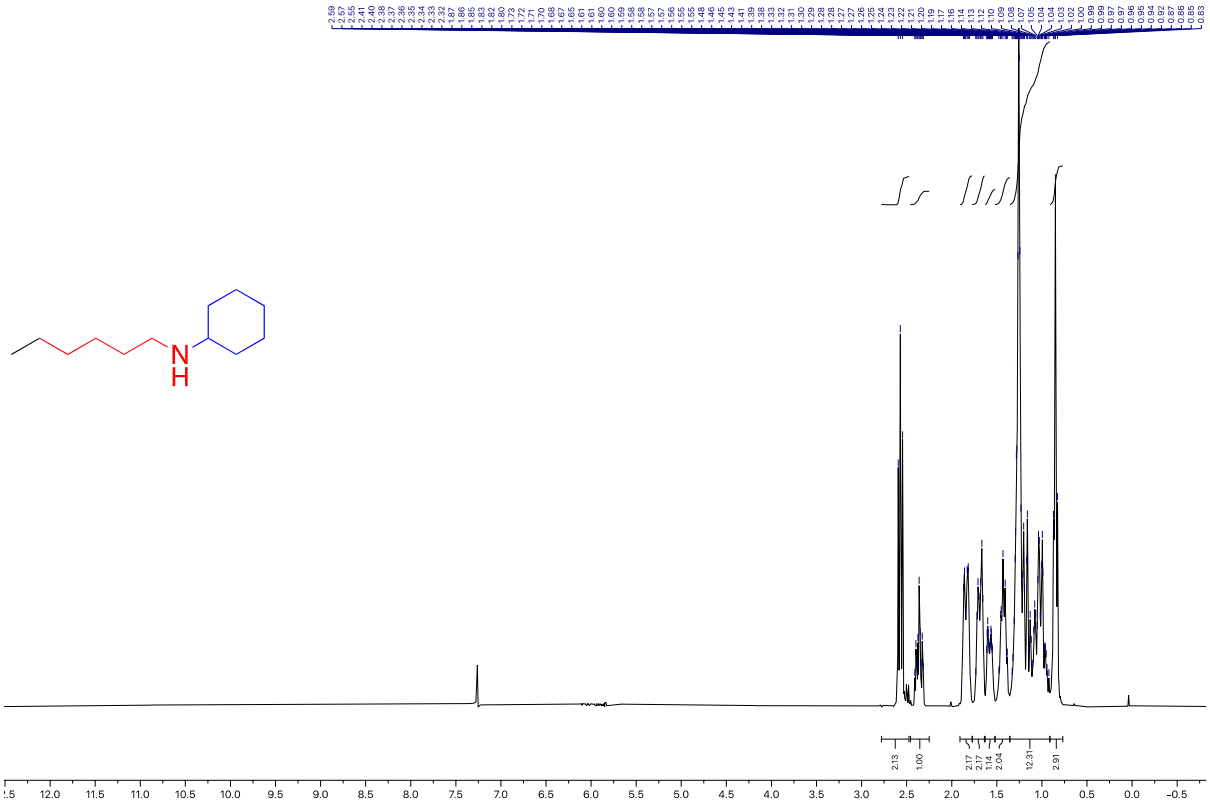
^{13}C NMR (75 MHz, Chloroform-*d)* of **6af**



^{1}H NMR (300 MHz, Chloroform-*d)* of **6ag**



^{13}C NMR (75 MHz, Chloroform-*d)* of **6ag**



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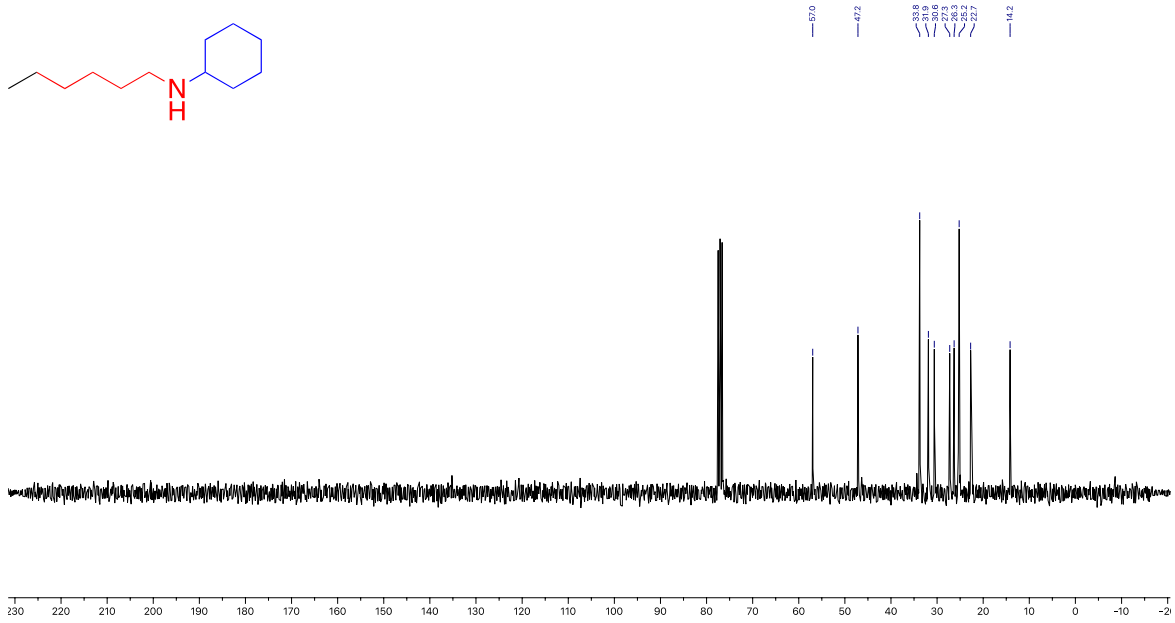
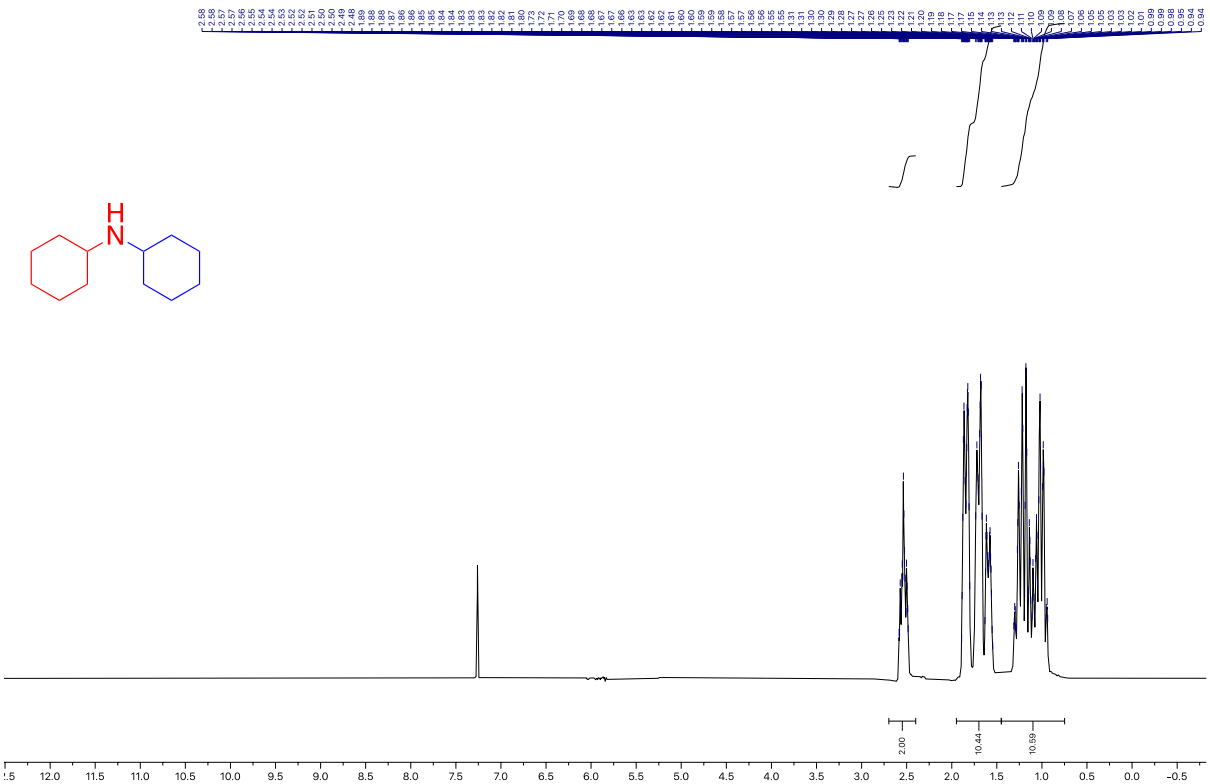
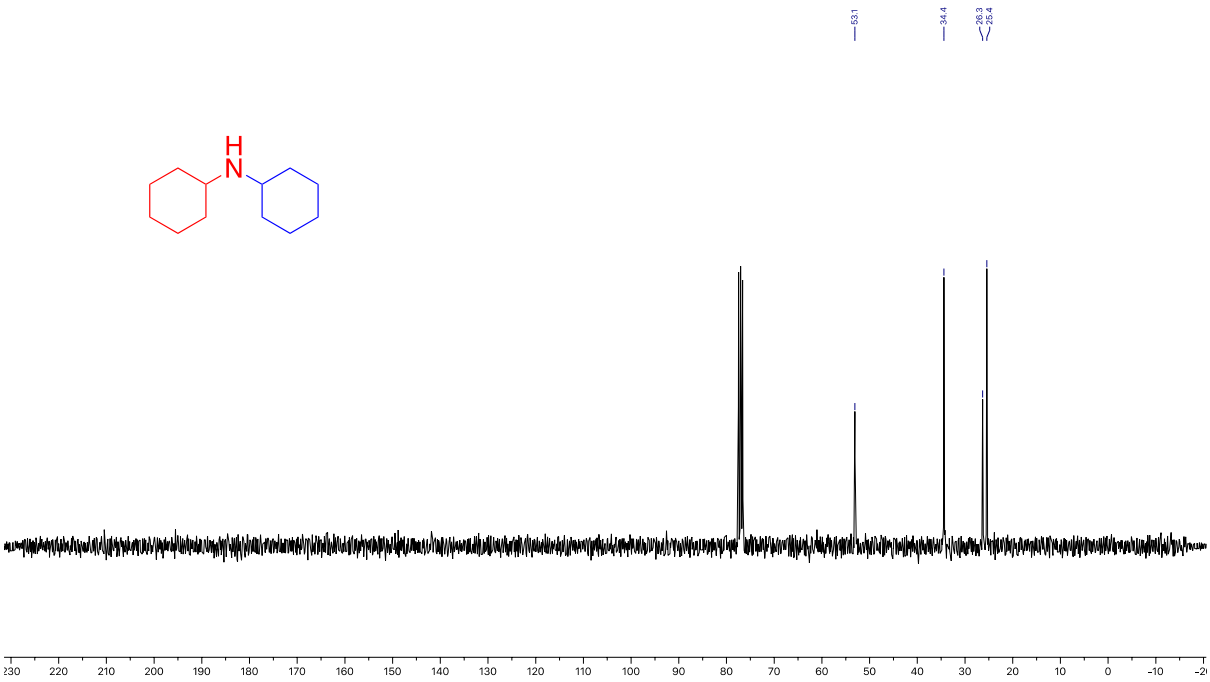


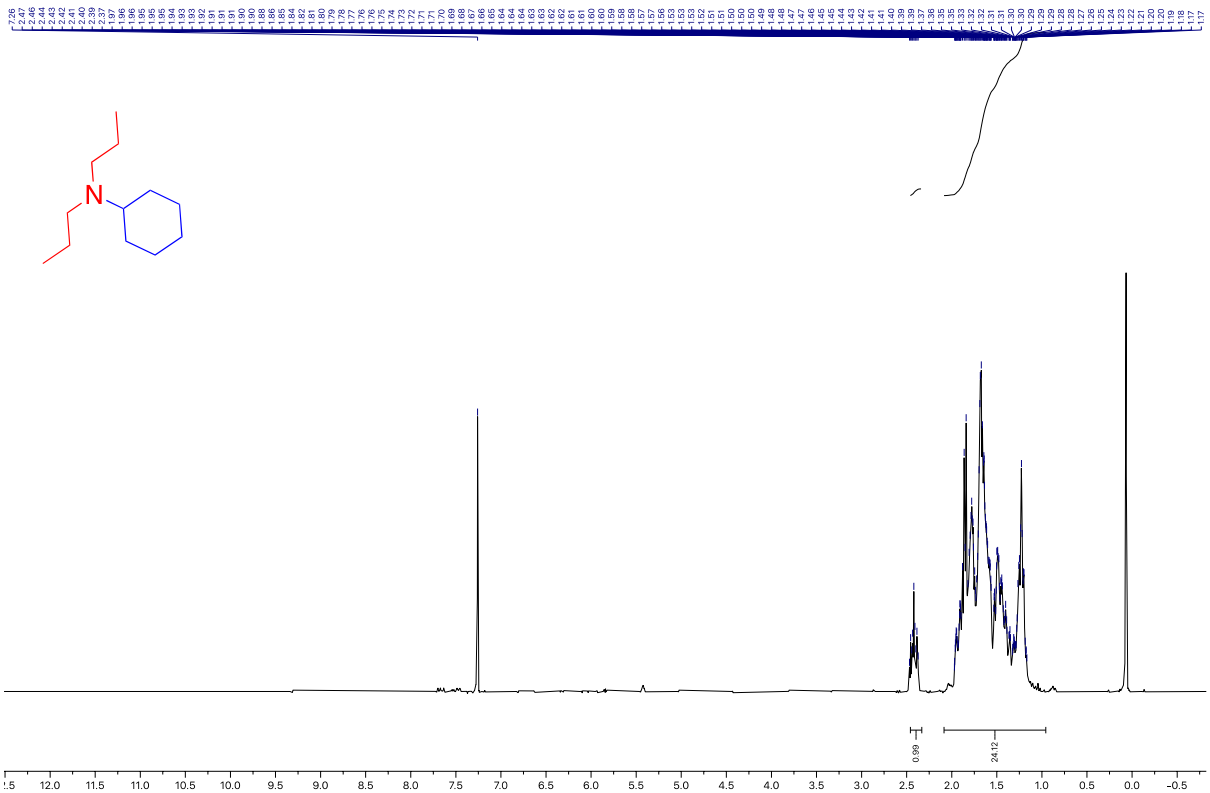
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ah**



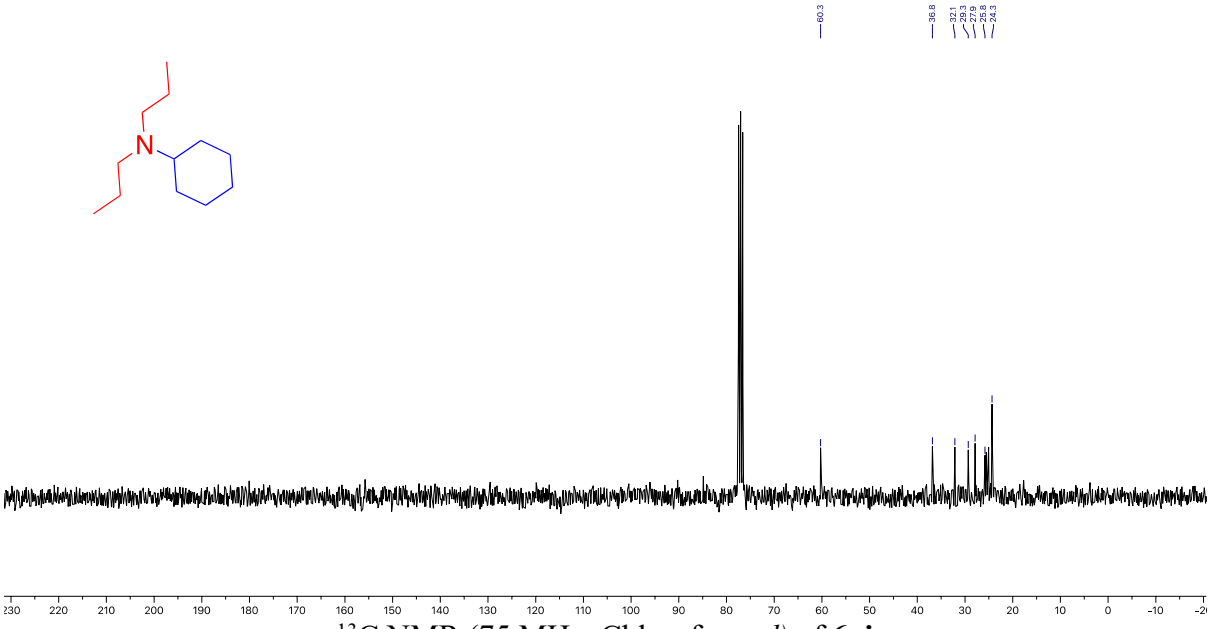
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ai**



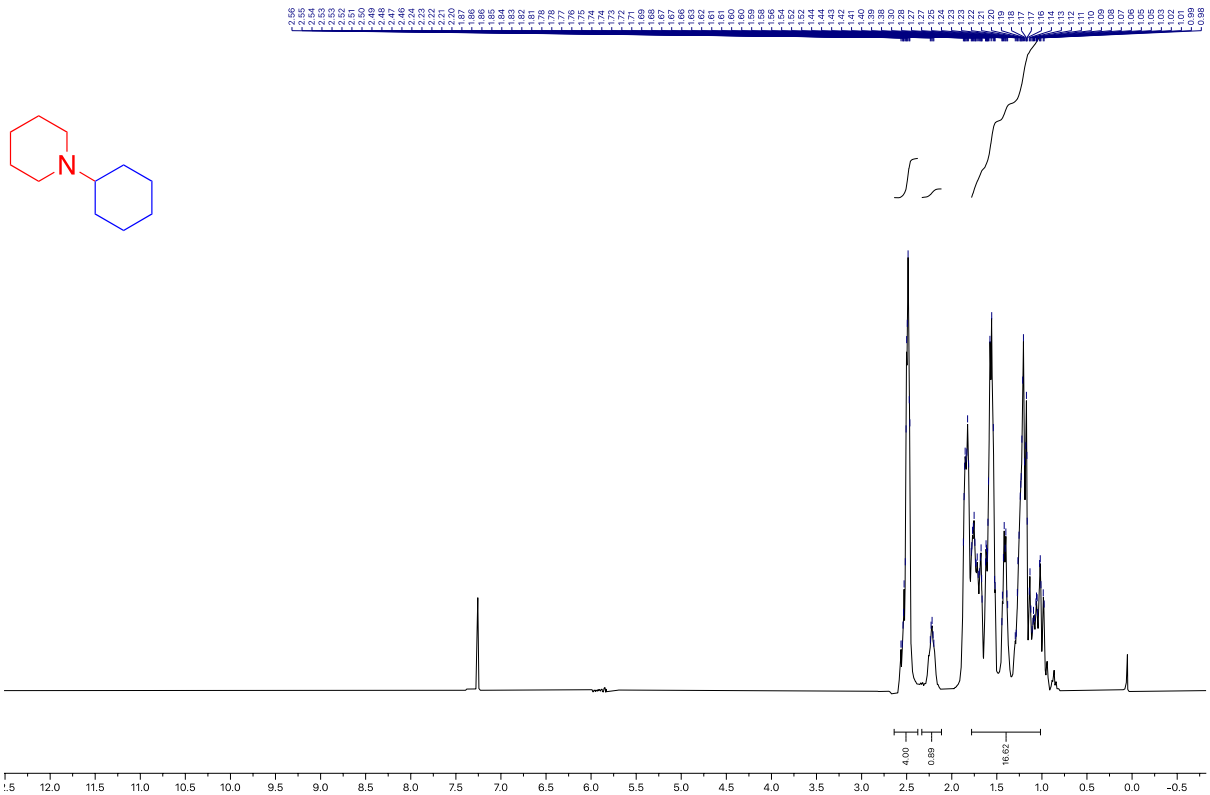
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ai**



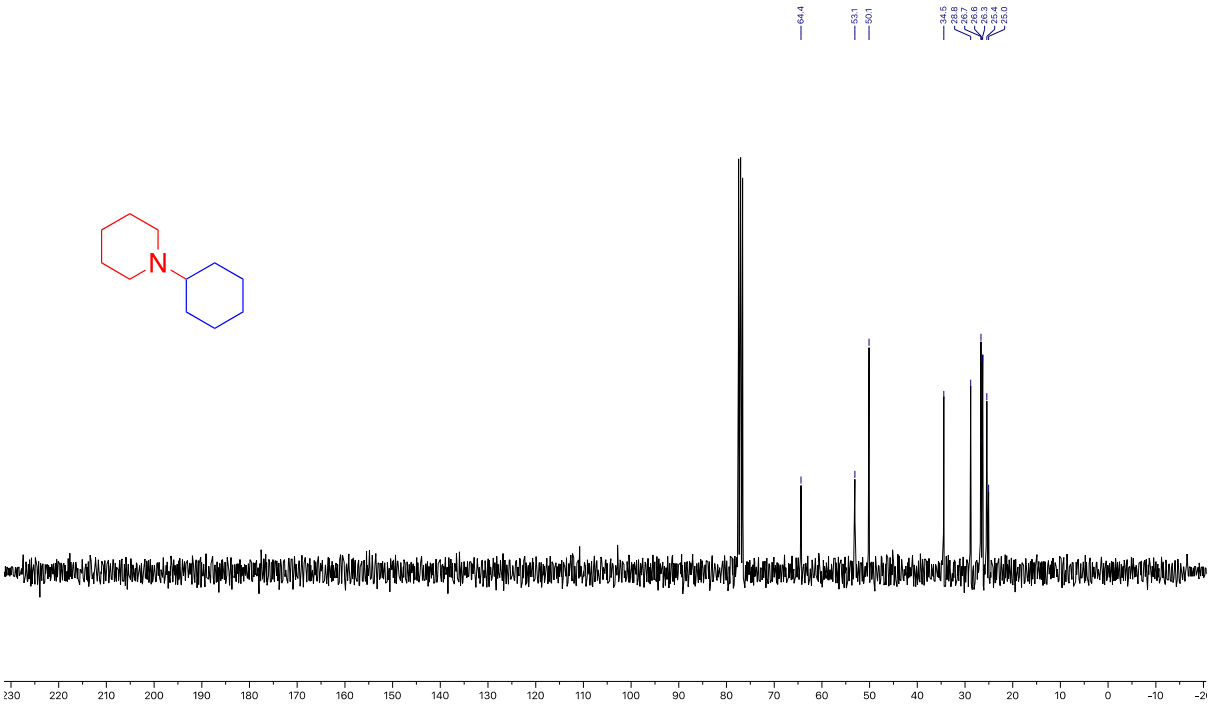
^{1}H NMR (300 MHz, Chloroform-*d)* of **6aj**



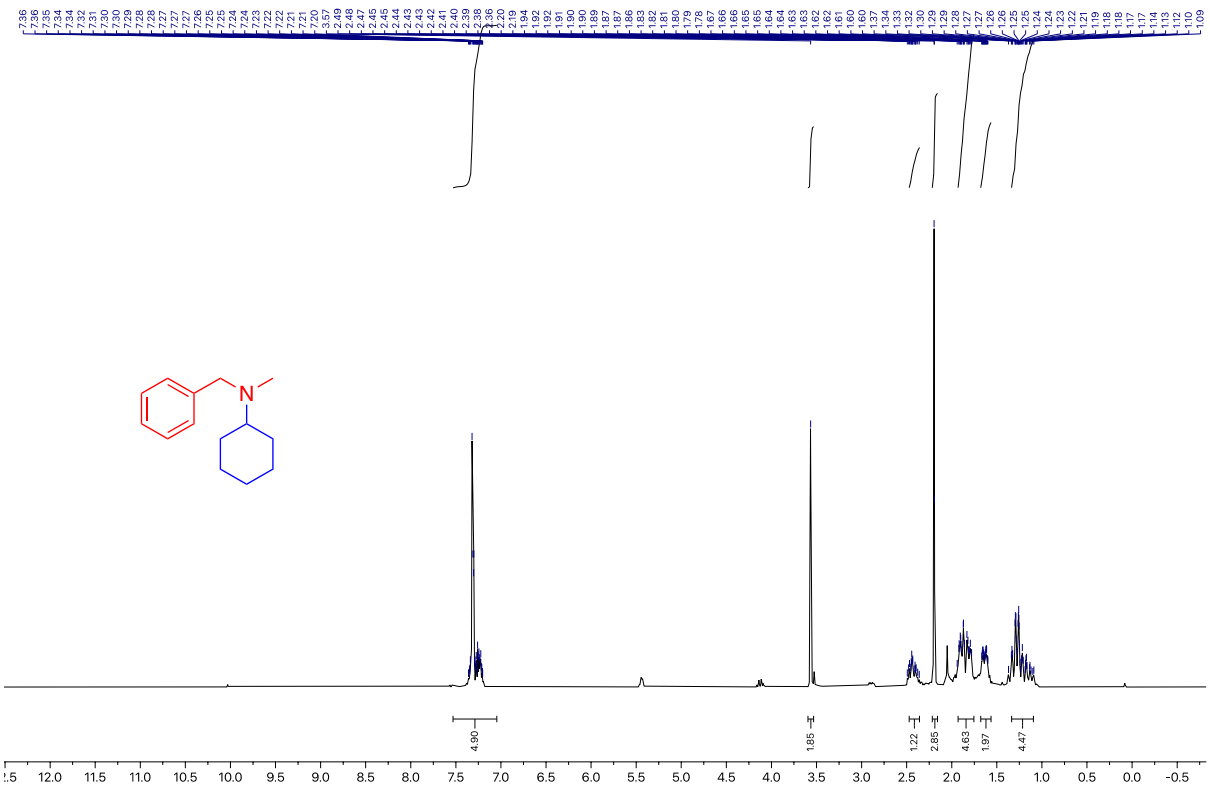
^{13}C NMR (75 MHz, Chloroform-*d)* of **6aj**



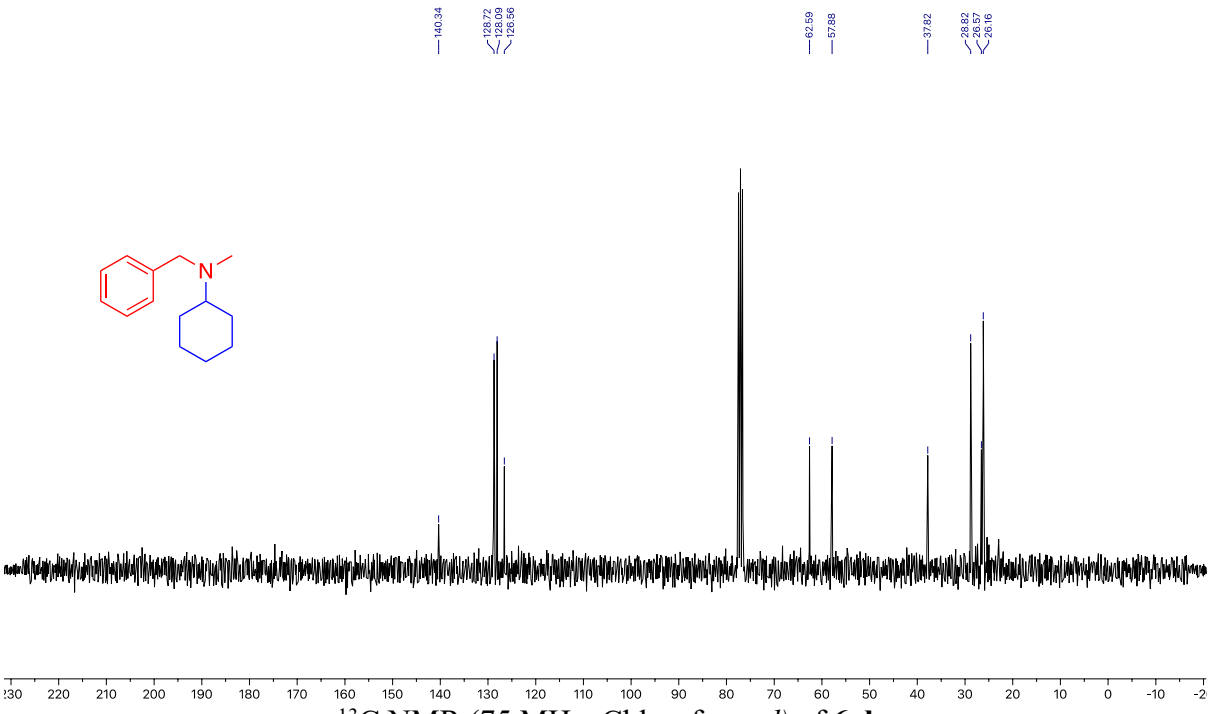
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ak**



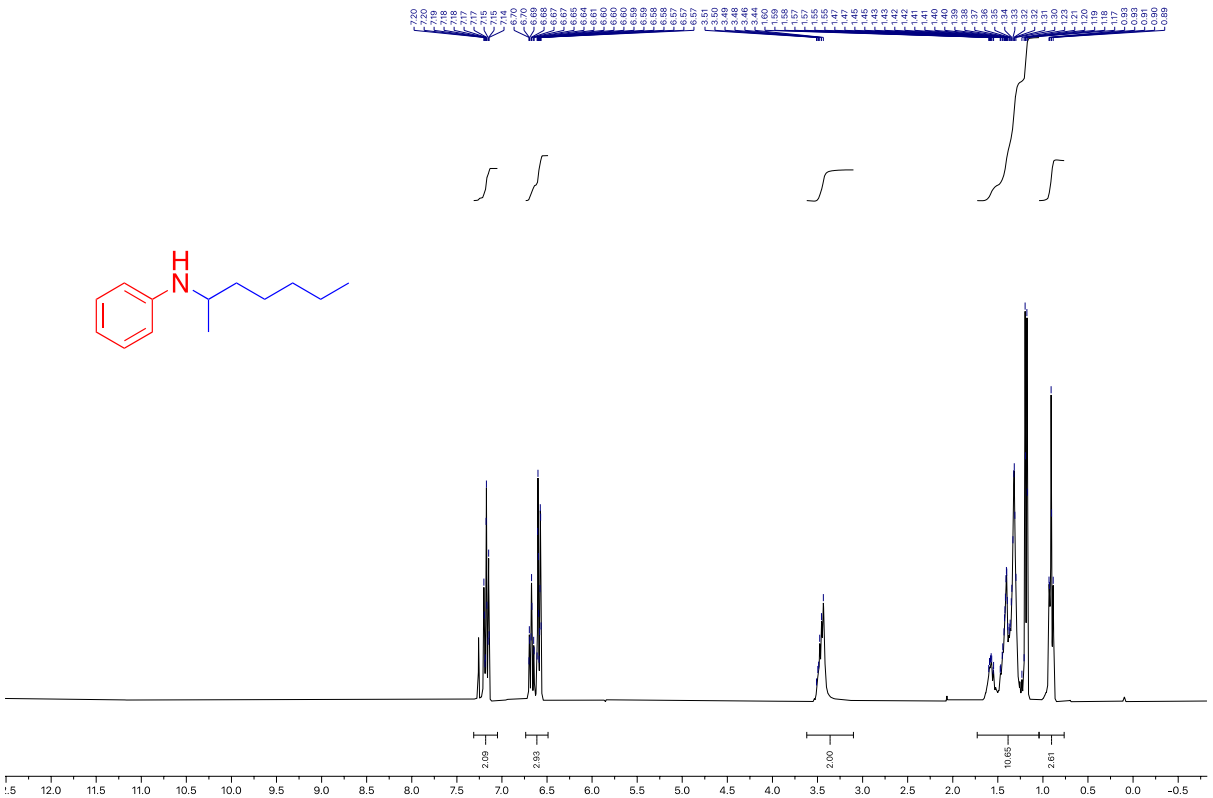
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ak**



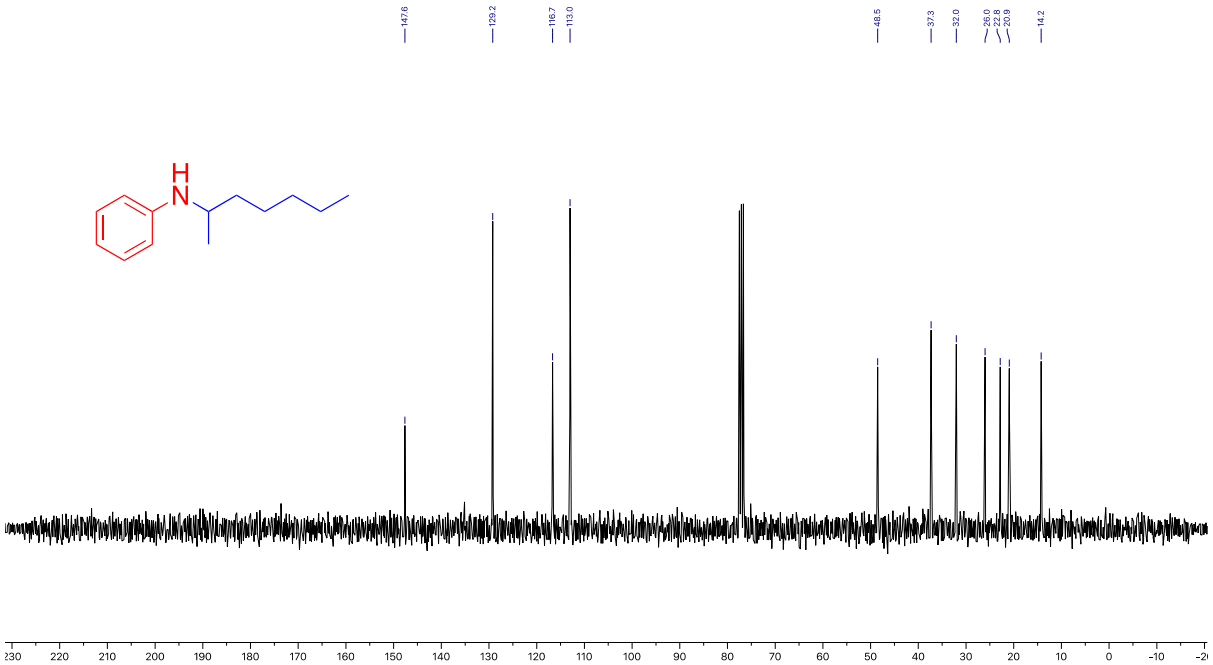
^{1}H NMR (300 MHz, Chloroform-*d)* of **6al**



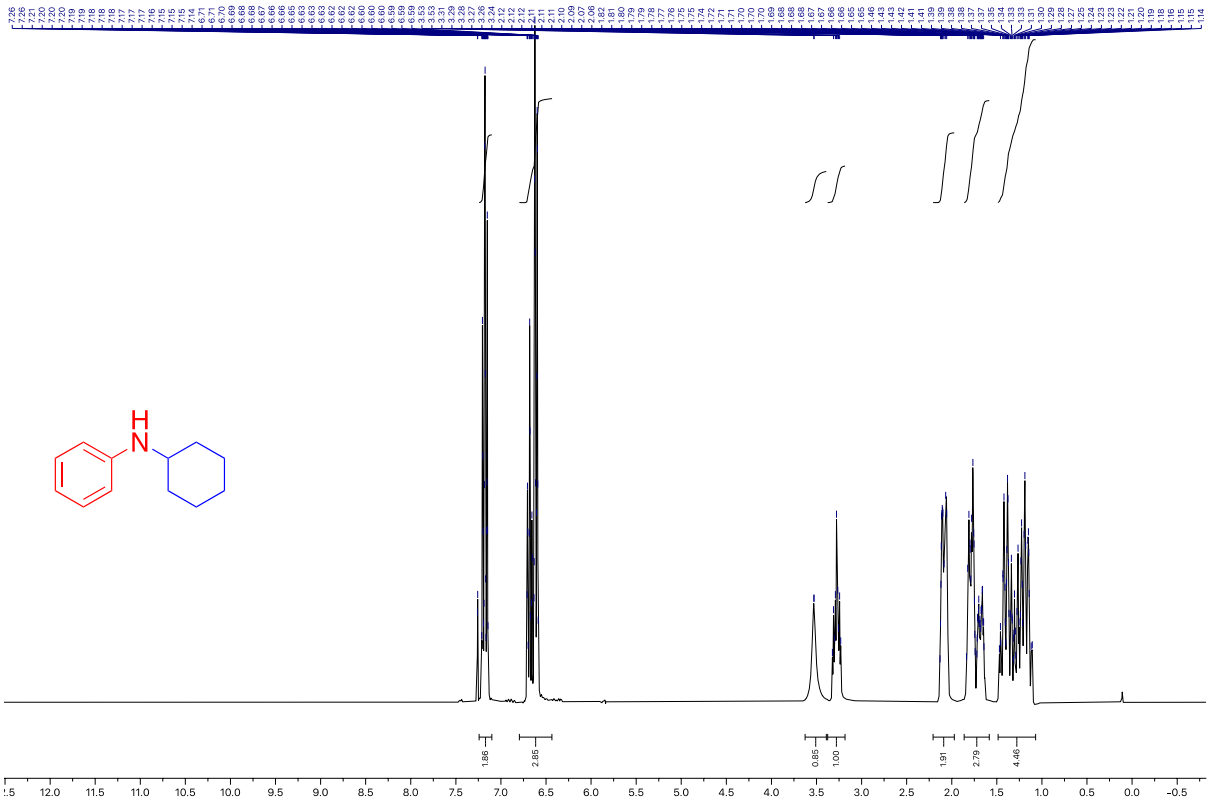
^{13}C NMR (75 MHz, Chloroform-*d)* of **6al**



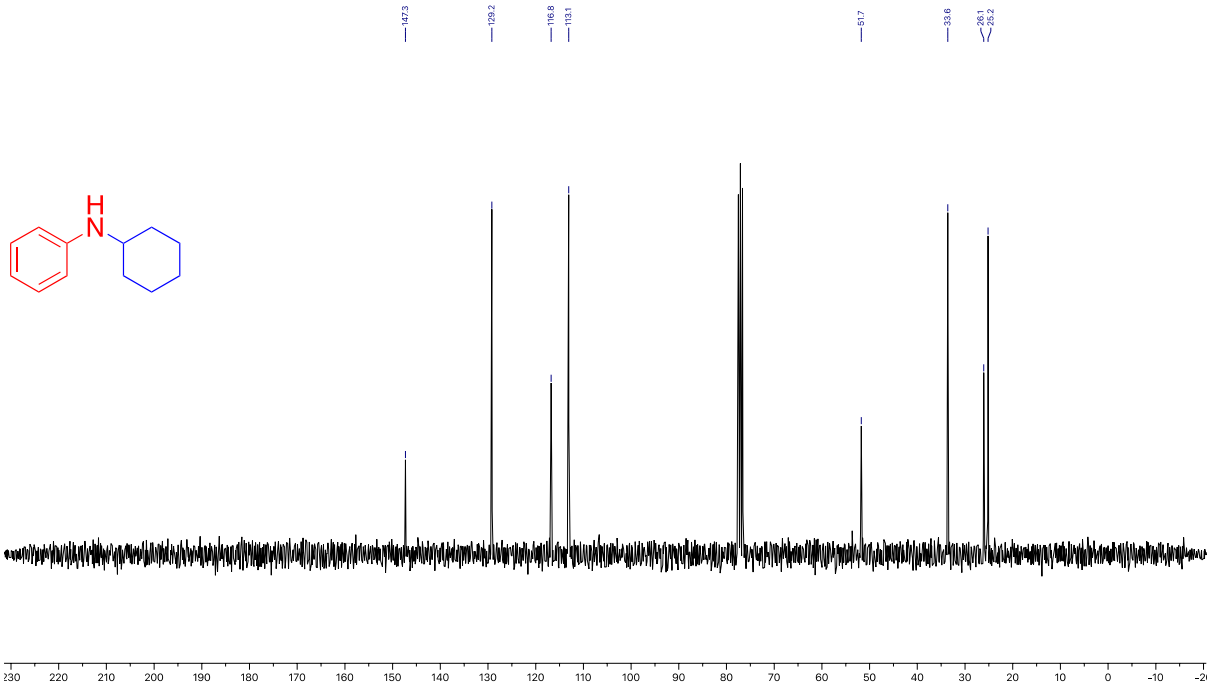
^{1}H NMR (300 MHz, Chloroform-*d)* of **6cm**



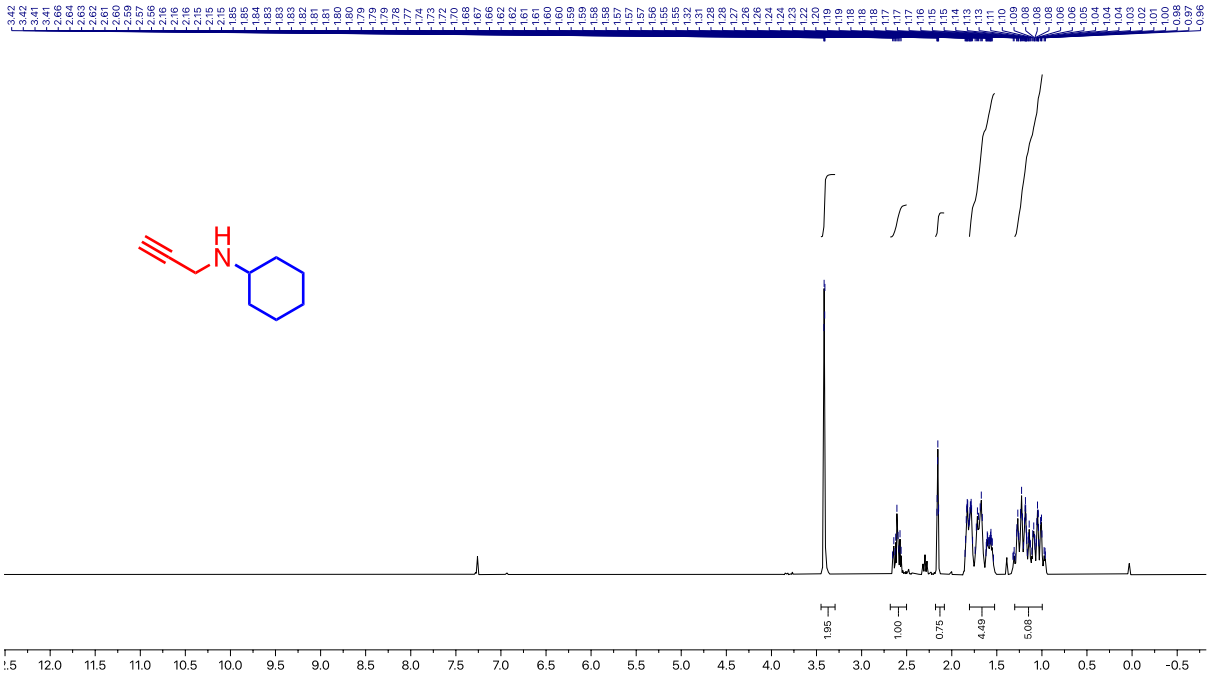
^{13}C NMR (75 MHz, Chloroform-*d)* of **6cm**



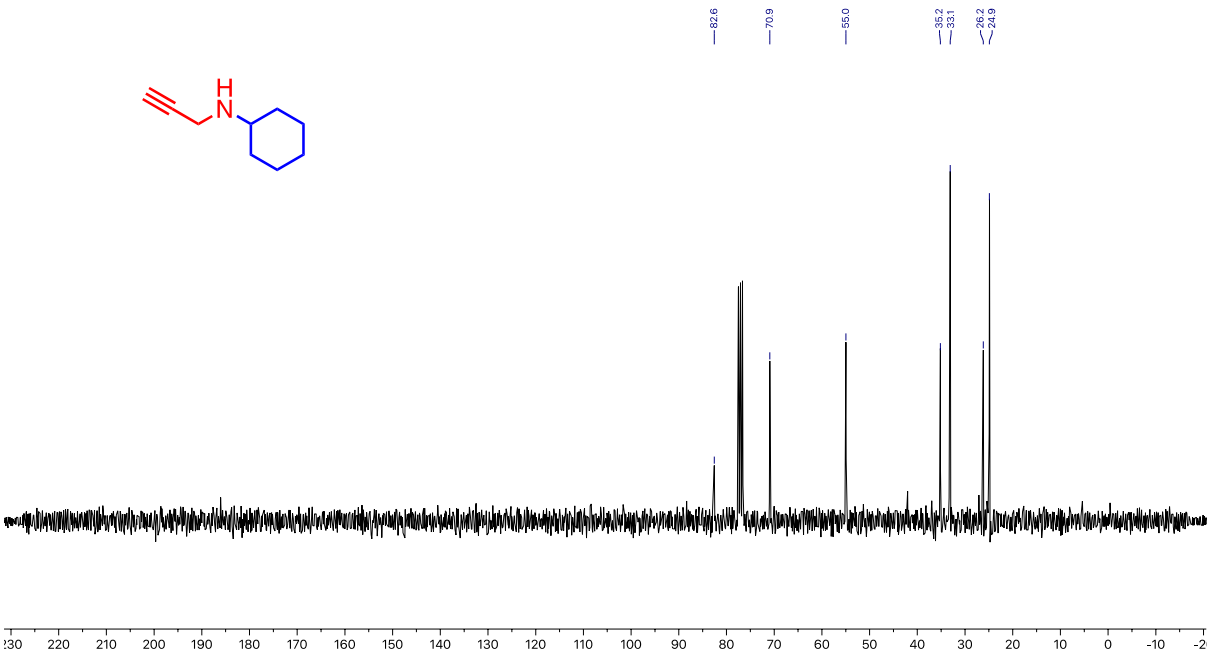
^{1}H NMR (300 MHz, Chloroform-*d)* of **6am**



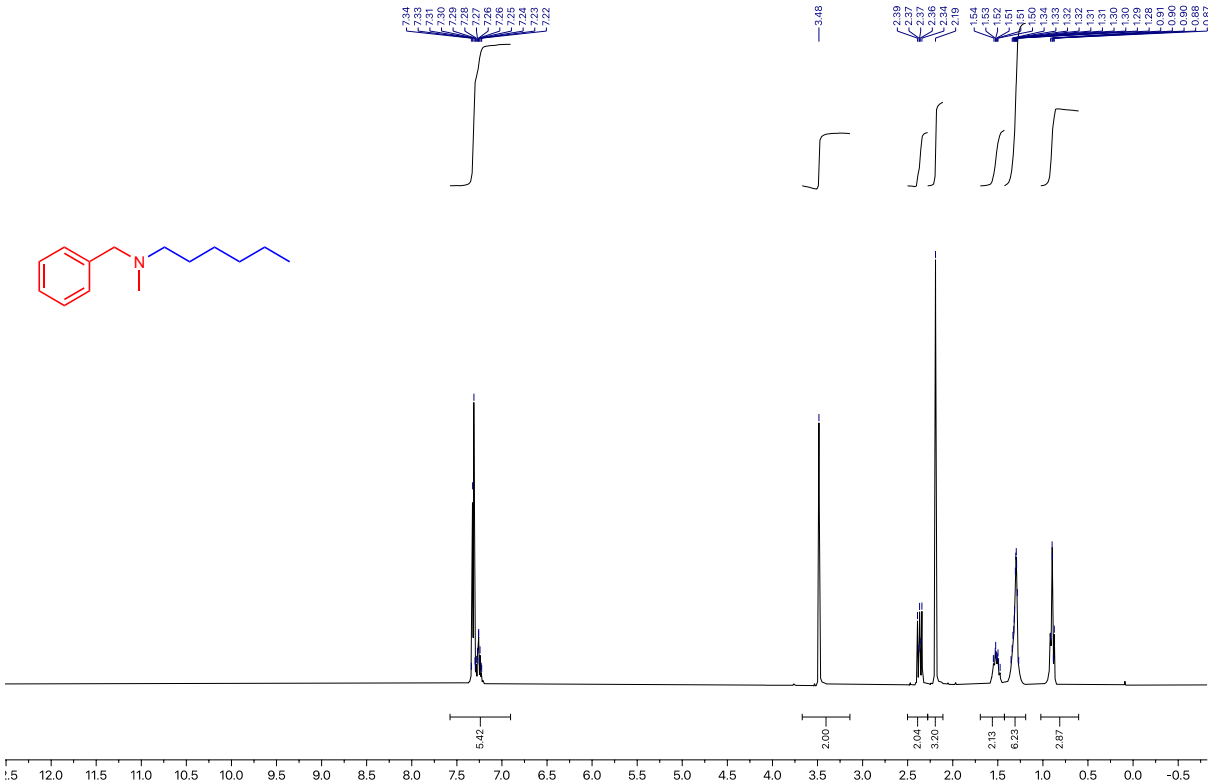
^{13}C NMR (75 MHz, Chloroform-*d)* of **6am**



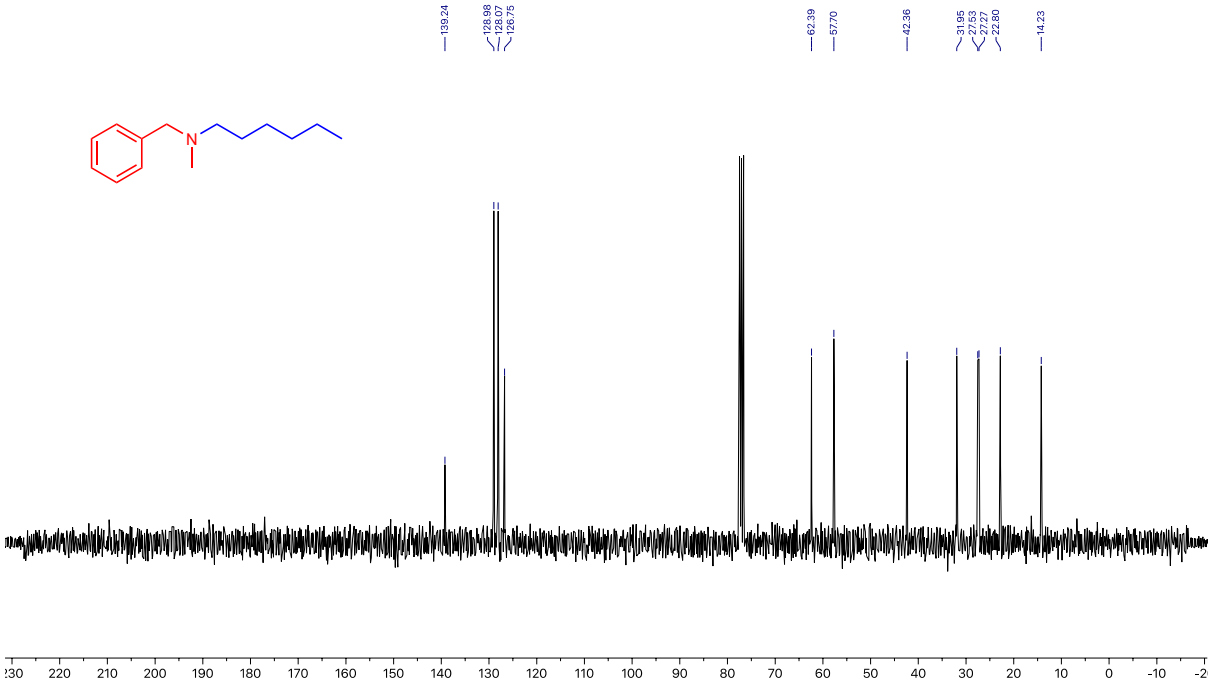
^{1}H NMR (300 MHz, Chloroform-*d)* of **6as**



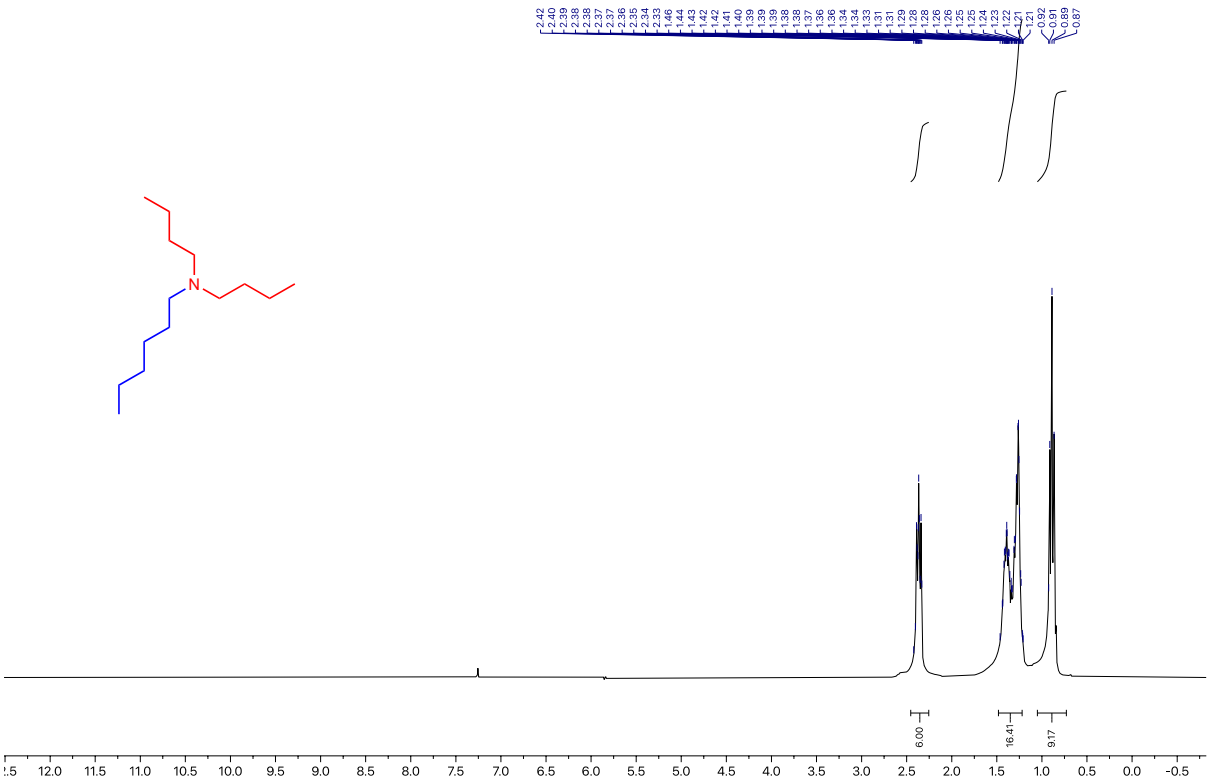
^{13}C NMR (75 MHz, Chloroform-*d)* of **6as**



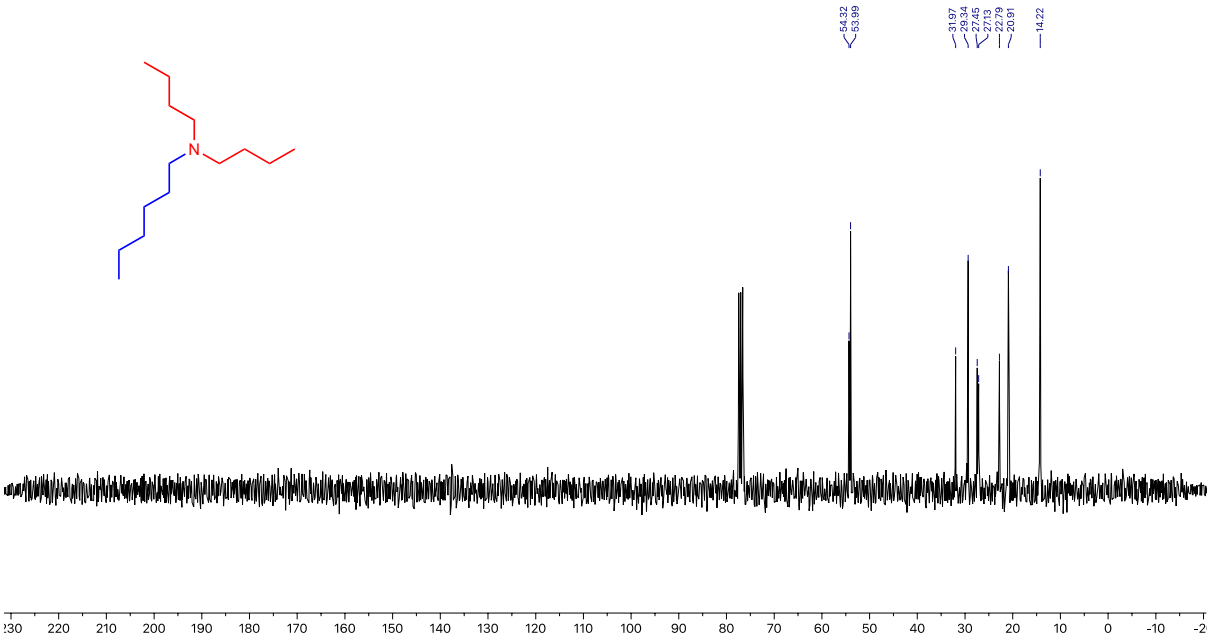
^{1}H NMR (300 MHz, Chloroform-*d)* of **7bl**



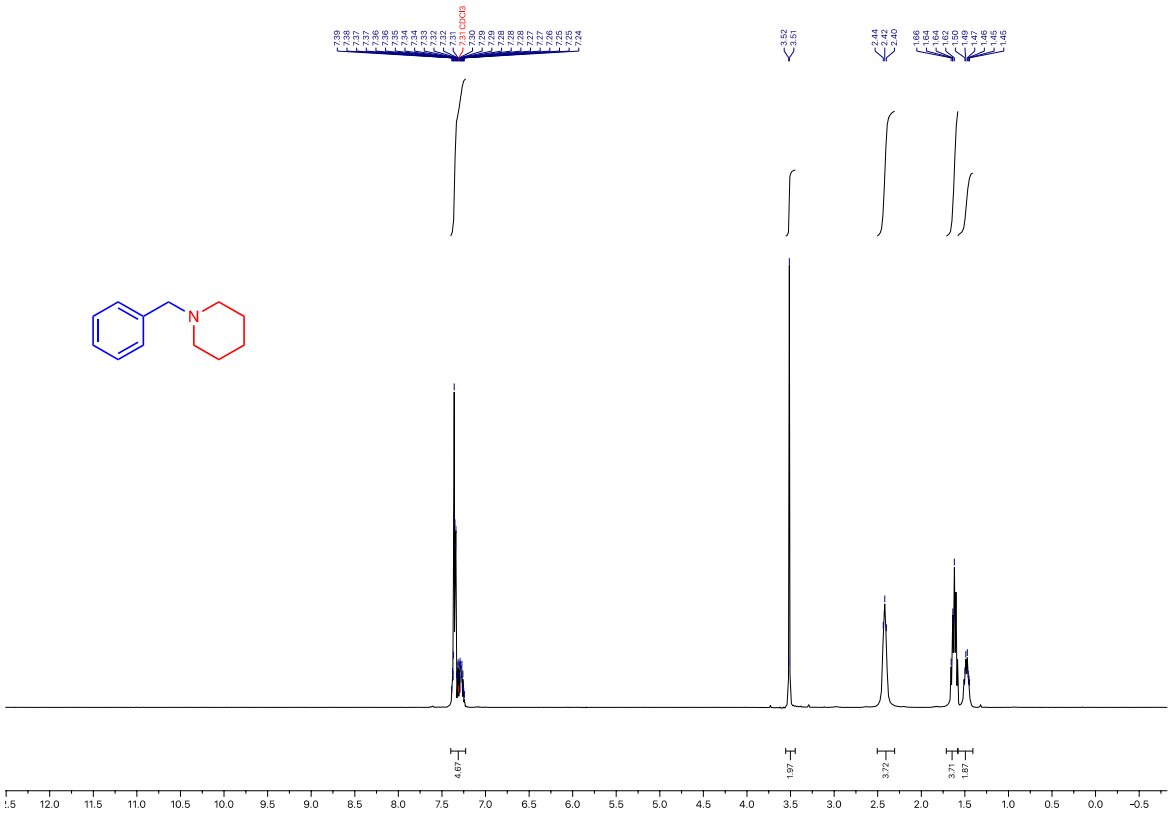
^{13}C NMR (75 MHz, Chloroform-*d)* of **7bl**



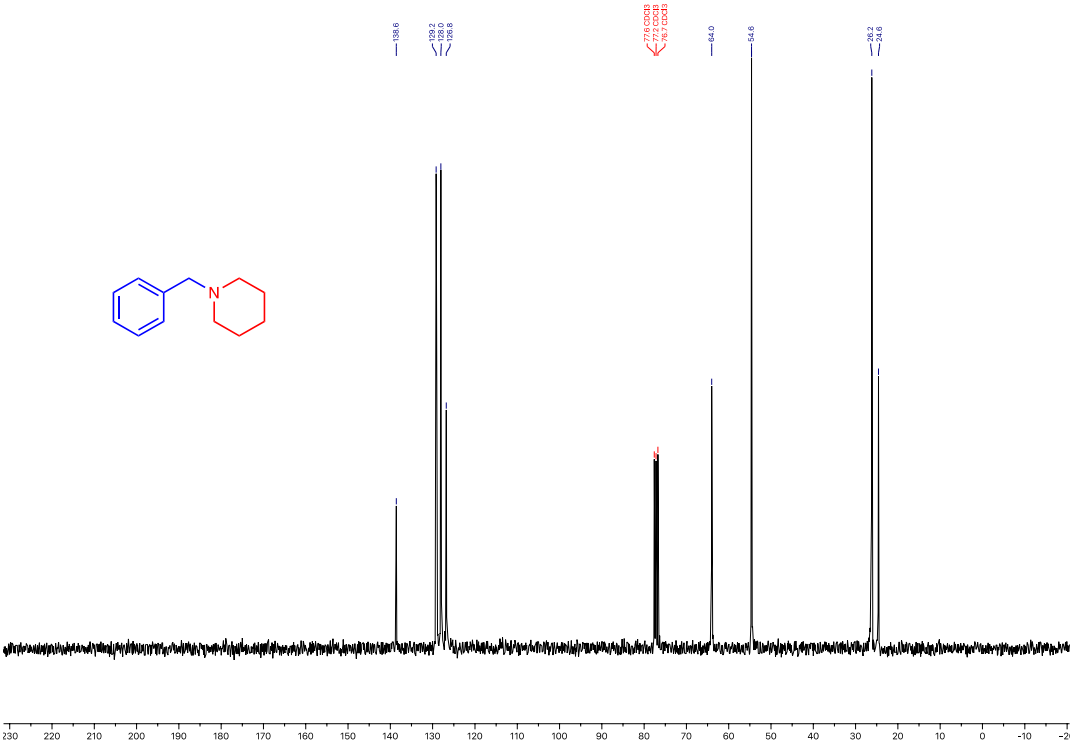
^{1}H NMR (300 MHz, Chloroform-*d)* of **7br**



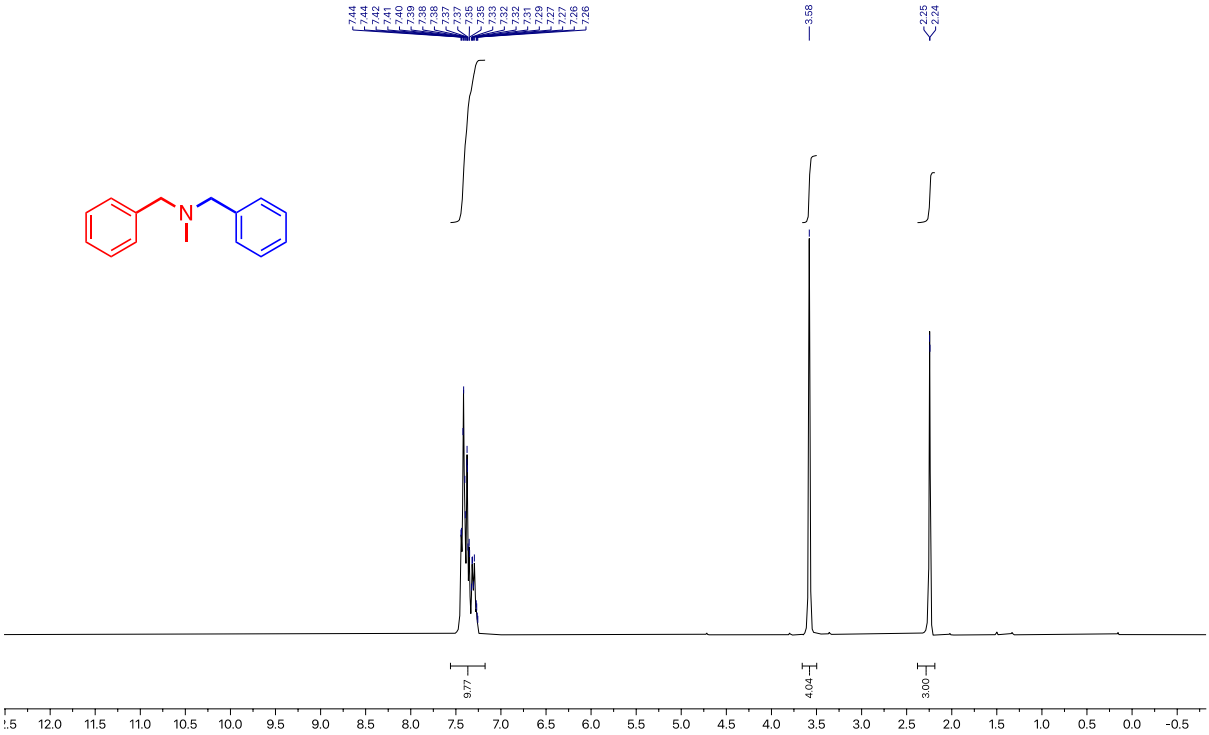
^{13}C NMR (75 MHz, Chloroform-*d)* of **7br**



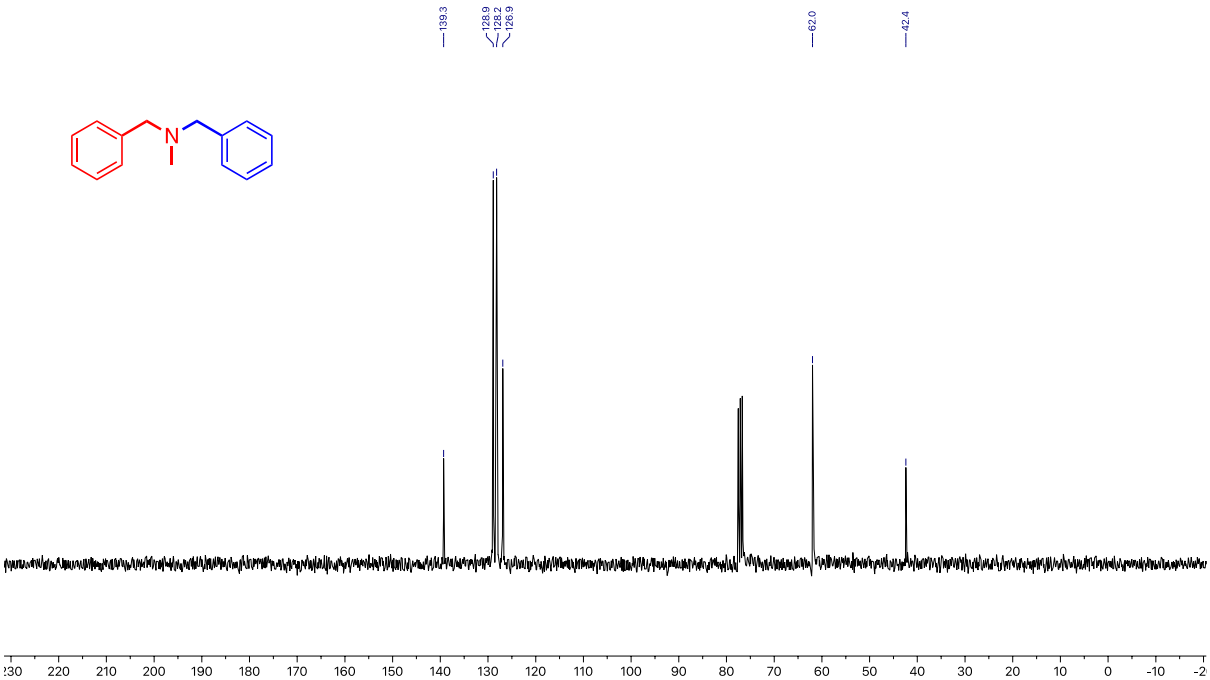
^{1}H NMR (300 MHz, Chloroform-*d)* of **7ak**



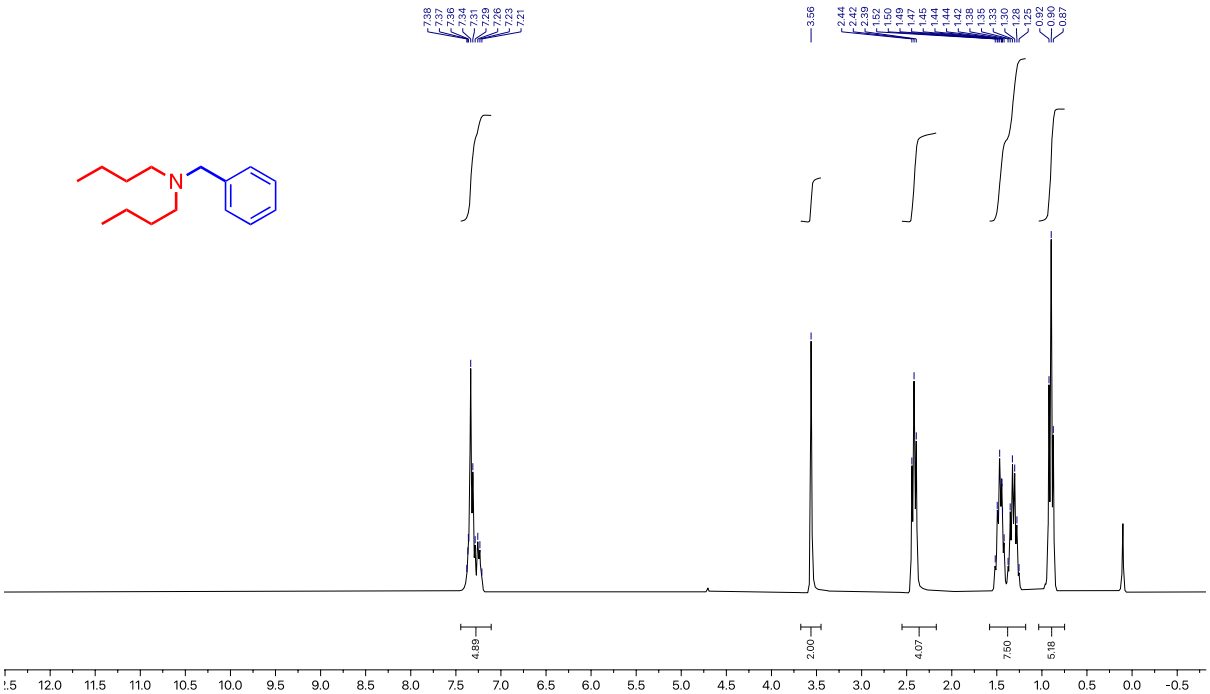
^{13}C NMR (75 MHz, Chloroform-*d)* of **7ak**



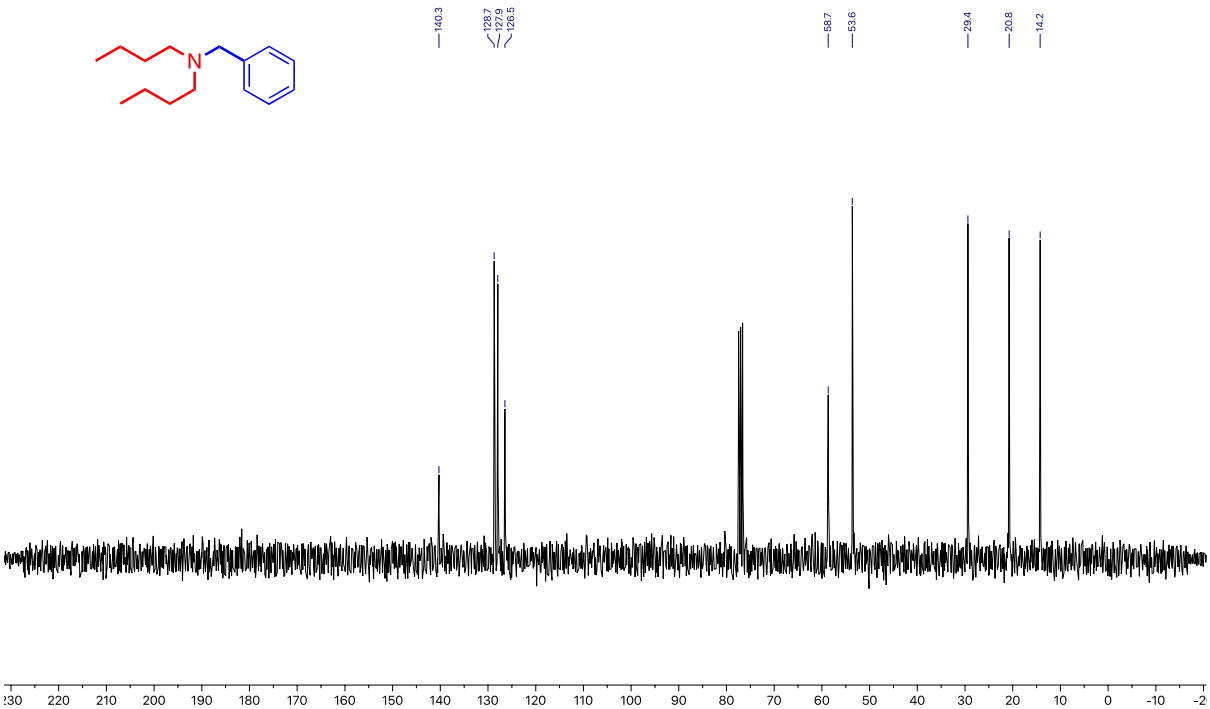
^{1}H NMR (300 MHz, Chloroform-*d)* of **7al**



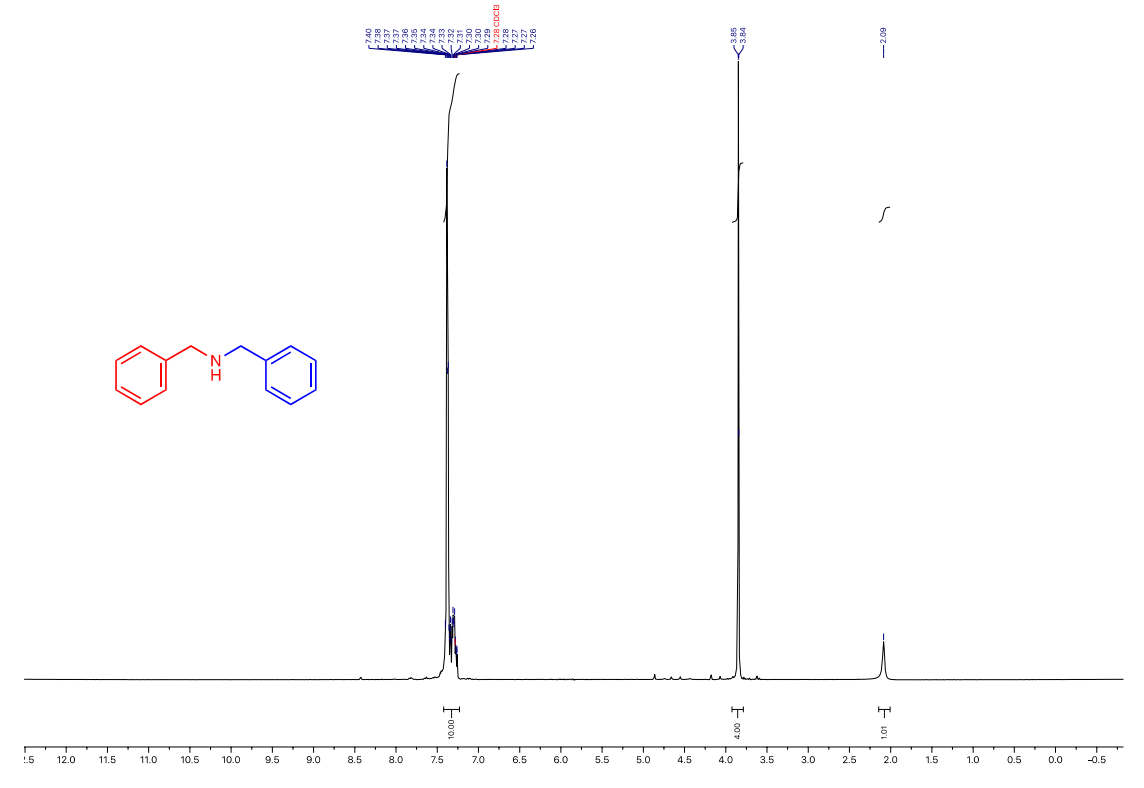
^{13}C NMR (75 MHz, Chloroform-*d)* of **7al**



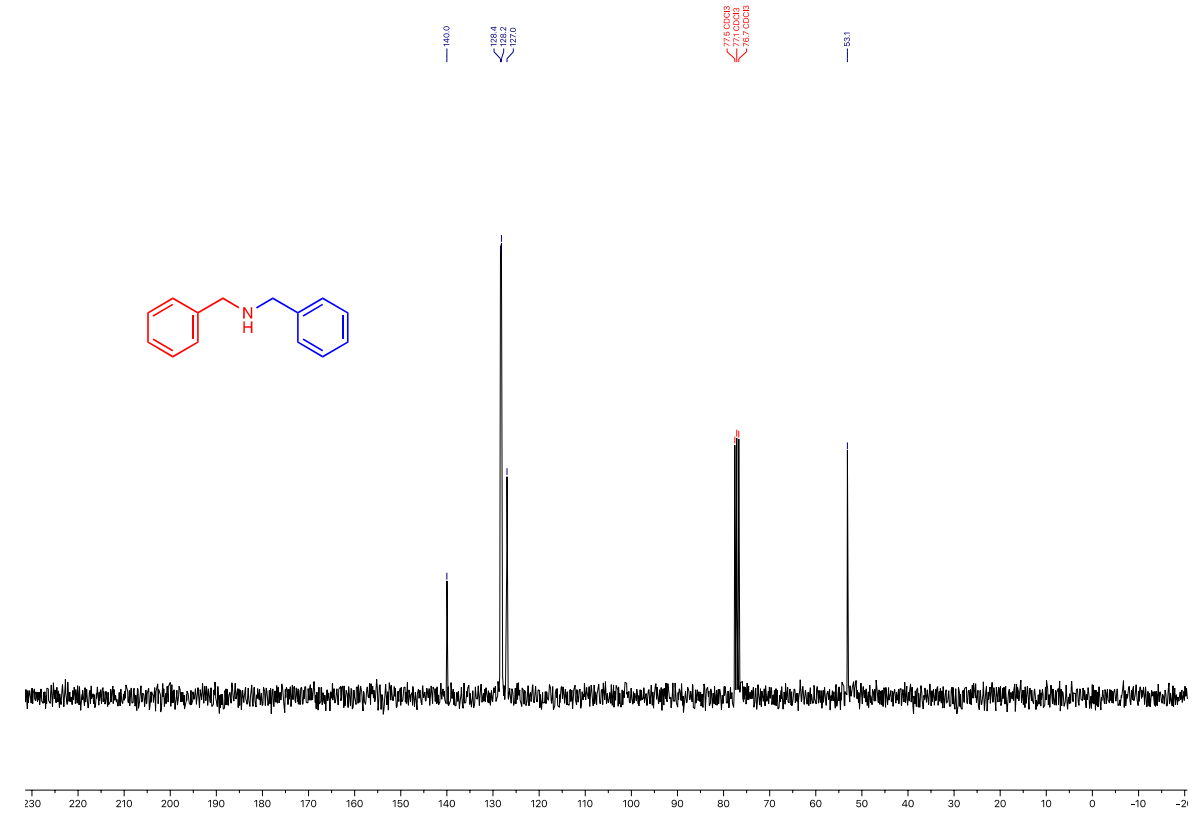
^{1}H NMR (300 MHz, Chloroform-*d)* of **7ar**



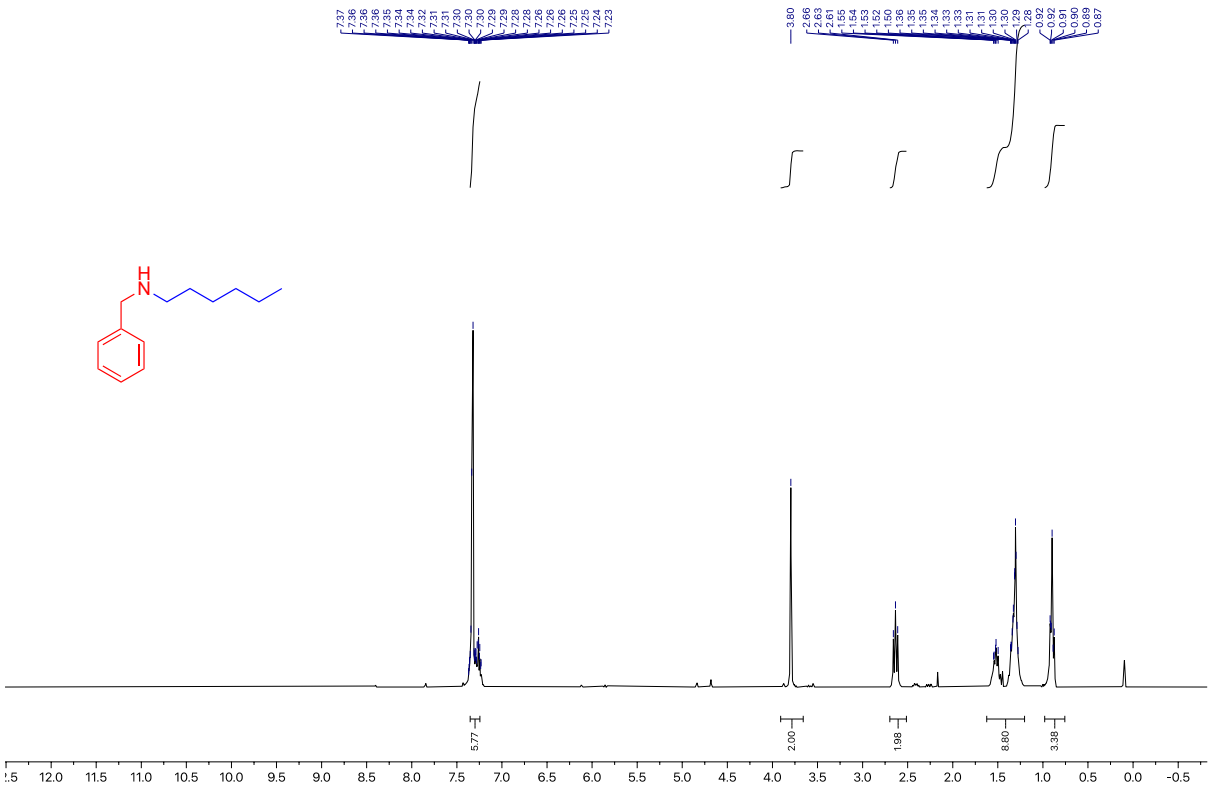
^{13}C NMR (75 MHz, Chloroform-*d)* of **7ar**



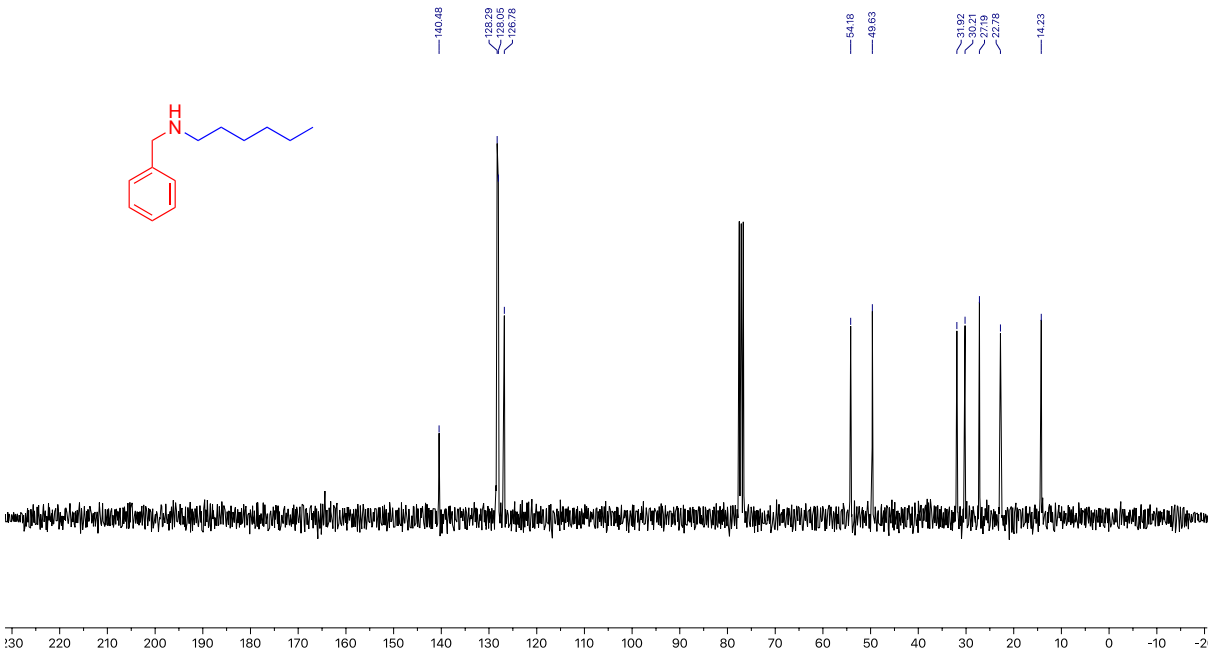
^{1}H NMR (300 MHz, Chloroform-*d)* of **7aa**



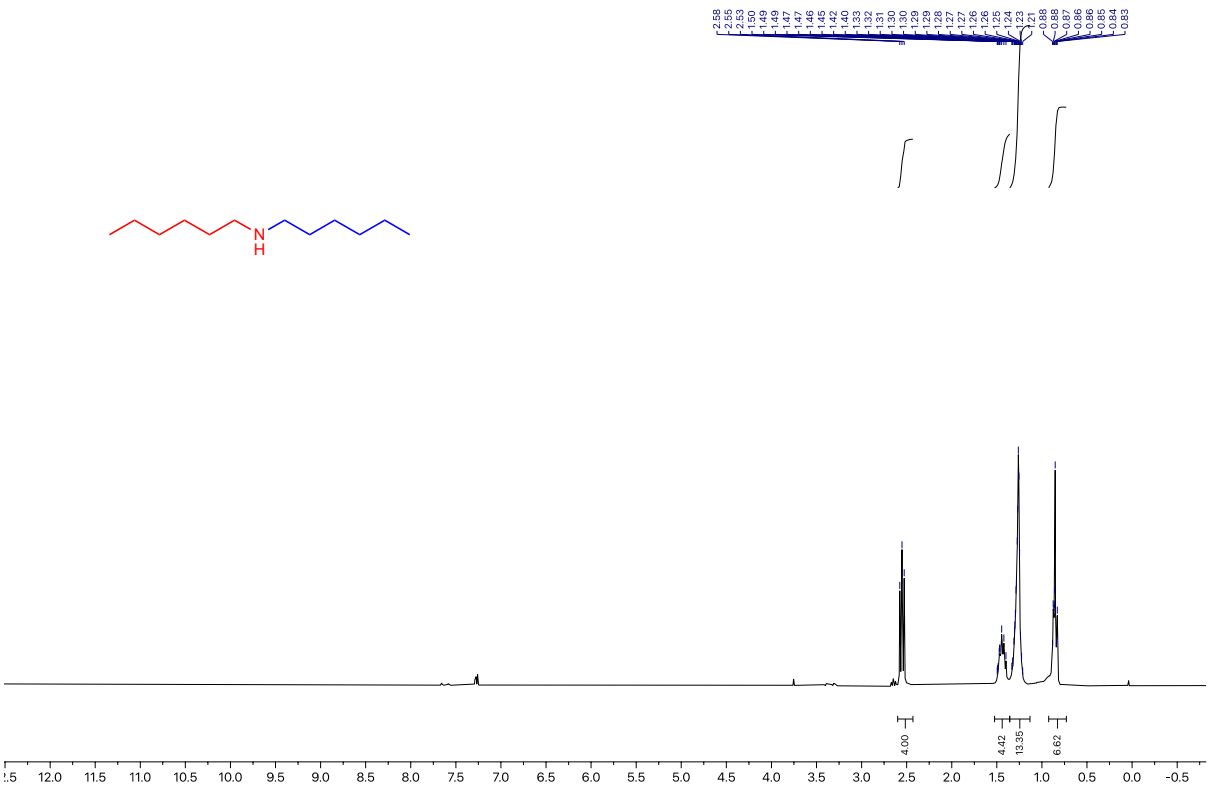
^{13}C NMR (75 MHz, Chloroform-*d)* of **7aa**



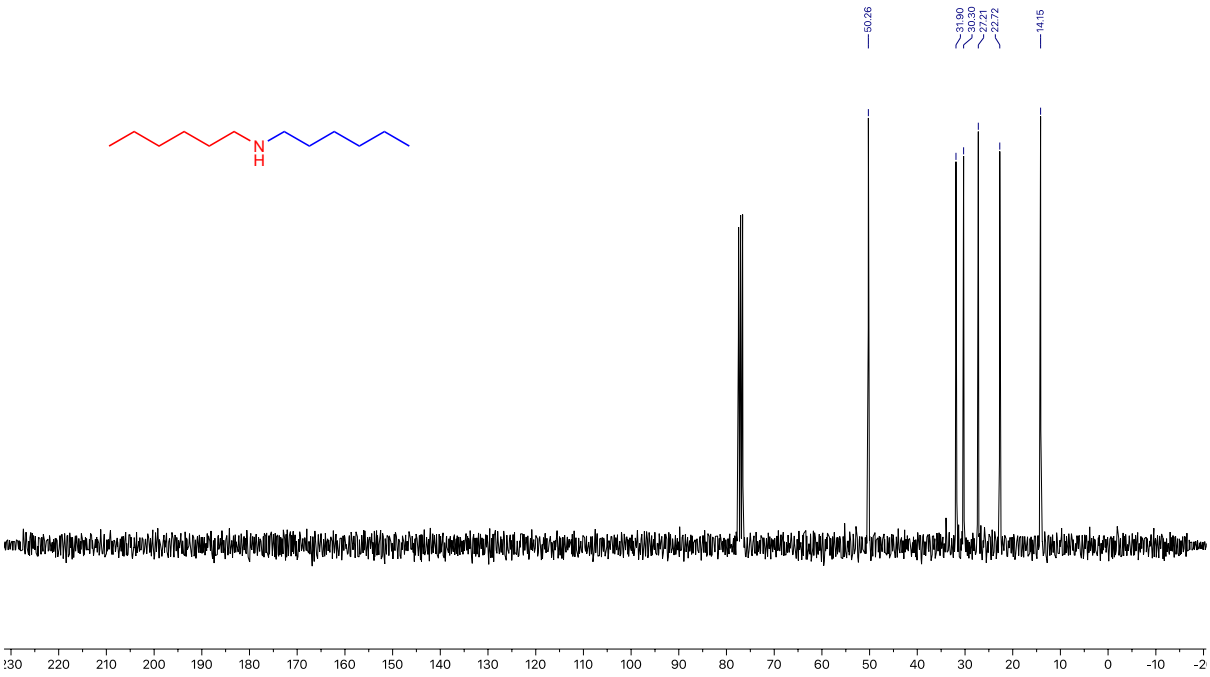
^{1}H NMR (300 MHz, Chloroform-*d)* of **7ca**



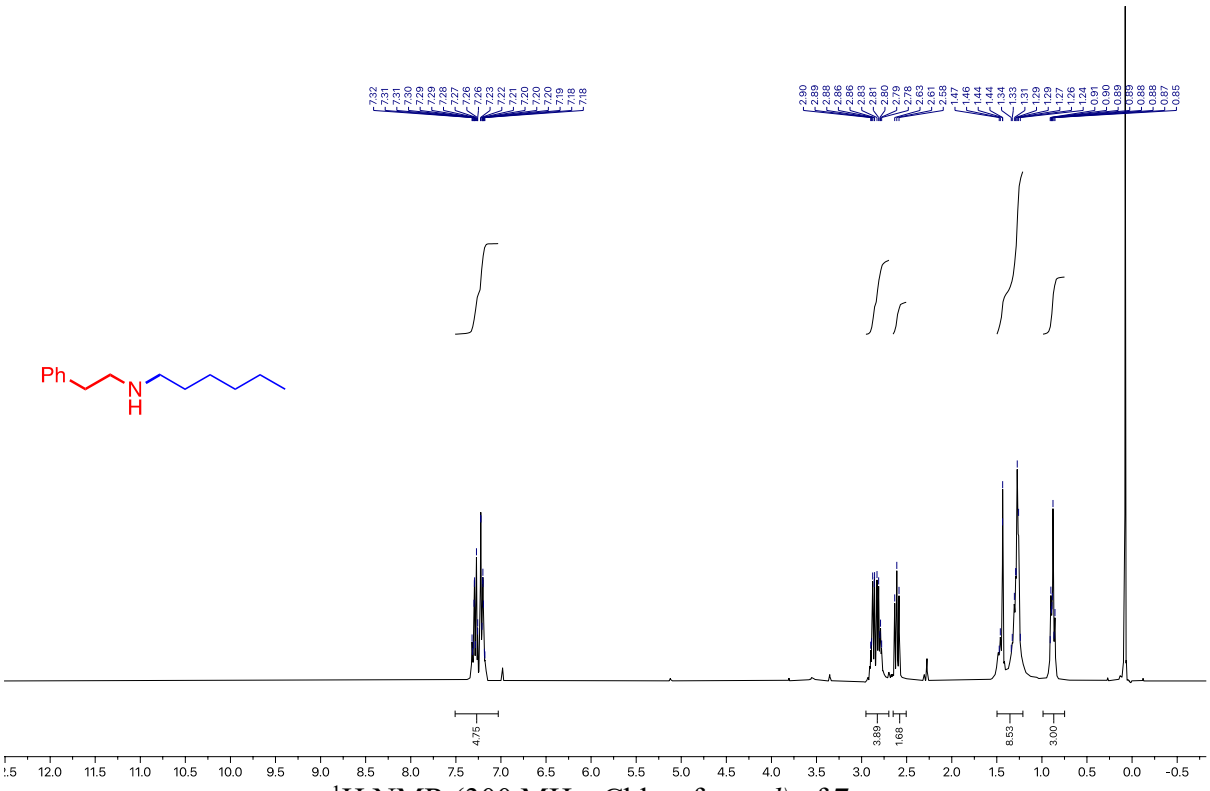
^{13}C NMR (75 MHz, Chloroform-*d)* of **7ca**



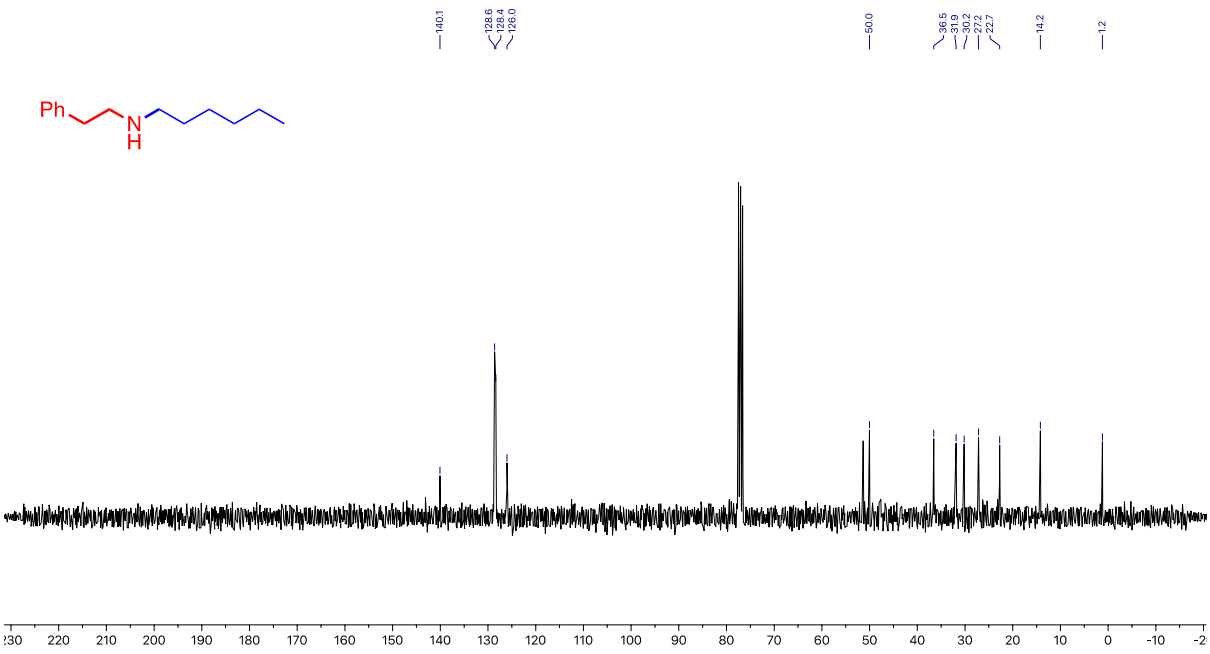
^{1}H NMR (300 MHz, Chloroform-*d)* of **7ch**



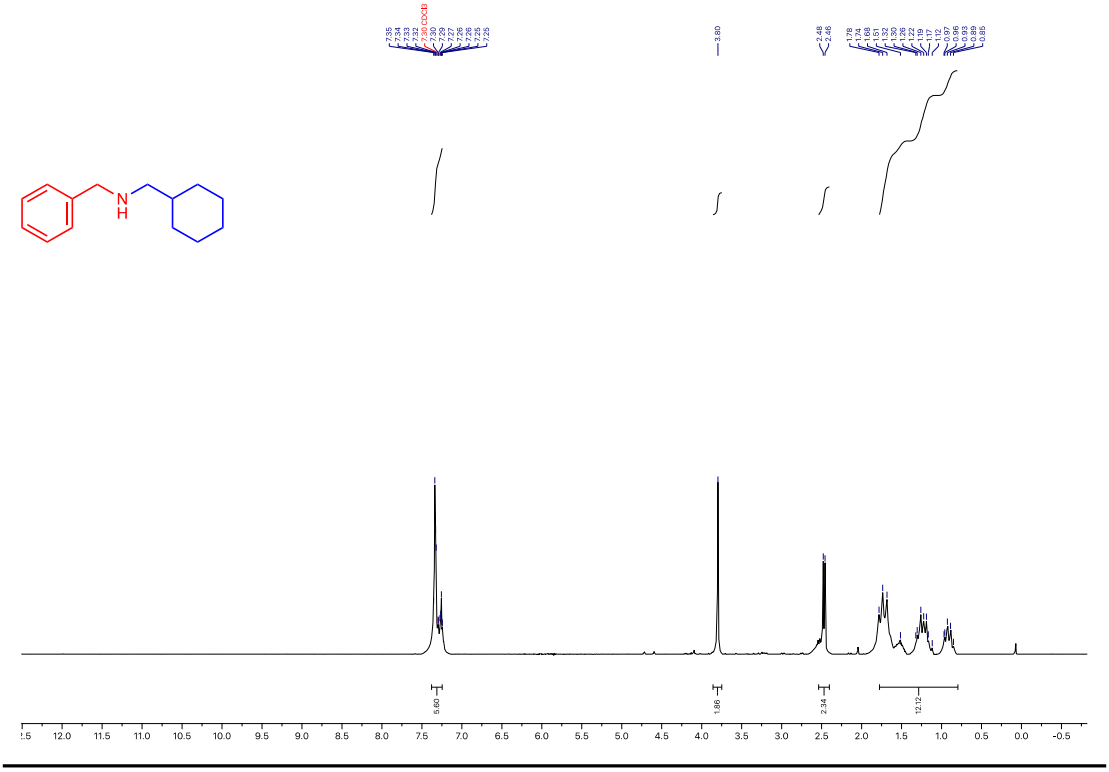
^{13}C NMR (75 MHz, Chloroform-*d)* of **7ch**



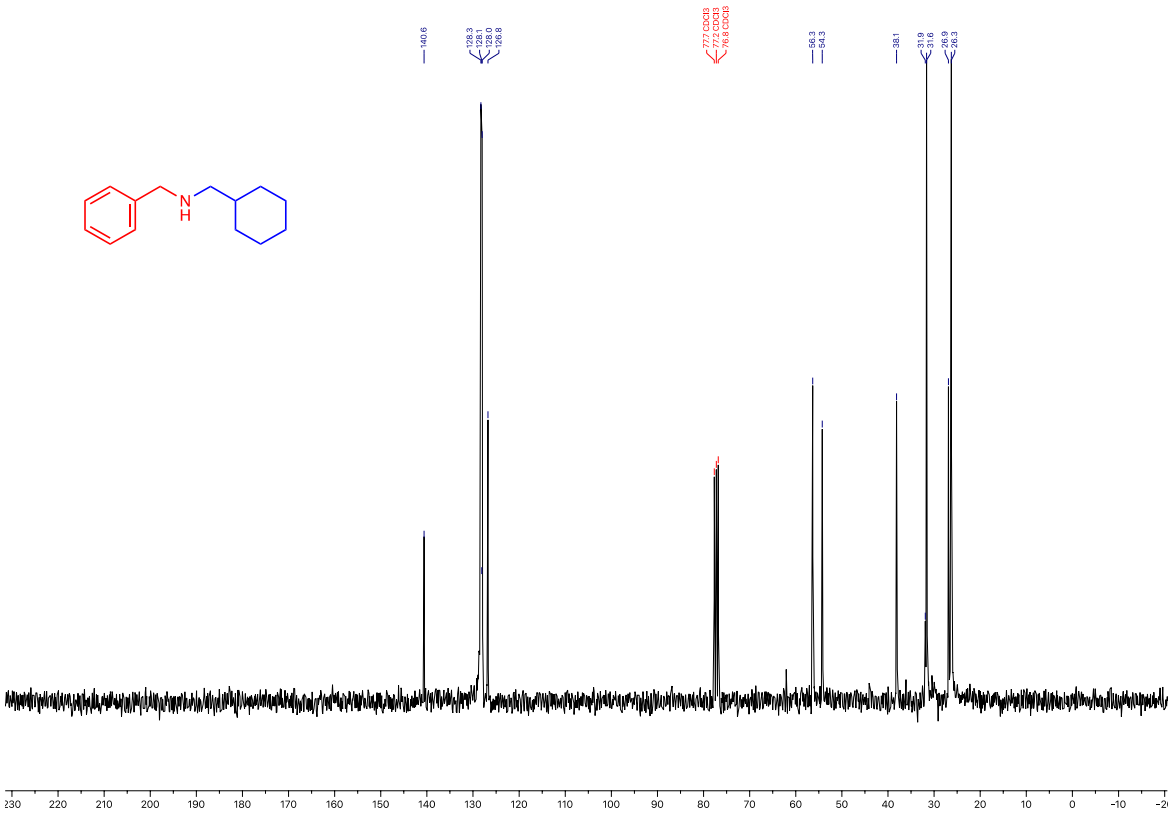
^{1}H NMR (300 MHz, Chloroform-*d)* of **7cg**



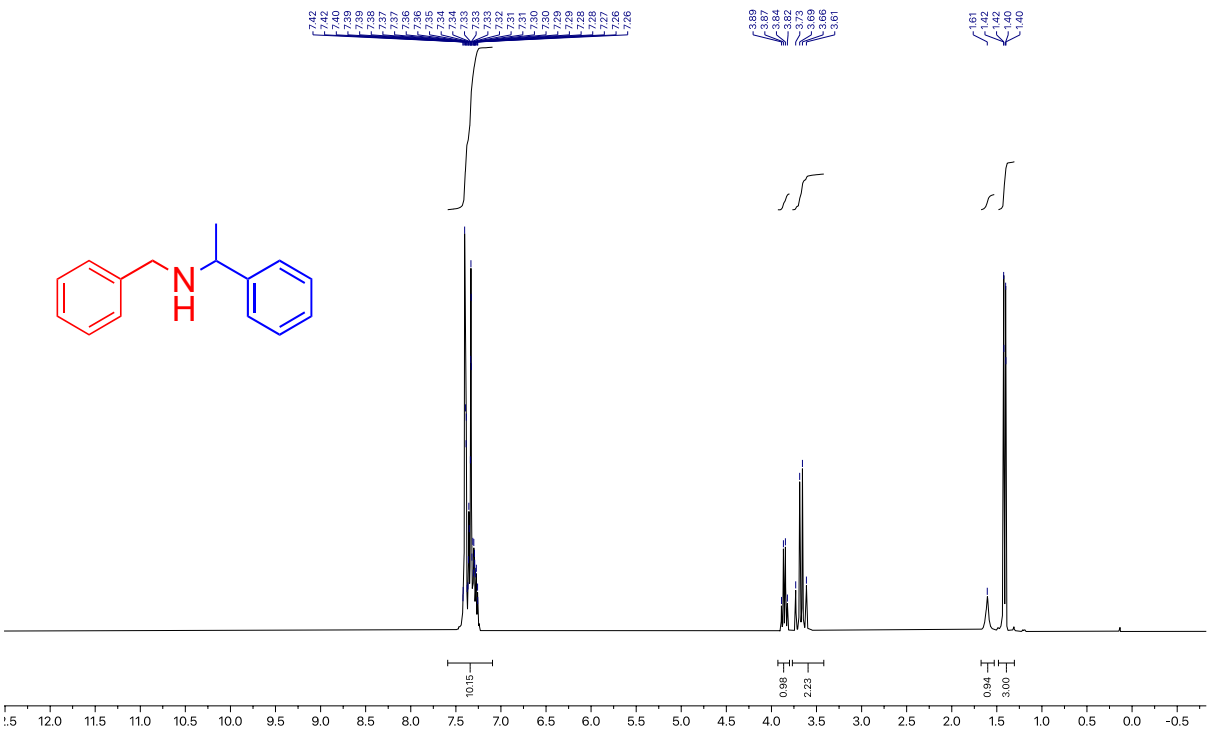
^{13}C NMR (75 MHz, Chloroform-*d)* of **7cg**



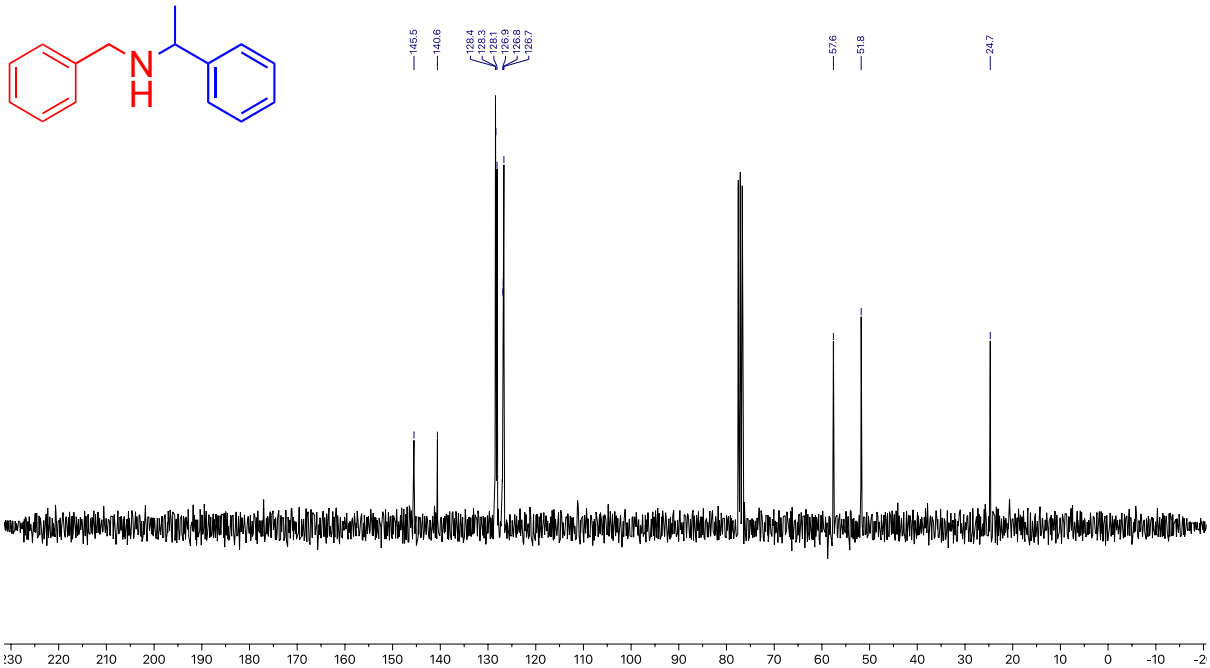
^{1}H NMR (300 MHz, Chloroform-*d)* of **7ba**



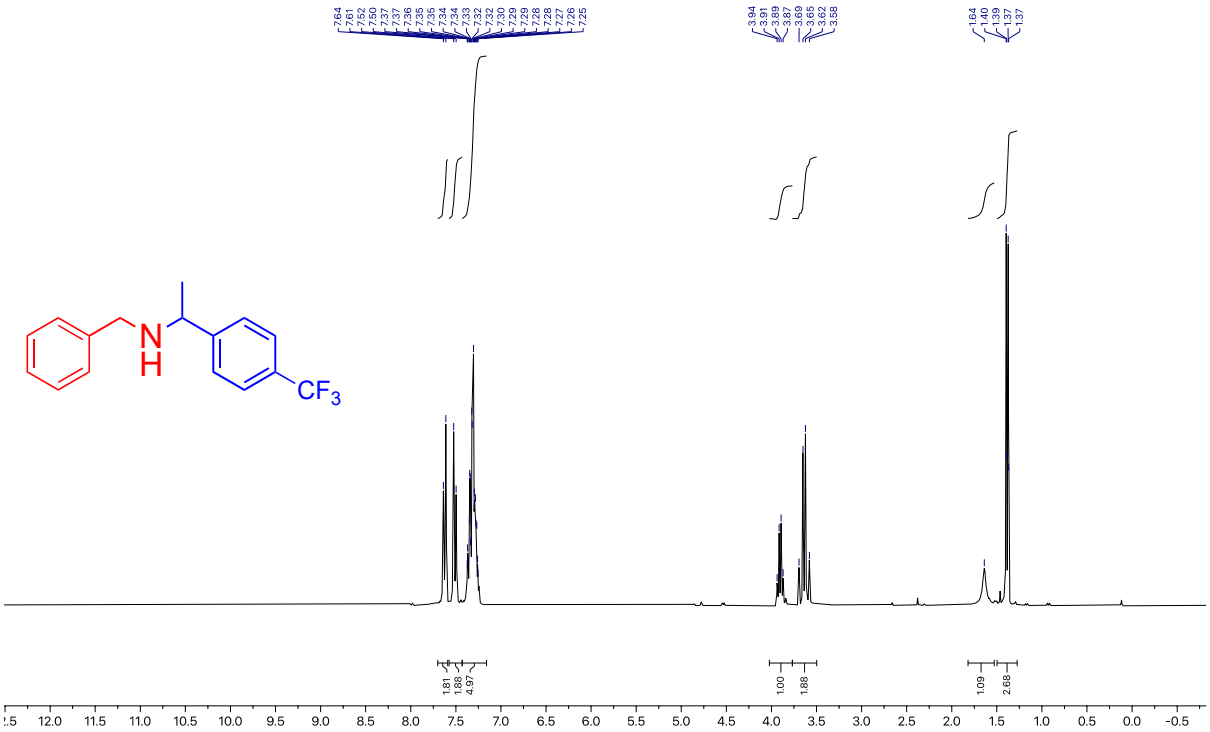
^{13}C NMR (75 MHz, Chloroform-*d)* of **7ba**



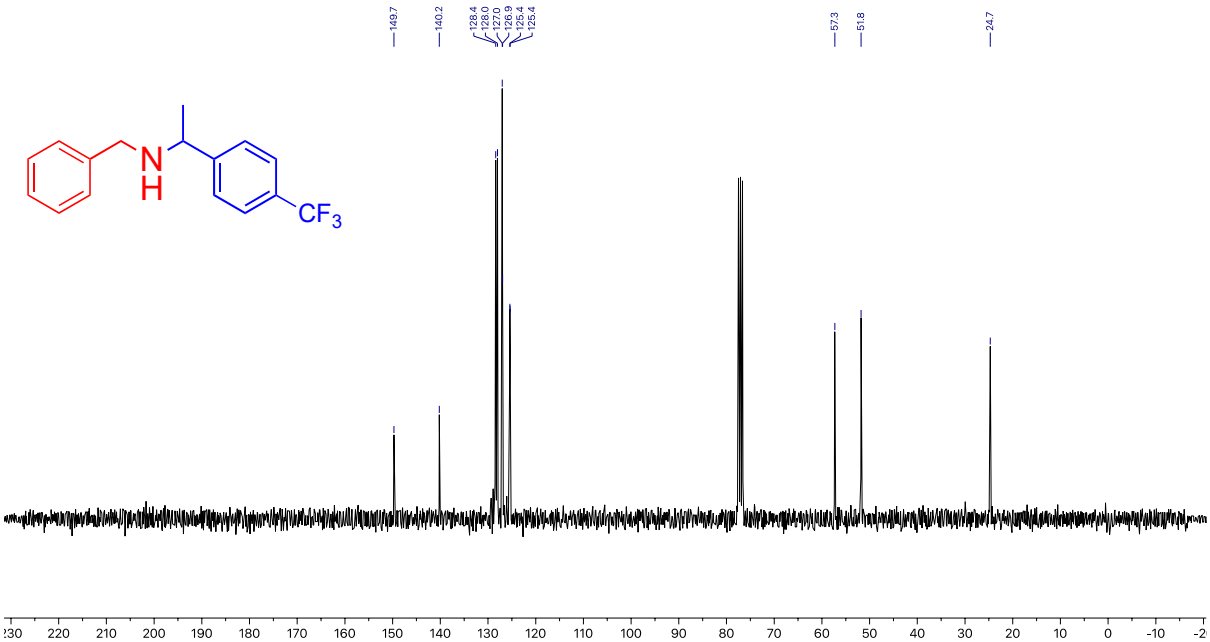
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ga**



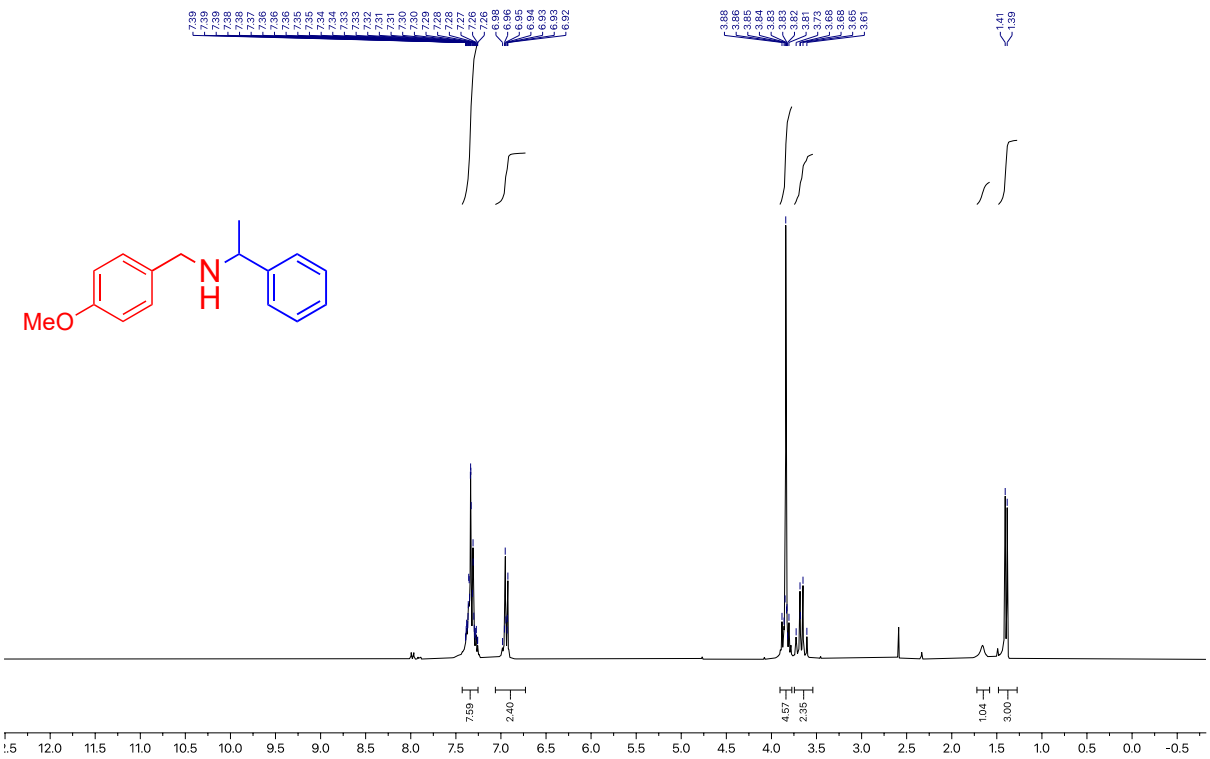
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ga**



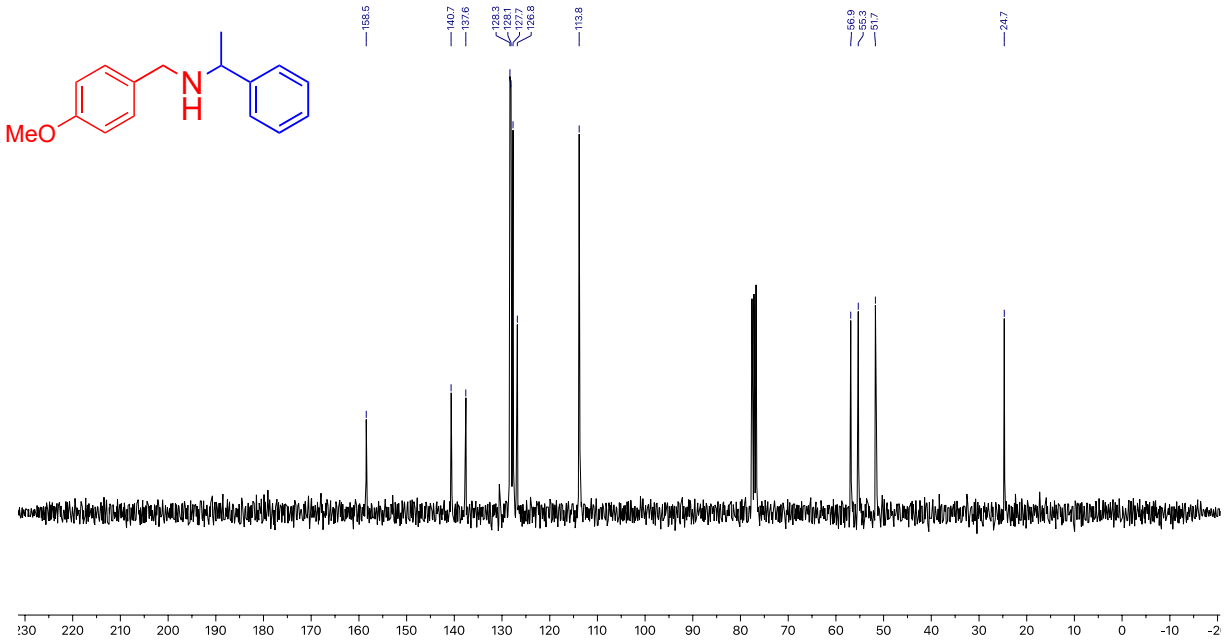
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ia**



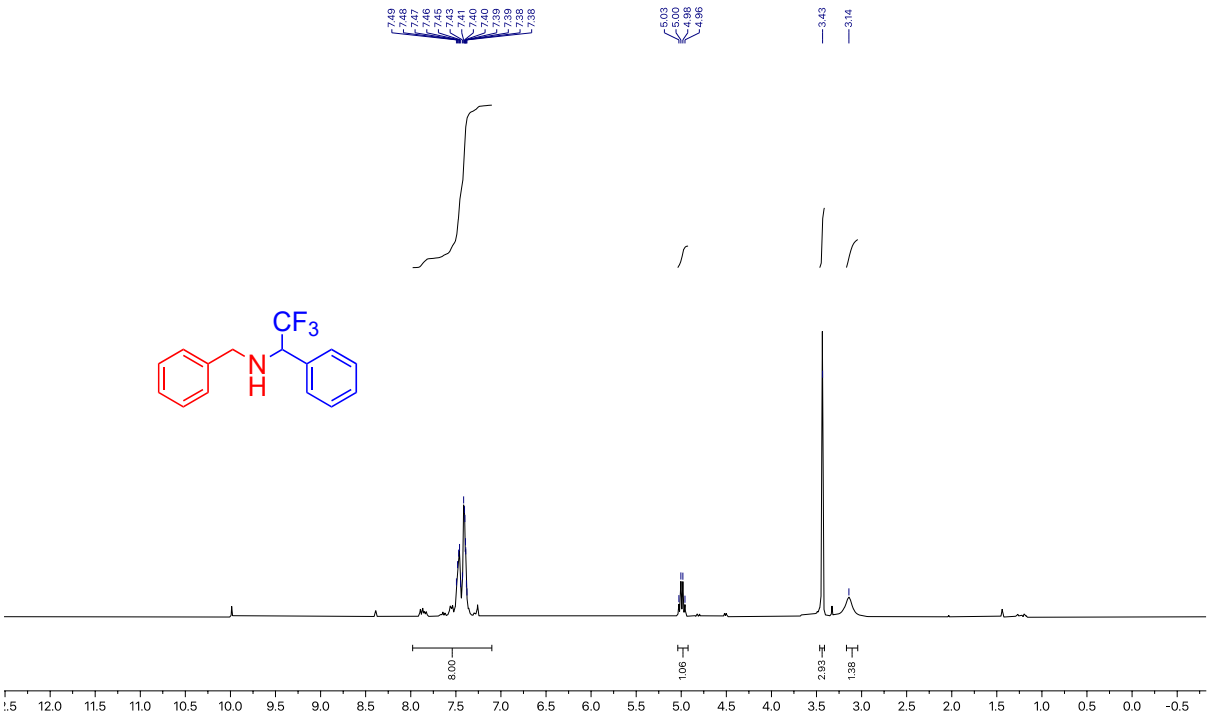
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ia**



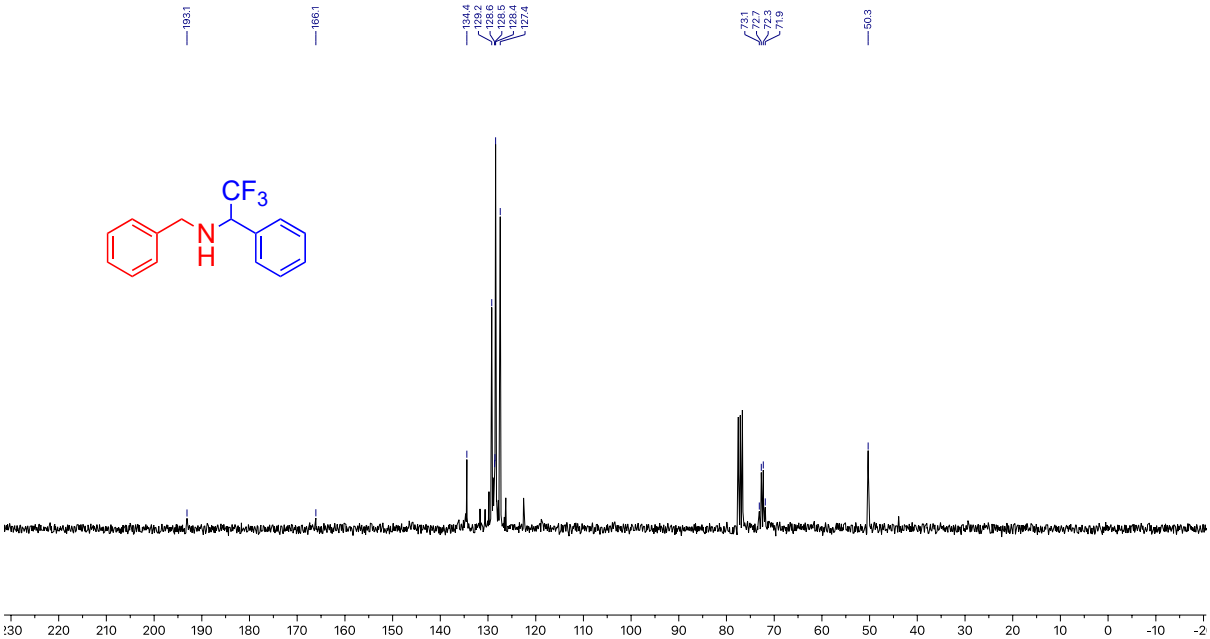
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ha**



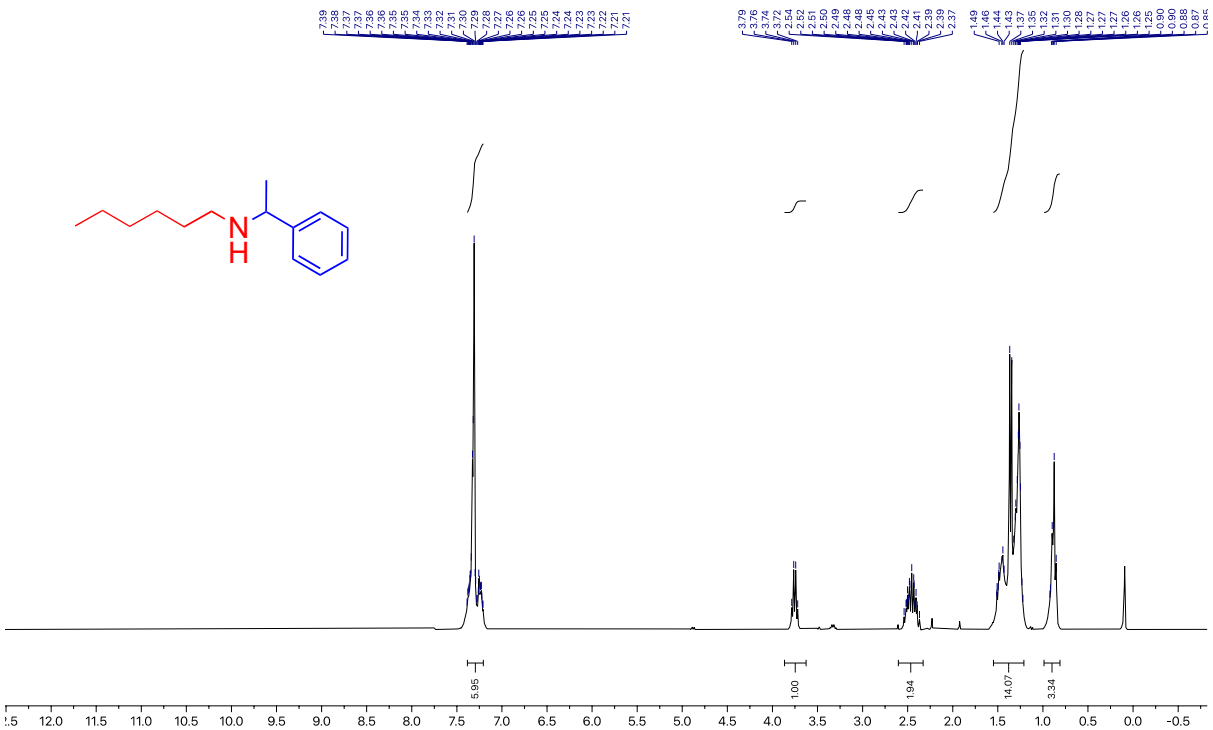
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ha**



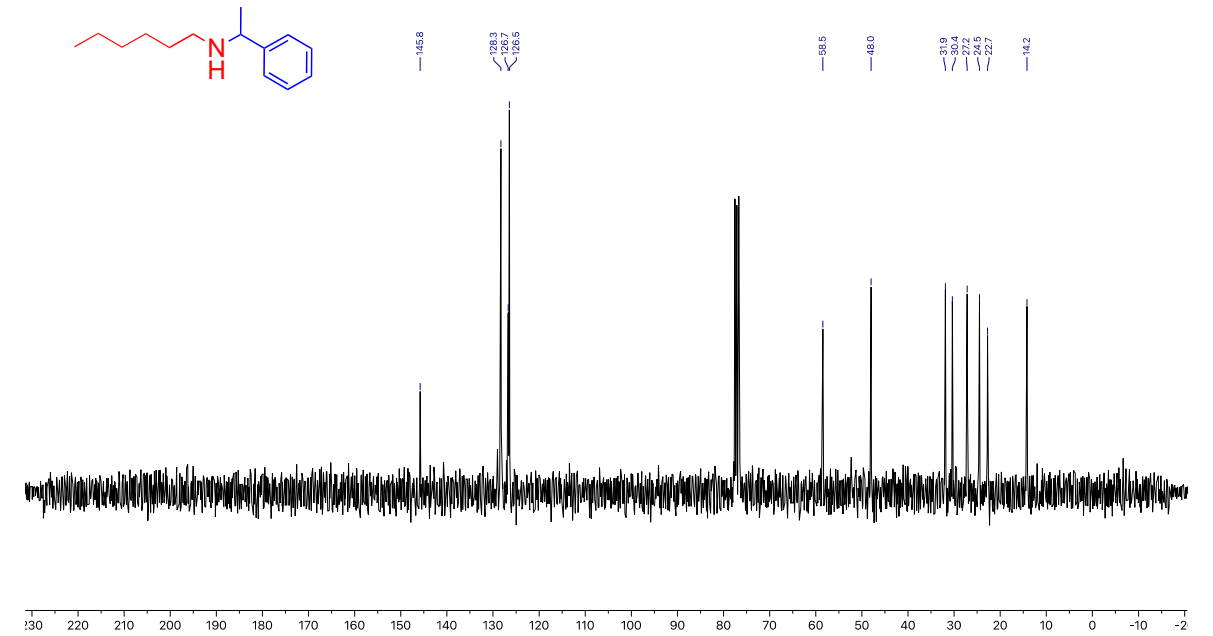
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ja**



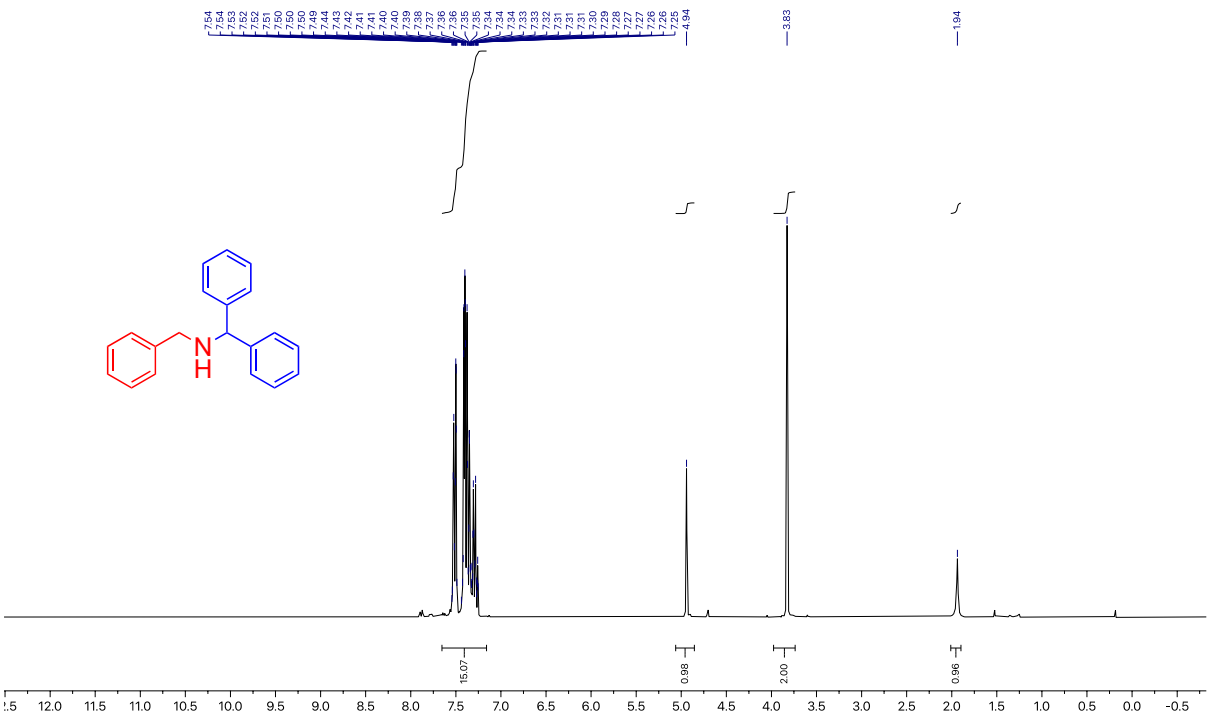
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ja**



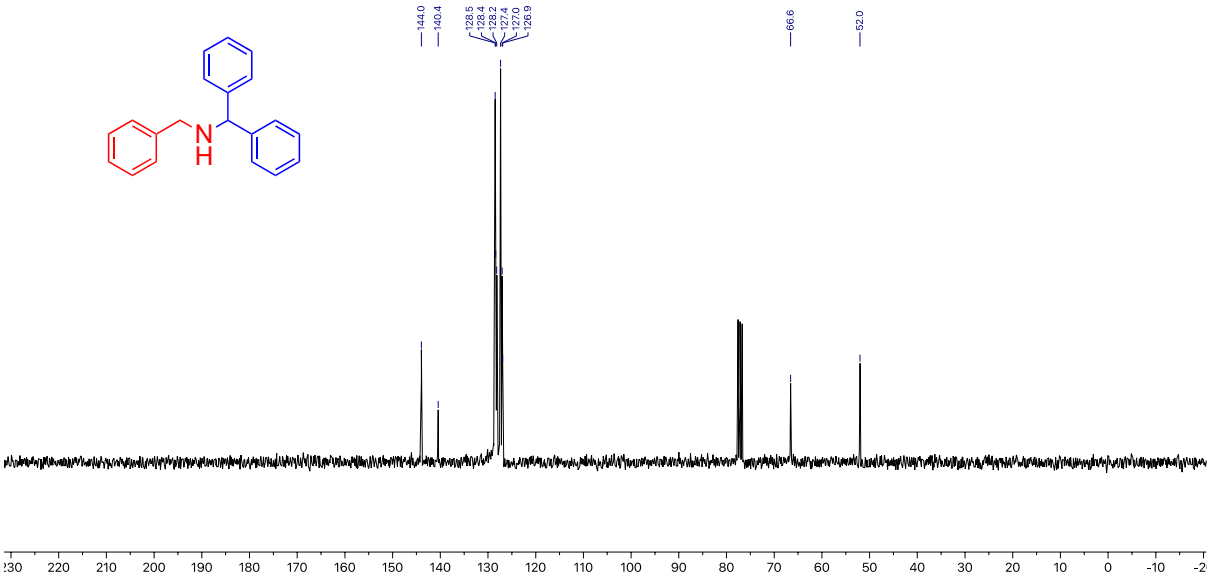
^{1}H NMR (300 MHz, Chloroform-*d)* of **6gh**



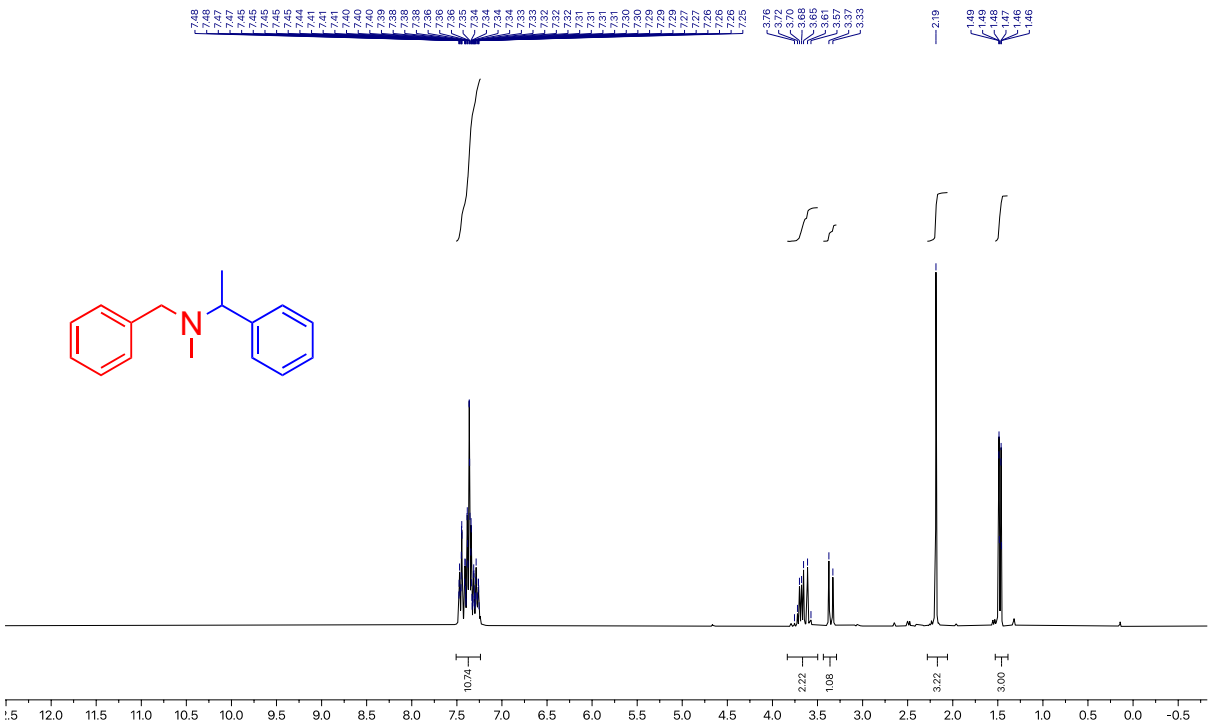
^{13}C NMR (75 MHz, Chloroform-*d)* of **6gh**



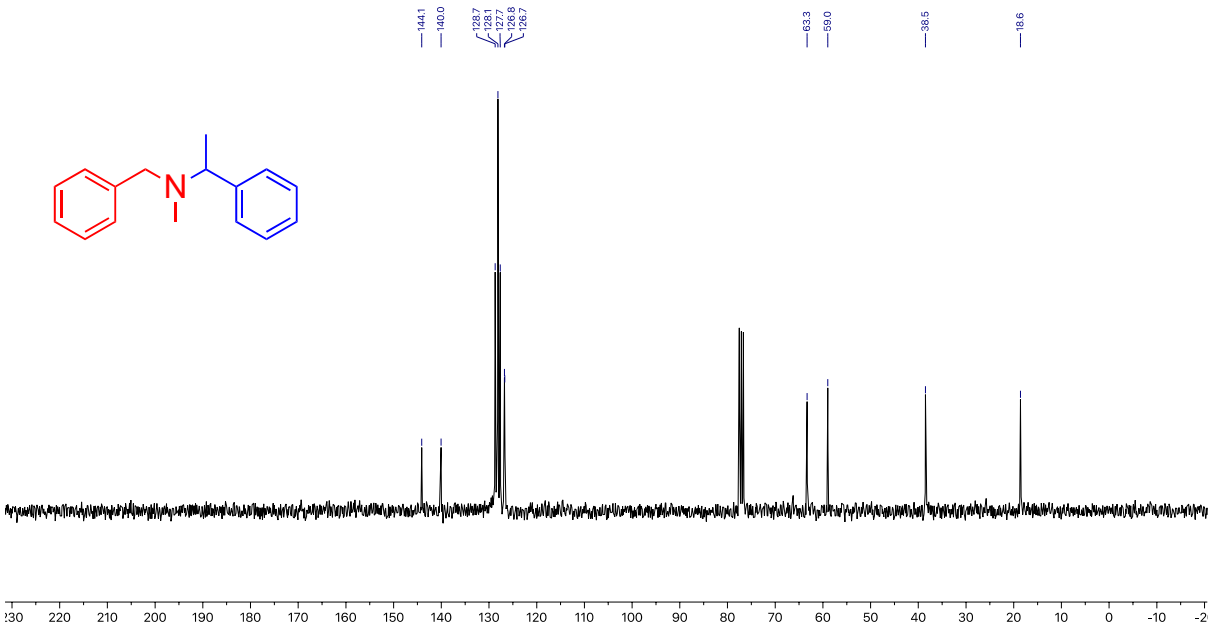
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ka**



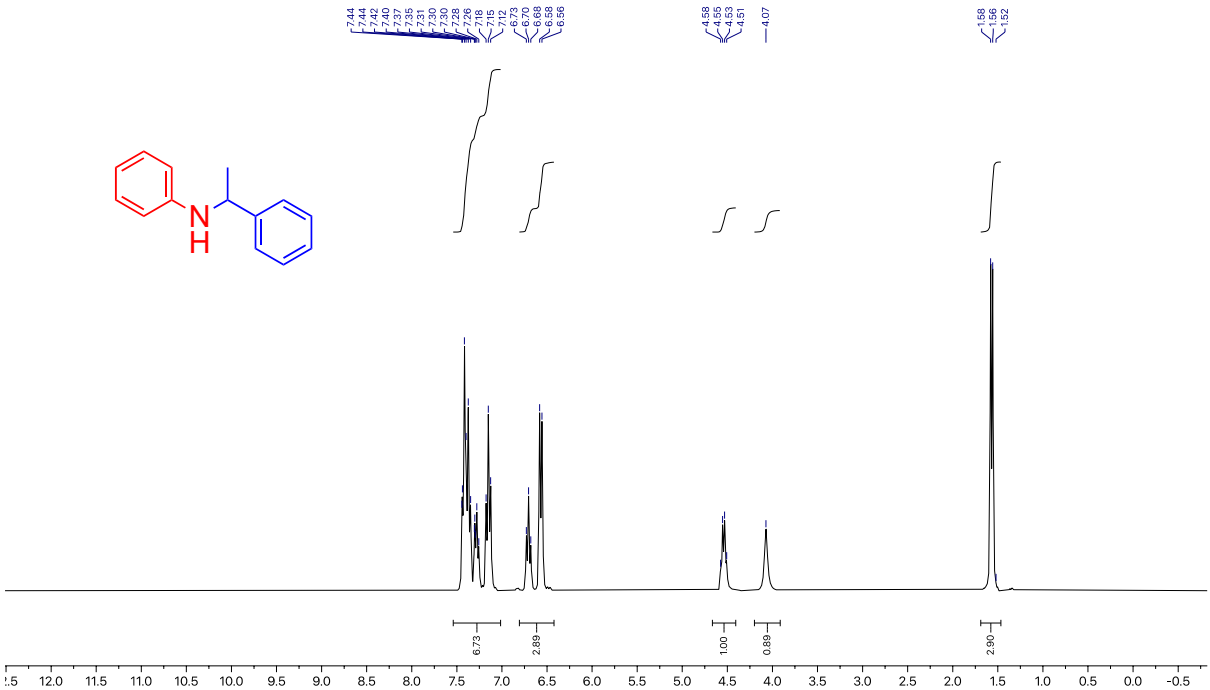
^{13}C NMR (75 MHz, Chloroform-*d)* of **6ka**



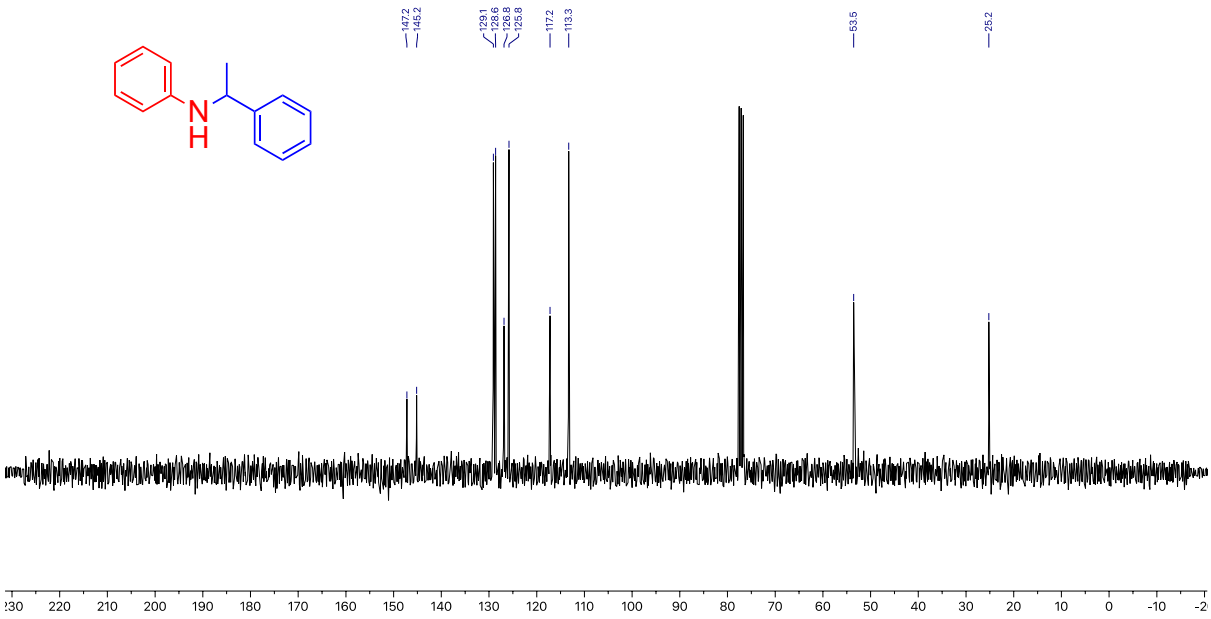
^{1}H NMR (300 MHz, Chloroform-*d)* of **6gl**



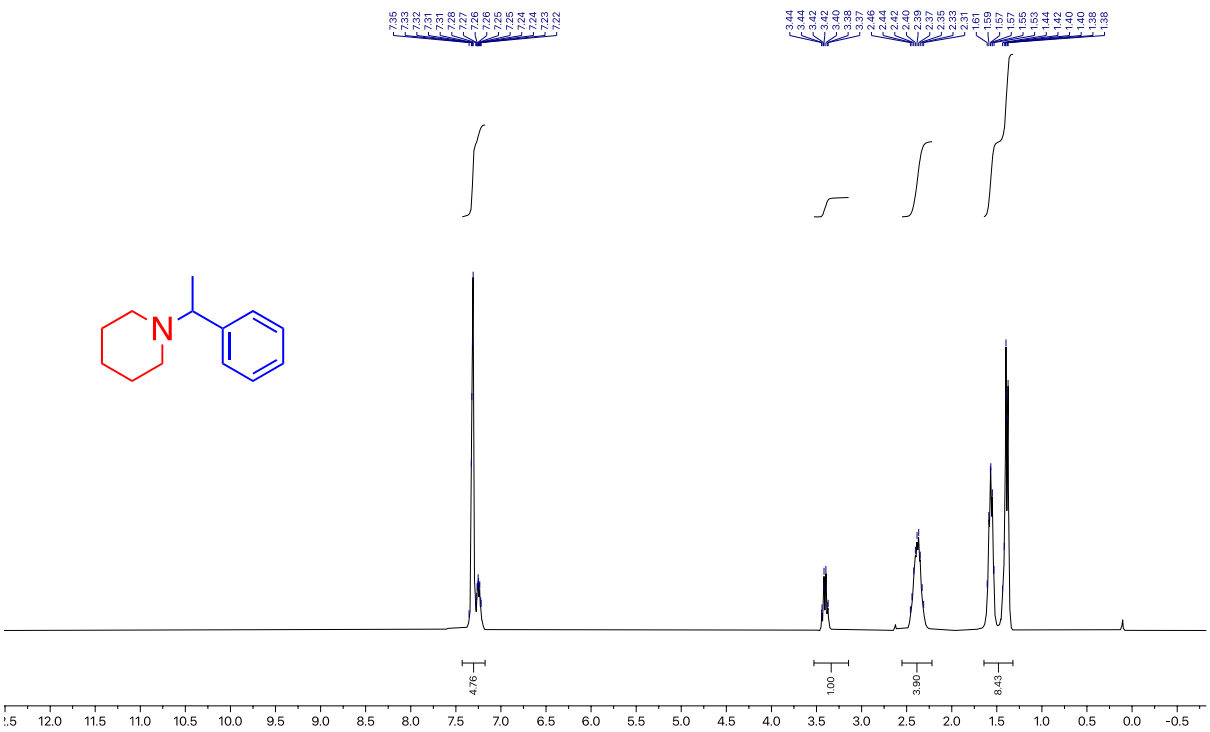
^{13}C NMR (75 MHz, Chloroform-*d)* of **6gl**



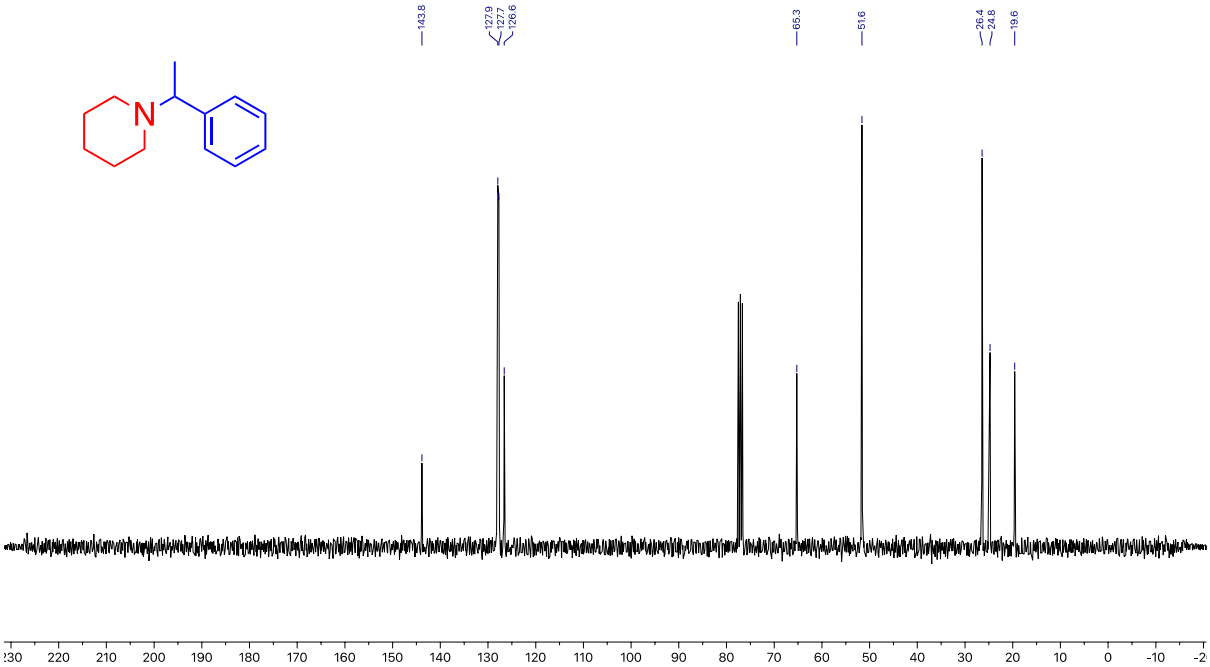
^{1}H NMR (300 MHz, Chloroform-*d)* of **6gm**



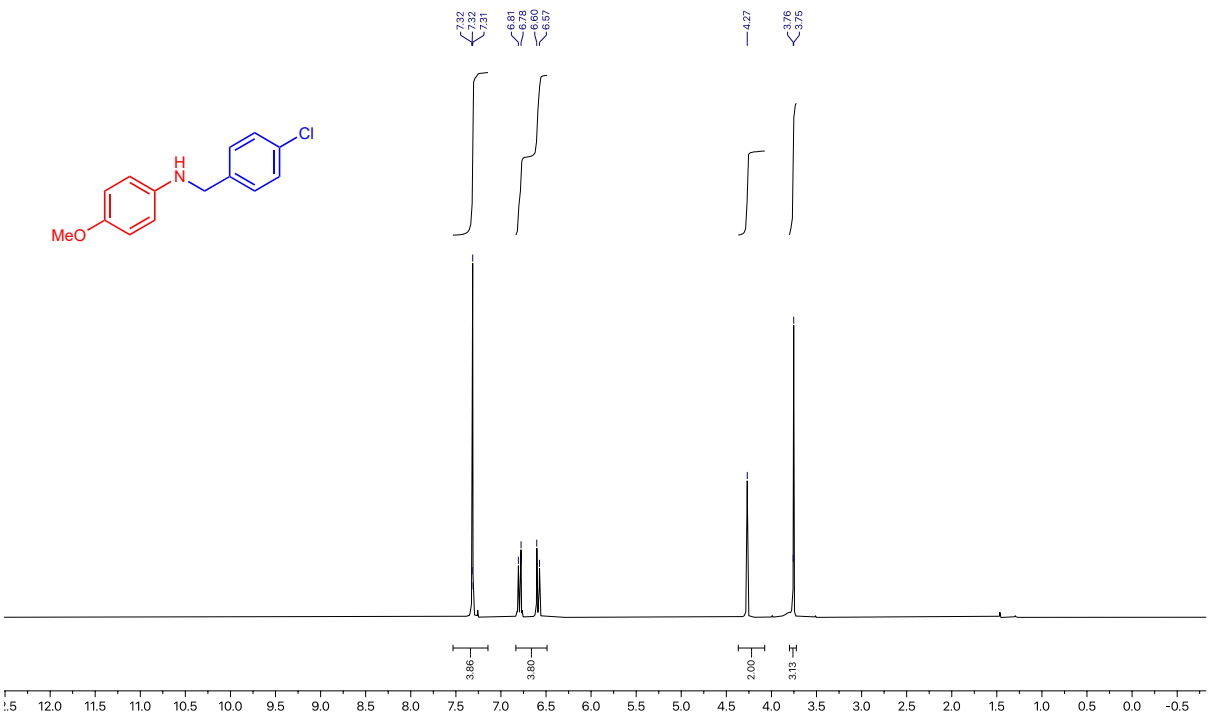
^{13}C NMR (75 MHz, Chloroform-*d)* of **6gm**



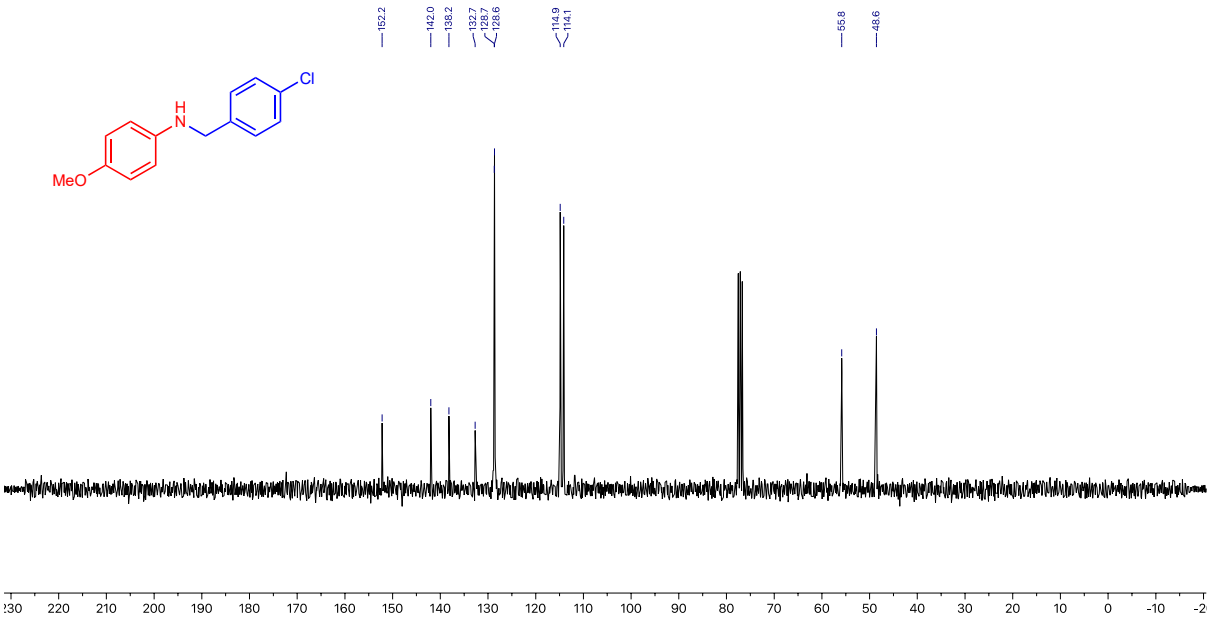
^{1}H NMR (300 MHz, Chloroform-*d)* of **6gk**



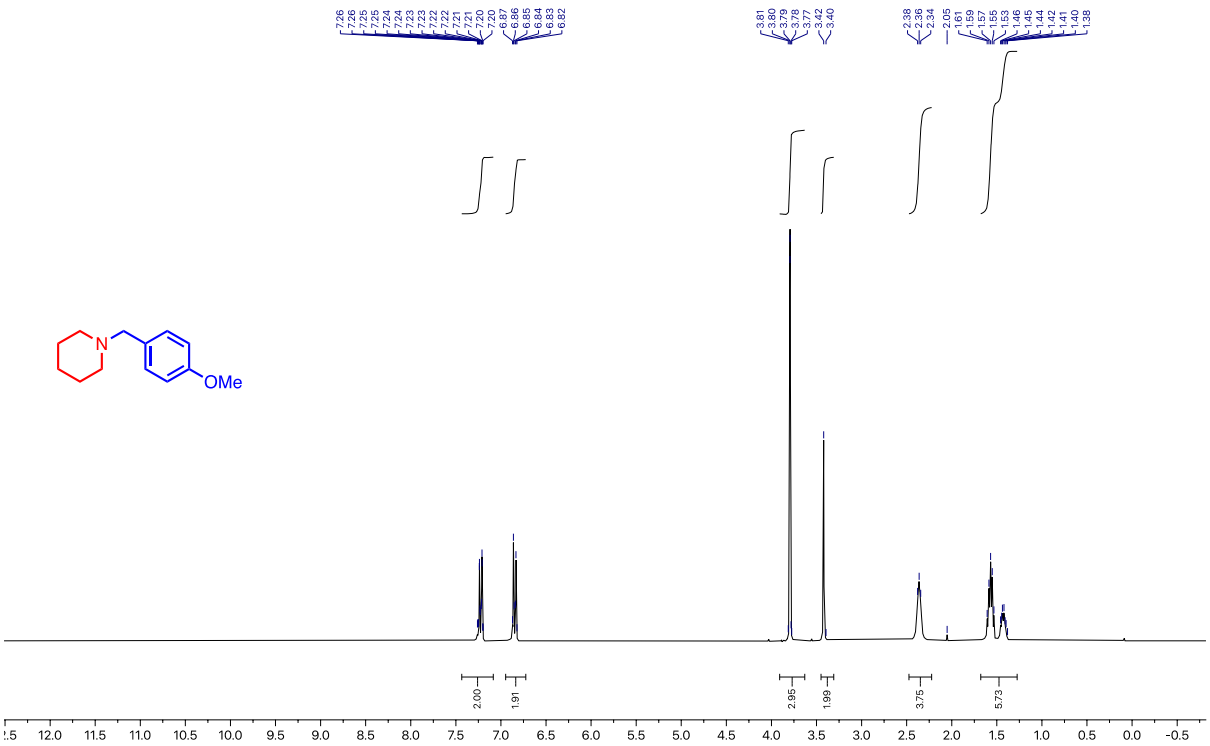
^{13}C NMR (75 MHz, Chloroform-*d)* of **6gk**



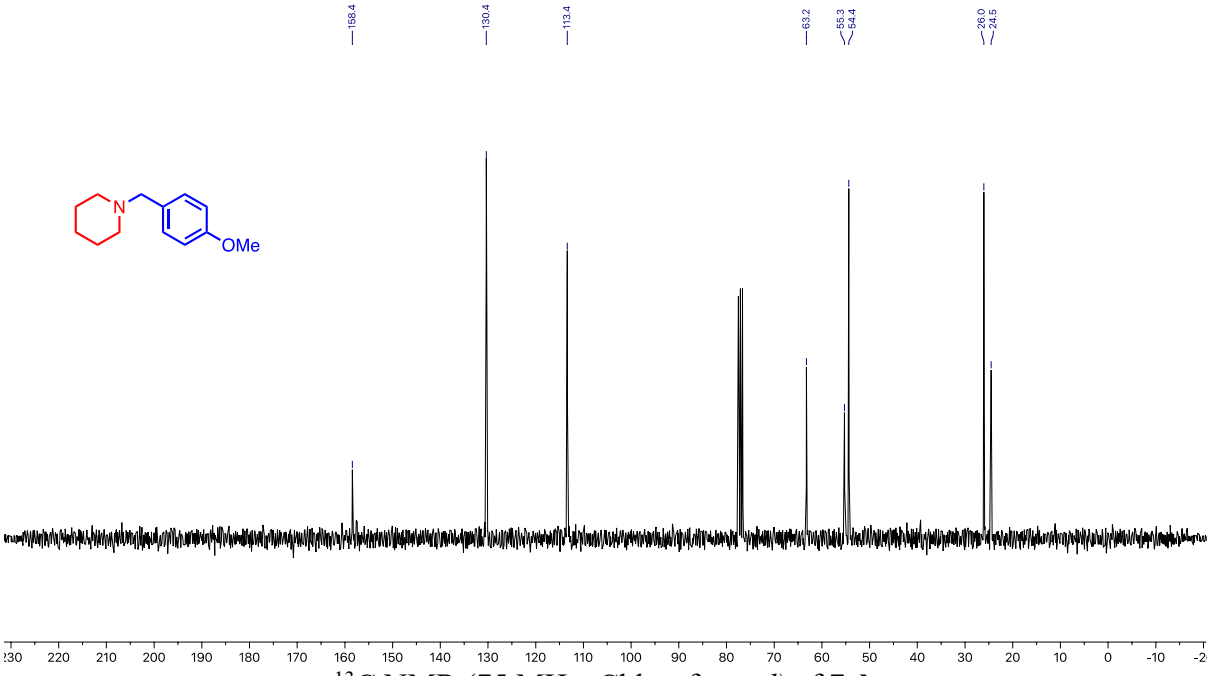
^{1}H NMR (300 MHz, Chloroform-*d)* of **7dq**



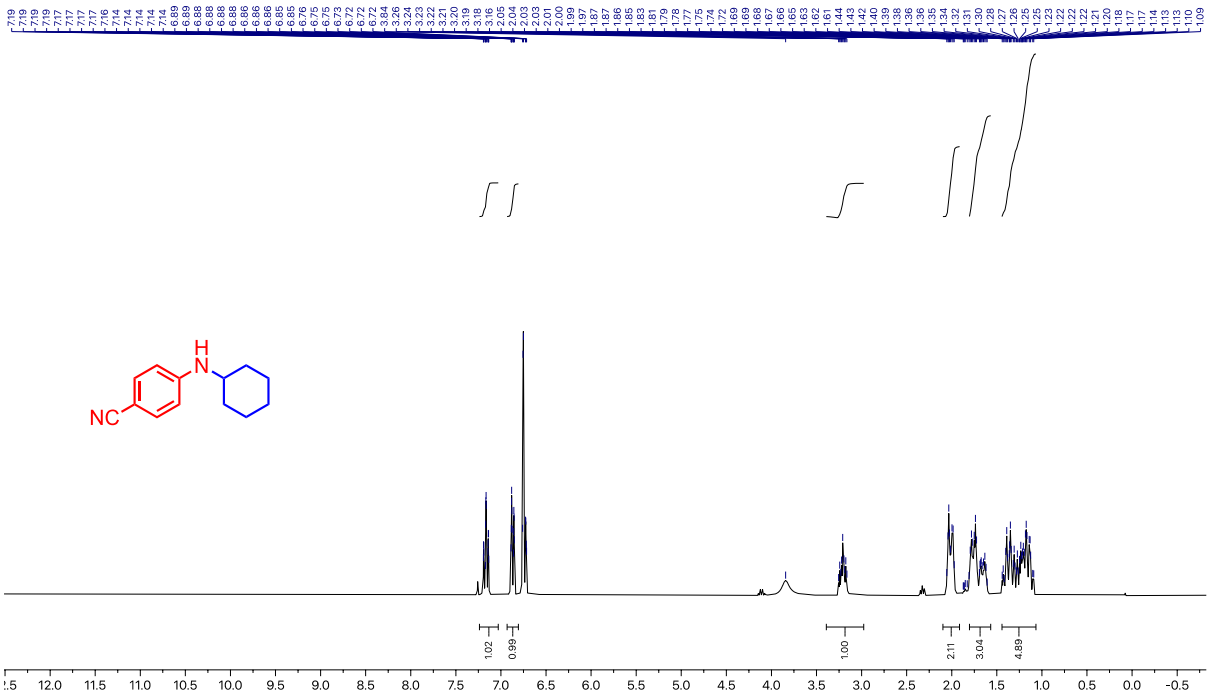
^{13}C NMR (75 MHz, Chloroform-*d)* of **7dq**



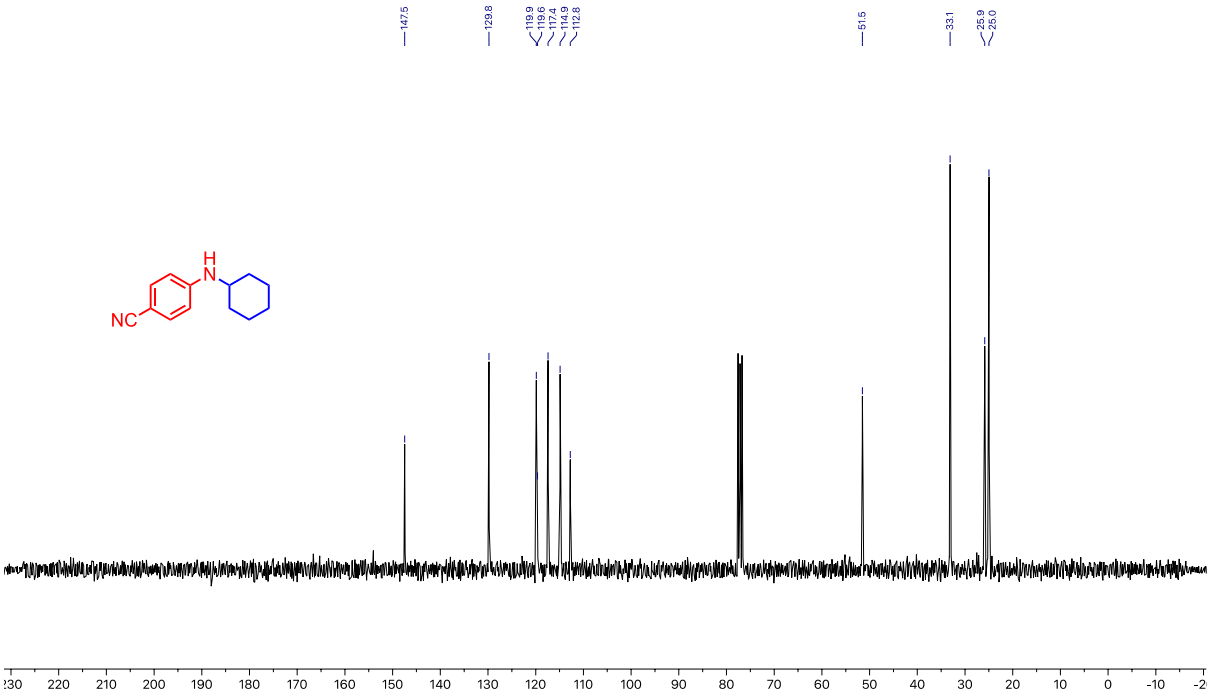
^{1}H NMR (300 MHz, Chloroform-*d)* of **7ek**



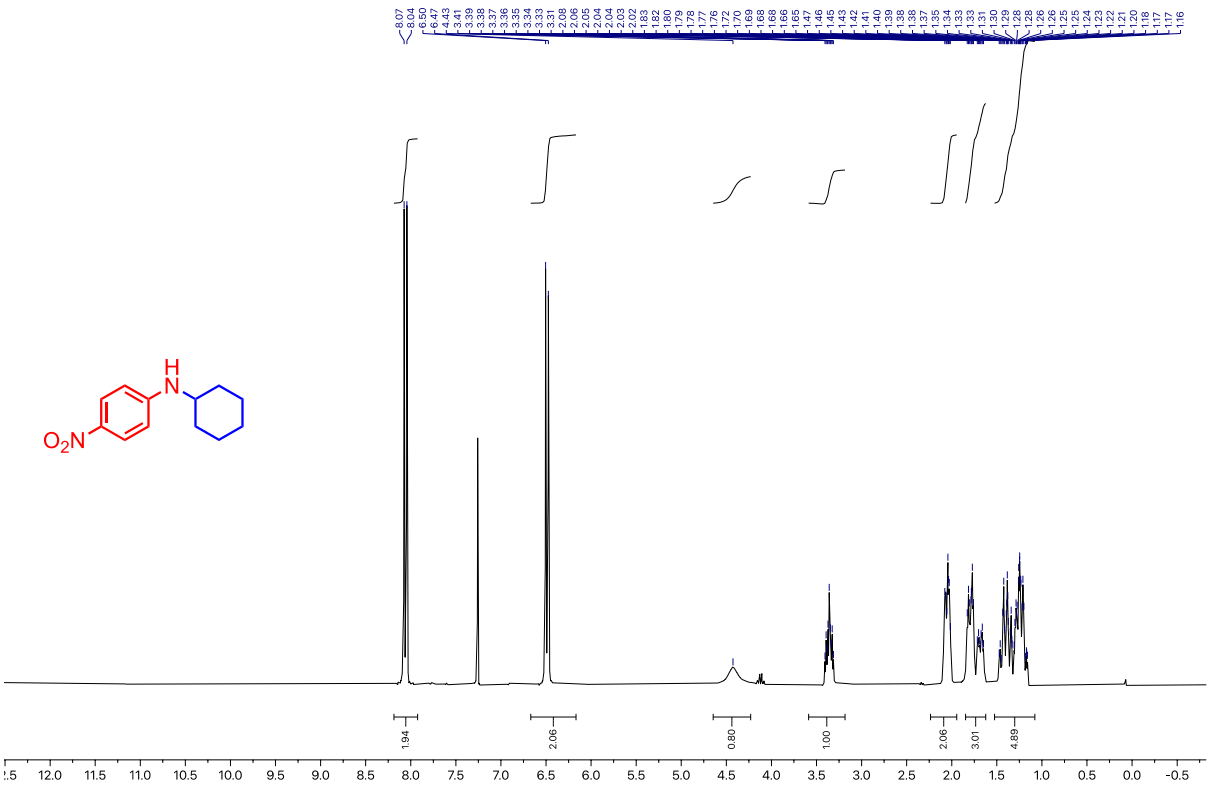
^{13}C NMR (75 MHz, Chloroform-*d)* of **7ek**



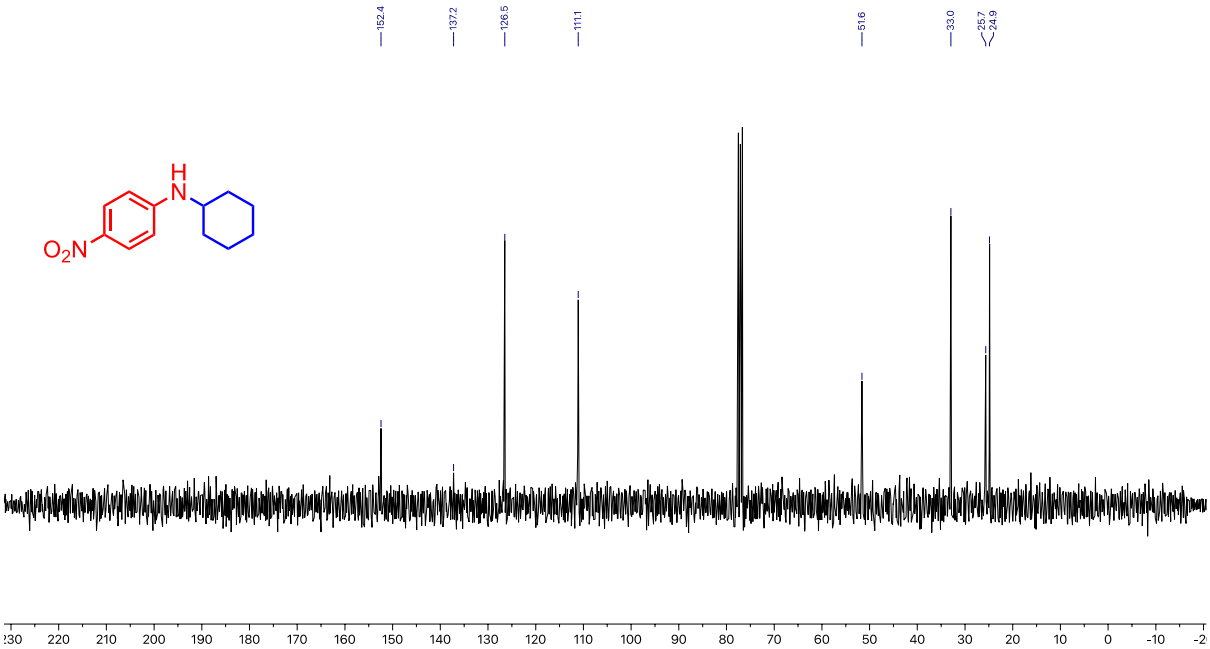
^{1}H NMR (300 MHz, Chloroform-*d)* of **6an**



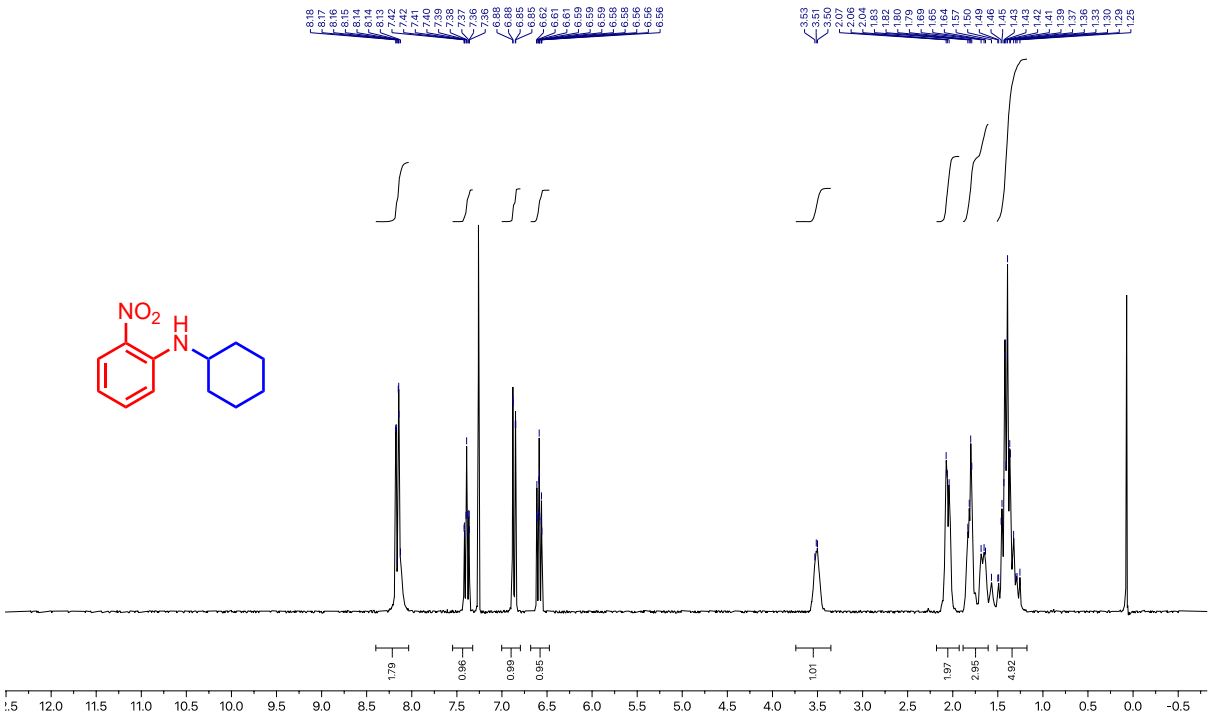
^{13}C NMR (75 MHz, Chloroform-*d)* of **6an**



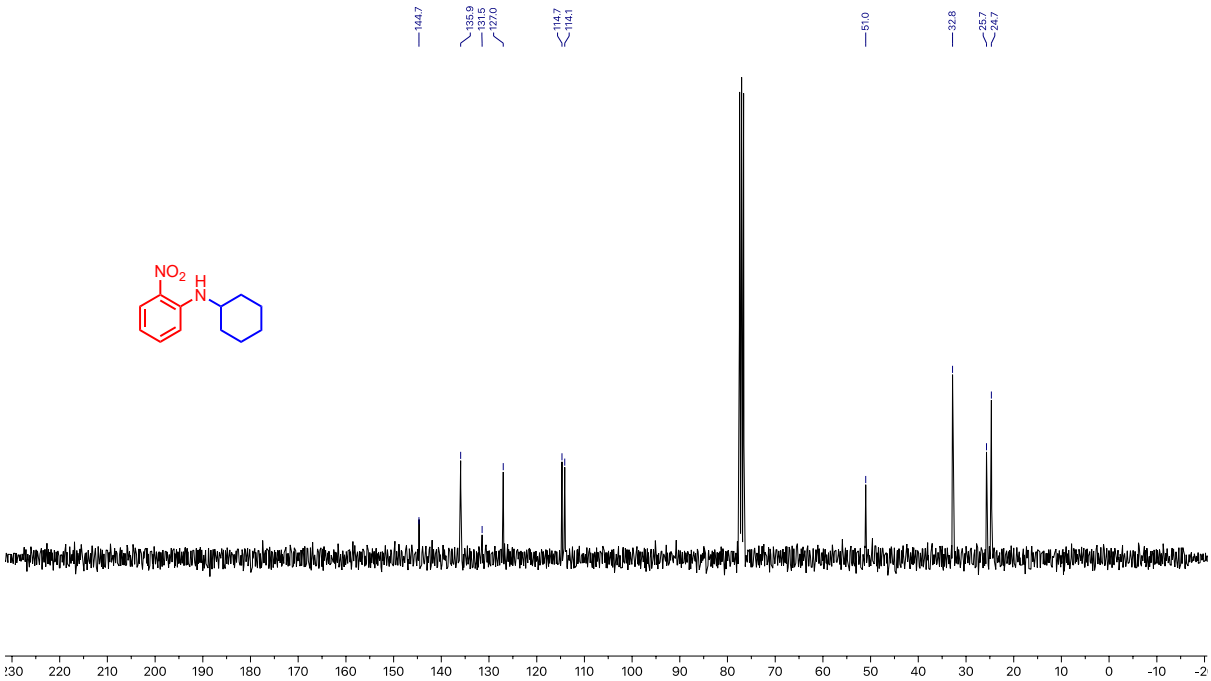
^{1}H NMR (300 MHz, Chloroform-*d)* of **6ao**



^{13}C NMR (75 MHz, Chloroform-*d)* of **6ao**



^{1}H NMR (300 MHz, Chloroform-*d)* of **6ap**



^{13}C NMR (75 MHz, Chloroform-*d)* of **6ap**