

## **A Copper Metal-Organic Hydrogel as a Heterogeneous and Reusable**

### **Catalyst for the Synthesis of $\beta$ -Amino Alcohols**

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## **Experimental**

### **Materials and physical measurements**

#### **<sup>1</sup>H and <sup>13</sup>C NMR spectrometry**

The <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded using a Bruker NMR spectrometer (<sup>1</sup>H frequency = 500 MHz, 600 MHz) in suitable deuterated solvents. The residual proton signal from the solvent was used as an internal standard.

#### **Mass spectrometry**

The high-resolution mass spectrometry (HRMS) was conducted on an Agilent TOF MS system using electrospray ionization in positive mode (ESI<sup>+</sup>). A methanol-water mixture served as the solvent.

#### **Field Emission Scanning Electron Microscope (FESEM)**

The FESEM images were recorded on a MERLIN microscope equipped with energy-dispersive X-ray spectroscopy (EDS, ZEISS EVO 60 with an Oxford detector) at an operating voltage of 5 kV. The sample was prepared by depositing a dilute aqueous dispersion of Cu-MOG onto thin aluminum sheets, followed by air or vacuum-drying overnight. To prevent sample charging, a thin gold coating was applied before imaging. FESEM images were acquired across various magnifications, with accelerating voltages varying from 5 to 16 kV, enabling a detailed structural and compositional assessment.

#### **Atomic Force Microscope (AFM)**

The atomic force microscopic images were recorded on Agilent 5500 microscope in tapping mode. For sample preparation, a highly diluted aqueous dispersion of the Cu-MOG was carefully deposited onto a glass slide and left to dry overnight, ensuring optimal surface adherence before imaging.

#### **Transmission Electron Microscopy (TEM)**

The TEM images were recorded using a FEG high-resolution TEM (JEM-2100F) and an analytical TEM (FEI TECNAI G220S-TWIN), both operating at 200 kV. The sample was

prepared by carefully depositing a highly diluted Cu-MOG solution onto a carbon-coated copper grid, followed by air-drying overnight to ensure optimal imaging conditions. Further structural examination was carried out using an Agilent 5500 microscope, providing additional nanoscale insights.

### **Rheological Measurements**

Rheological experiments were performed using an MCR 102 rheometer (Anton Paar) to gain detailed insights into the viscoelastic behavior of the gel. The instrument operated in steady shear mode for oscillatory testing, employing a modular compact rheometer equipped with a 25 mm diameter parallel plate and a fixed tool gap of 0.5 mm. To explore the gel's structural integrity, a stress amplitude sweep experiment was conducted at a constant angular frequency of 1 rad/s at 298 K. Additionally, a frequency sweep measurement was carried out across a broad range (0.1 to 100 rad/s) with a fixed strain of 0.1%, ensuring the analysis remained within the linear viscoelastic region. The flow behavior of the gel was further examined through an amplitude sweep experiment, applying shear stress from 0.1 to 10 Pa. All measurements were performed using freshly prepared gel samples, ensuring the accuracy and reproducibility of the rheological characterization.

### **MALDI-TOF Mass Spectrometry**

The MALDI-TOF spectrum of the MOG was acquired using a Bruker Daltonics Ultra Extreme apparatus, operating in positive ion mode with a pulsed nitrogen laser ( $\lambda = 337$  nm). The laser intensity was set to 50%, and each spectrum was generated by accumulating 100 laser shots over an acquisition range of  $m/z$  100 to  $m/z$  600. A finely attenuated metallogel sample was meticulously blended with a 2,5-dihydroxybenzoic acid matrix to enhance ionization efficiency and allowed to dry, ensuring optimal crystallization for high-resolution spectral analysis.

## **XPS**

The surface chemical composition of the materials was investigated using a K- $\alpha$  X-ray photoemission spectrometer (PHI 5000 VERSA PROBE III). The survey spectra were recorded with scans conducted at 1 eV intervals for precise characterization. To ensure accurate calibration, all binding energies in the X-ray photoelectron spectroscopy (XPS) spectra were referenced to the C 1s peak of adventitious hydrocarbons, set at 284.6 eV, enabling reliable spectral interpretation.

## **FTIR**

The infrared spectra of the dried gels were recorded on a Fourier Transform Infrared Spectrometer (NICOLET 6700) in the 4000 to 400  $\text{cm}^{-1}$ , with a resolution of 4  $\text{cm}^{-1}$ . For optimal spectral acquisition, 2 mg of the dried gel was finely ground and blended with KBr, and carefully pressed into a pellet, maximizing transparency and analytical accuracy.

## **Synthesis of {(2-hydroxy-naphthalen-1-yl)methyl}glycine (H3L)**

Glycine (0.525 g, 7 mmol) was dissolved in 10 mL of water, and aqueous formaldehyde (1 mL, 37%) was added to it and was stirred at room temperature (RT) for 30 minutes. To this, an ethanolic solution of  $\beta$ -naphthol (1.009 g, 7 mmol in 30 mL of ethanol) was added dropwise. The reaction solution was stirred for 8 hours, whereupon a fine white solid precipitated. The precipitate was filtered, washed with hot distilled water (3 times), ethanol (3 times), and finally, with hexane. Yield: 95% yield. IR ( $\text{cm}^{-1}$ ): 3370 to 2980 (broad), 1575. HRMS:  $m/z = 232.0978$  [M ( $\text{C}_{13}\text{H}_{13}\text{NO}_3 + \text{H}^+$ )] {Calculated for  $\text{C}_{13}\text{H}_{14}\text{NO}_3 = 232.0973$ }.  $^1\text{H}$  NMR (500 MHz,  $\text{D}_2\text{O}$ )  $\delta$  3.20 (s, 2H), 4.01 (s, 2H), 6.96 (d,  $J = 8.9$  Hz, 1H), 7.09 (t,  $J = 7.1$ , 1H), 7.36 (t,  $J = 7.2$  Hz, 1H), 7.56 (d,  $J = 9.0$  Hz, 1H), 7.64 (d,  $J = 8.8$  Hz, 1H), 7.82 (d,  $J = 8.7$  Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{D}_2\text{O}$ )  $\delta$  42.6, 52.0, 116.4, 120.3, 121.5, 124.4, 125.8, 126.2, 128.4, 128.8, 134.5, 163.7, 180.0

### **Preparation of the Cu-MOG**

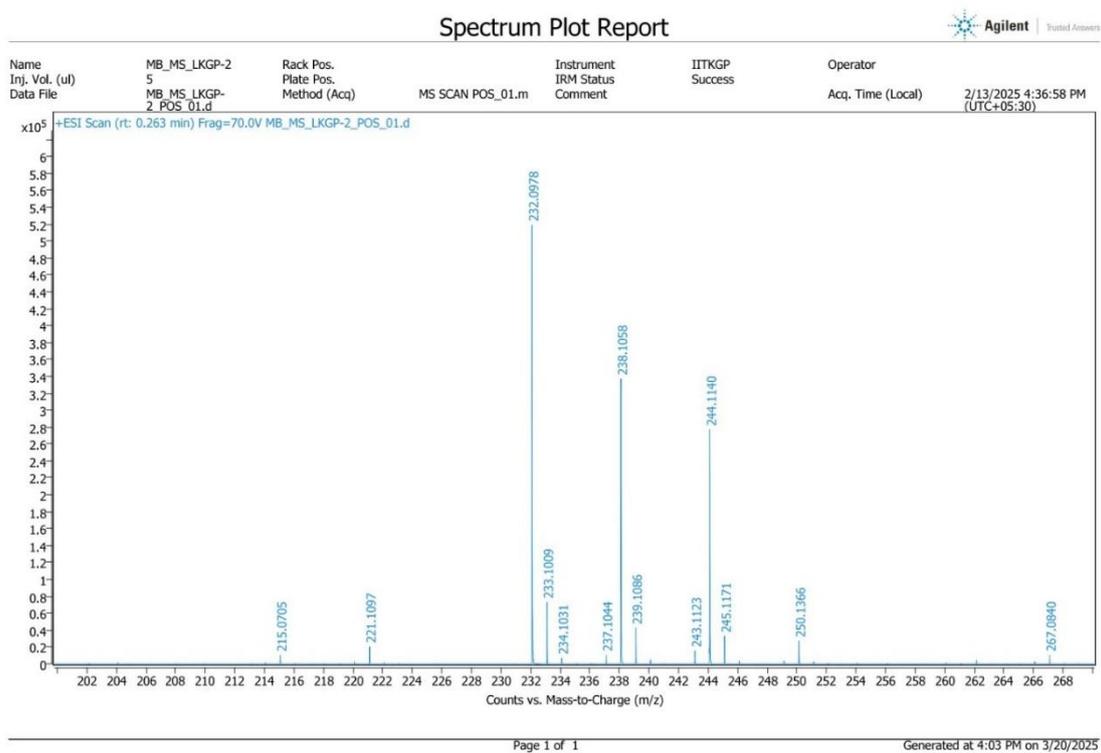
The ligand, **H<sub>3</sub>L** (0.011 g, 0.05 mmol), was suspended in water (0.5 mL) in a glass vial. To this was added LiOH (0.002 g; 0.10 mmol), and a clear solution of the dilithium salt of the ligand (**Li<sub>2</sub>HL**) was obtained. To this was added an aqueous solution (0.5 mL) of Cu(CH<sub>3</sub>COO)<sub>2</sub>·H<sub>2</sub>O (0.01 g, 0.05 mmol). The Cu-MOG formed instantaneously as shown by the inverted vial method. The hydrogel was dried under vacuum to get the xerogel.

### **General Procedure for the Cu-MOG Catalyzed Synthesis of β-Amino Alcohols for the Reaction of Epoxides with Amines**

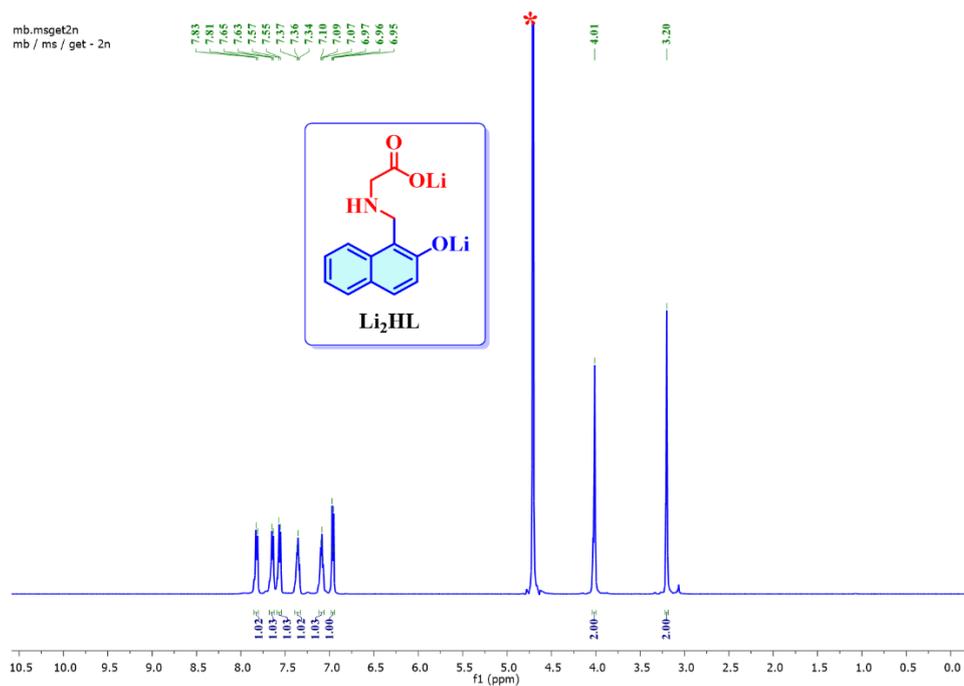
In a typical procedure, an oven-dried reaction tube or round-bottom flask equipped with a magnetic stirring bar was charged with epoxide (2 mmol) and amine (2 mmol). The Cu-MOG in xerogel form (0.002 g) was then added to the mixture. The reaction mixture was stirred at room temperature for 3.5 hours. The reaction was monitored periodically by thin-layer chromatography (TLC) and gas chromatography (GC). Upon completion of the reaction, dichloromethane (15 mL) was added to the reaction mixture. The catalyst was recovered by centrifugation, the organic layer was filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography using hexane/ethyl acetate as eluent, and the β-amino alcohol was isolated in pure form.

For catalyst recycling, the separated Cu-MOG xerogel was washed with a 1:1 mixture of water and acetone, followed by diethyl ether to remove residual organic contaminants. The recovered material was dried under vacuum overnight or in an oven at a controlled temperature for 3 hours. The regenerated catalyst was reused in subsequent reaction cycles with fresh substrates (e.g., cyclohexene oxide and aniline). All the products were characterized by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy.

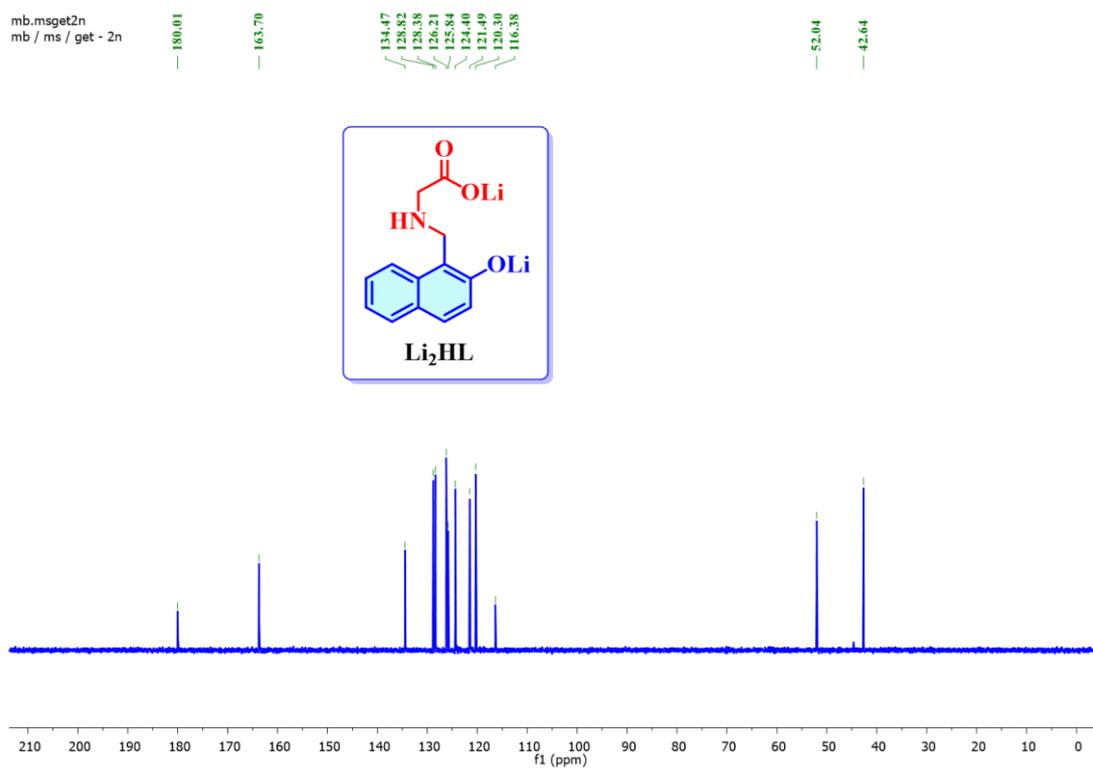
# Figures



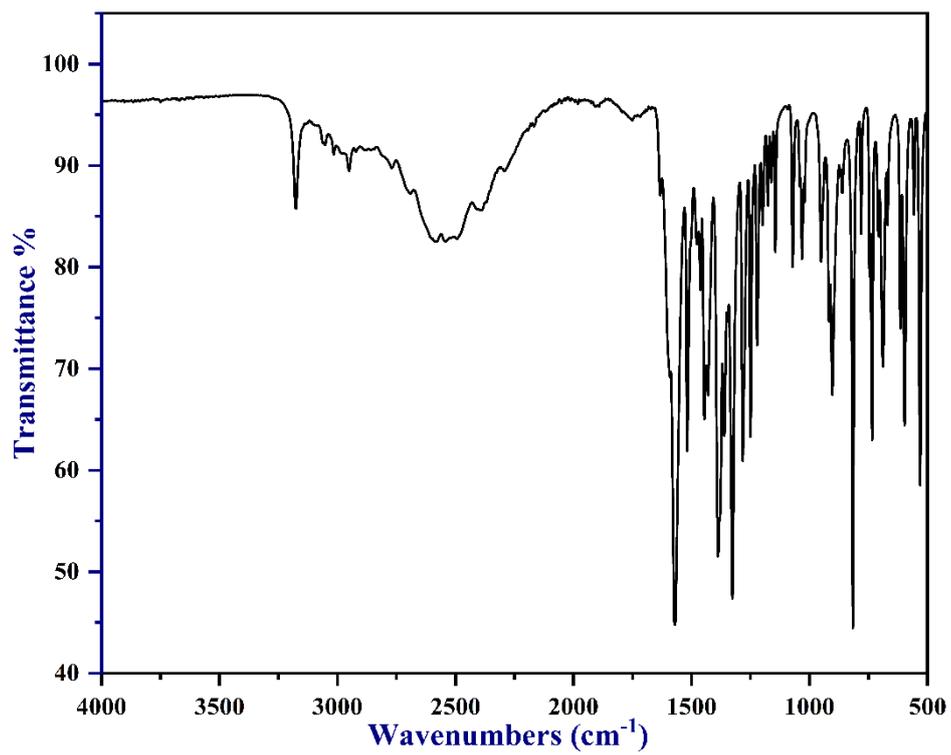
**Figure S1** HRMS spectrum of the dilithium form of gelator **H<sub>3</sub>L** in H<sub>2</sub>O



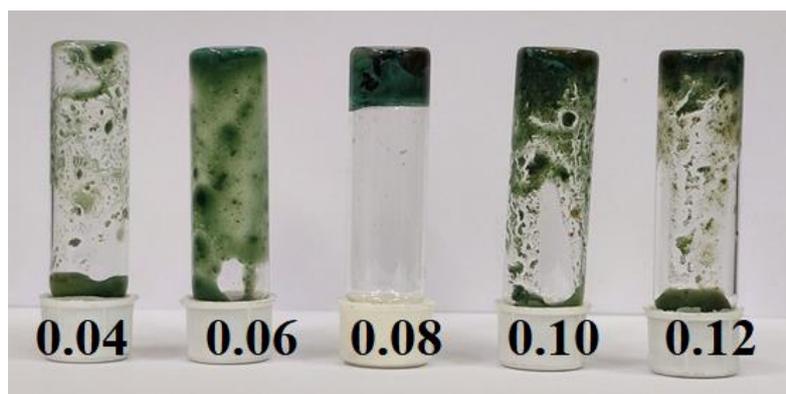
**Figure S2**  $^1\text{H}$  spectrum of the dilithium form of gelator **H<sub>3</sub>L** in  $\text{D}_2\text{O}$



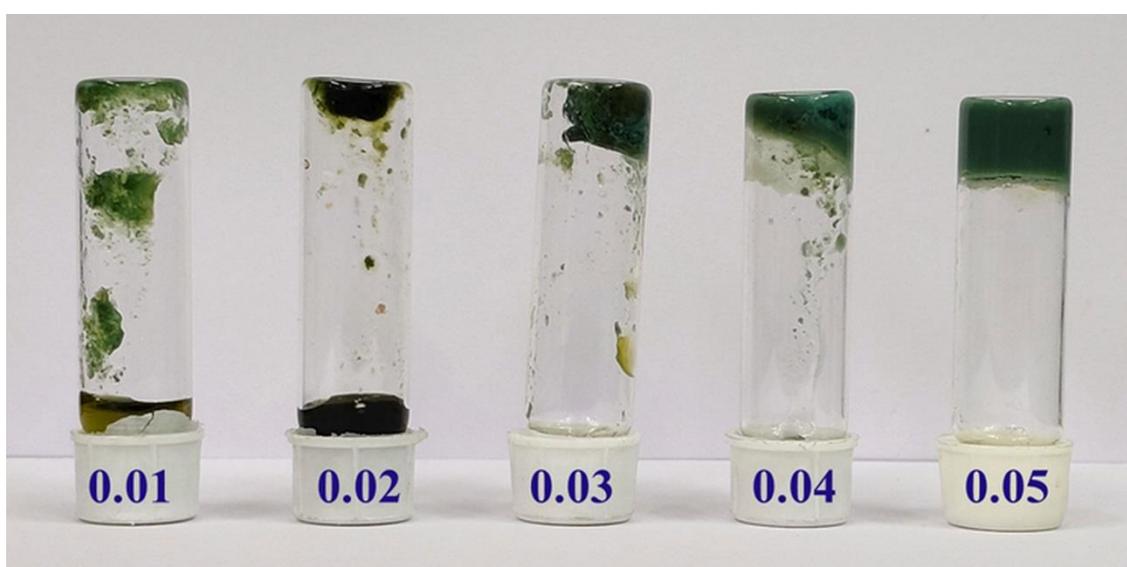
**Figure S3**  $^{13}\text{C}$  spectrum of the dilithium form of gelator **H<sub>3</sub>L** in  $\text{D}_2\text{O}$



**Figure S4** FTIR spectrum of gelator **H<sub>3</sub>L**

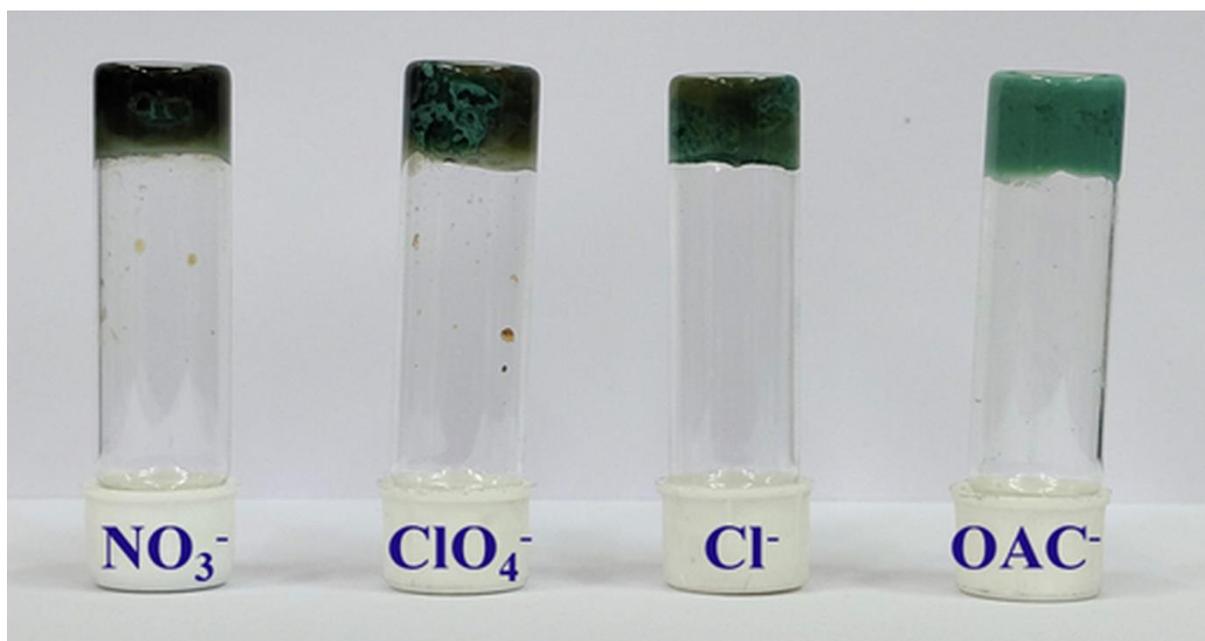


(a)

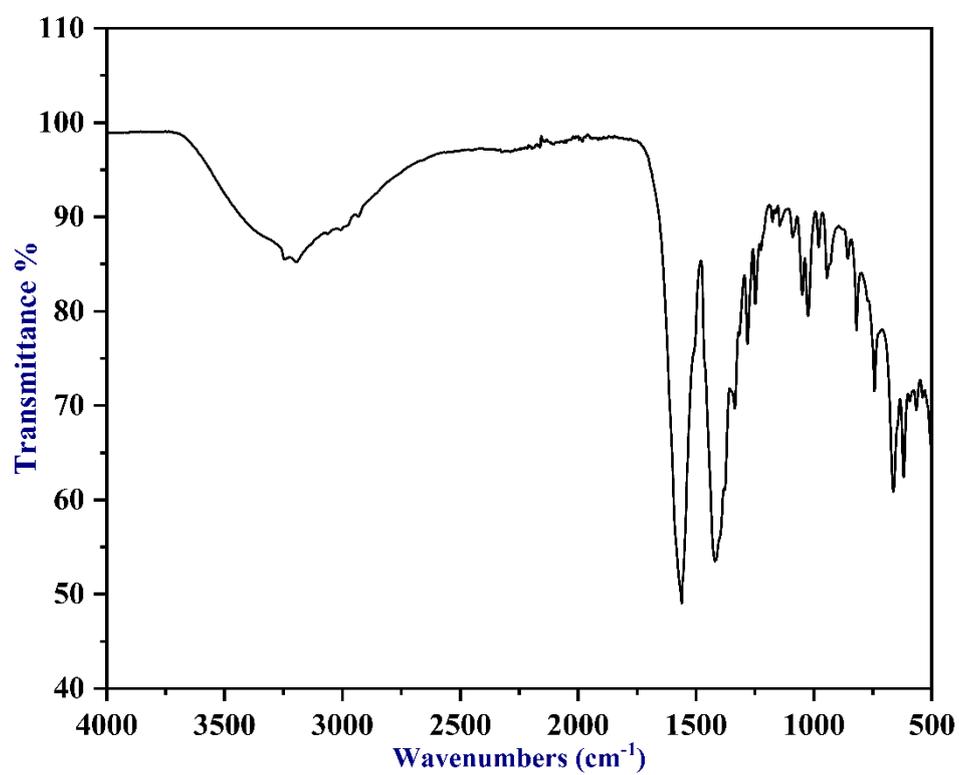


(b)

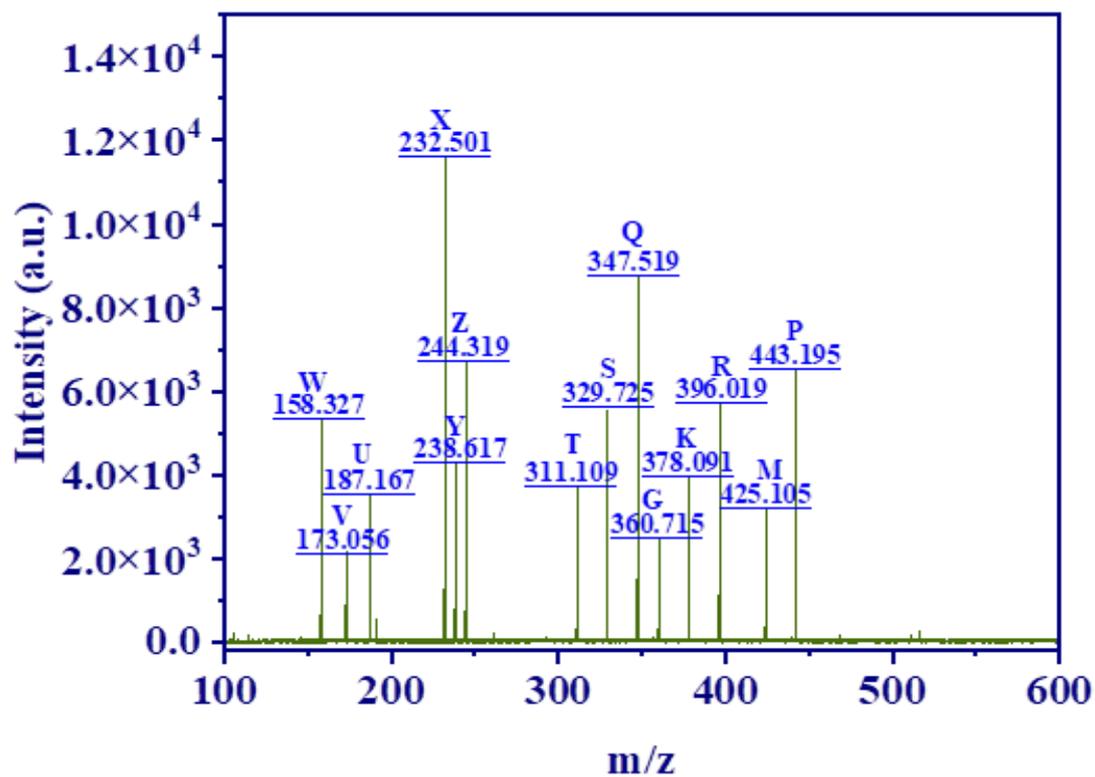
**Figure S5** Optical images of (a) Gel formation by mixing  $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$  (concentration varies from 0.04 to 0.12 equivalent with respect to  $\text{Li}_2\text{HL}$ ). Stable Cu-MOG was obtained when the concentration of  $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$  was 0.08 (from left to right). (b) Stable Cu-MOG formation at  $\text{MGC} = 21.50 \text{ mg. mL}^{-1}$ , as confirmed by the inverted vial method



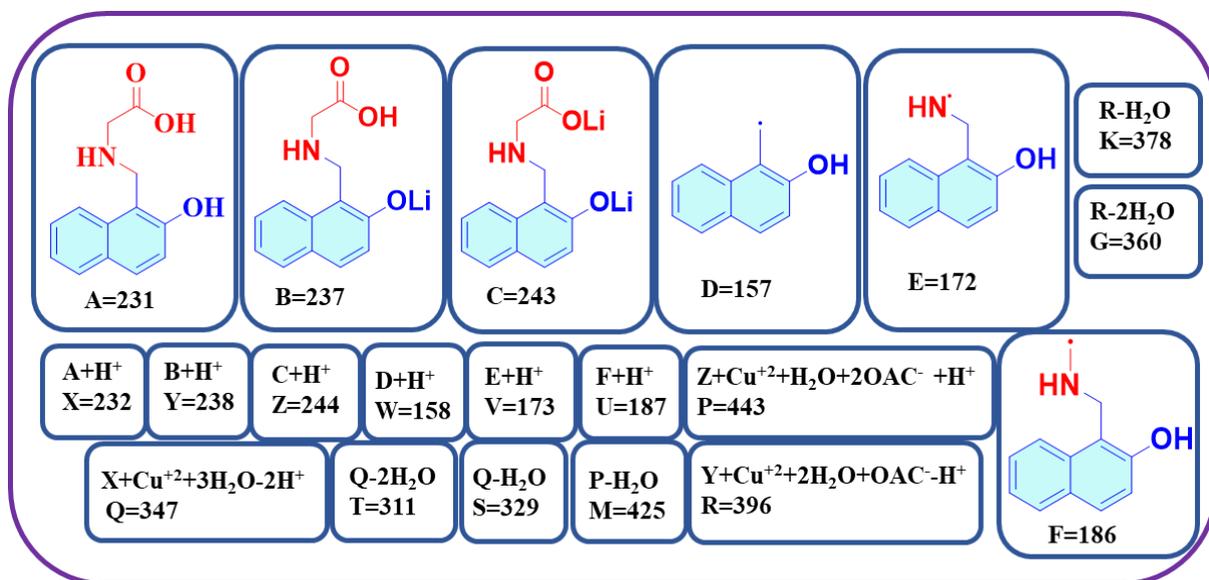
**Figure S6** Anion-specific formation test for Cu-MOG



**Figure S7** IR spectrum of Cu-MOG

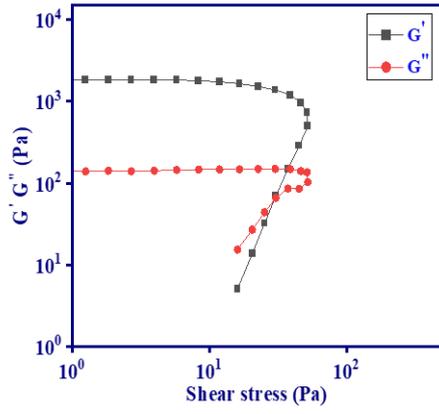


(a)

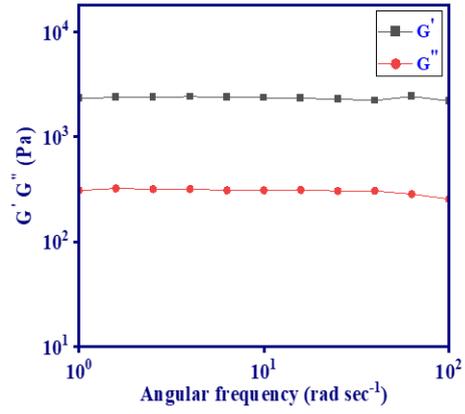


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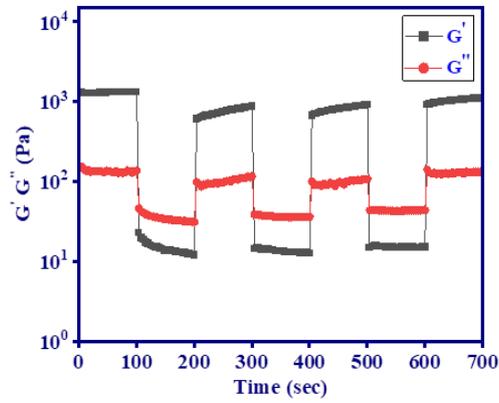
Figure S8 (a) MALDI TOF spectrum and (b) peak indexing of Cu-MOG



(a)

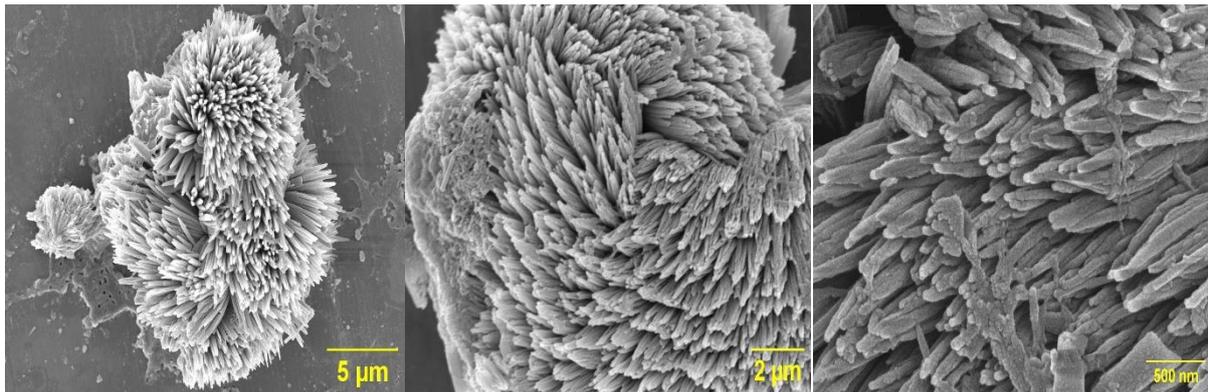


(b)

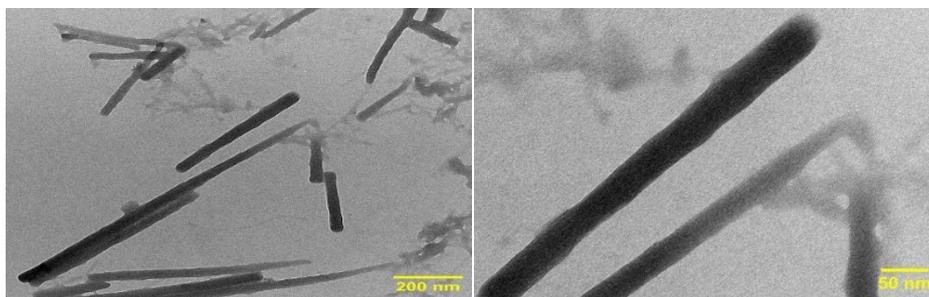


(c)

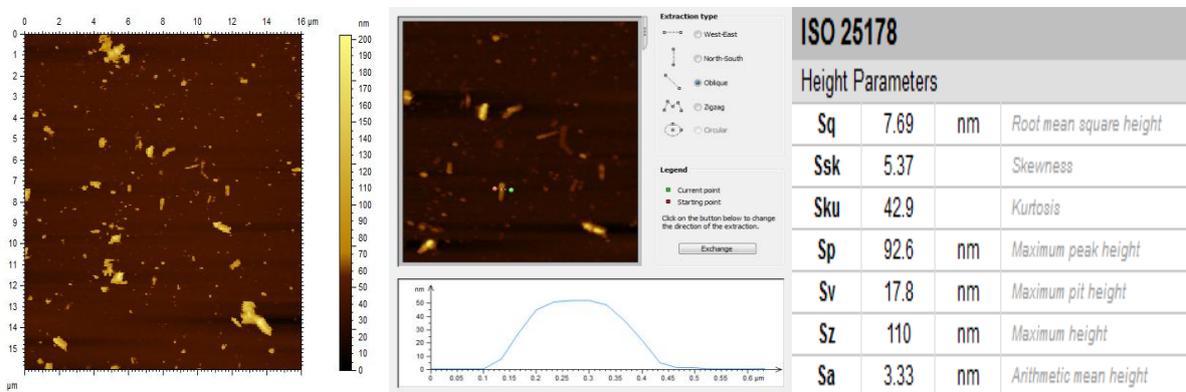
**Figure S9** Rheological measurement of Cu-MOG (a) Amplitude sweep, (b) Frequency sweep, and (c) Plot of the step strain experiment



(a)

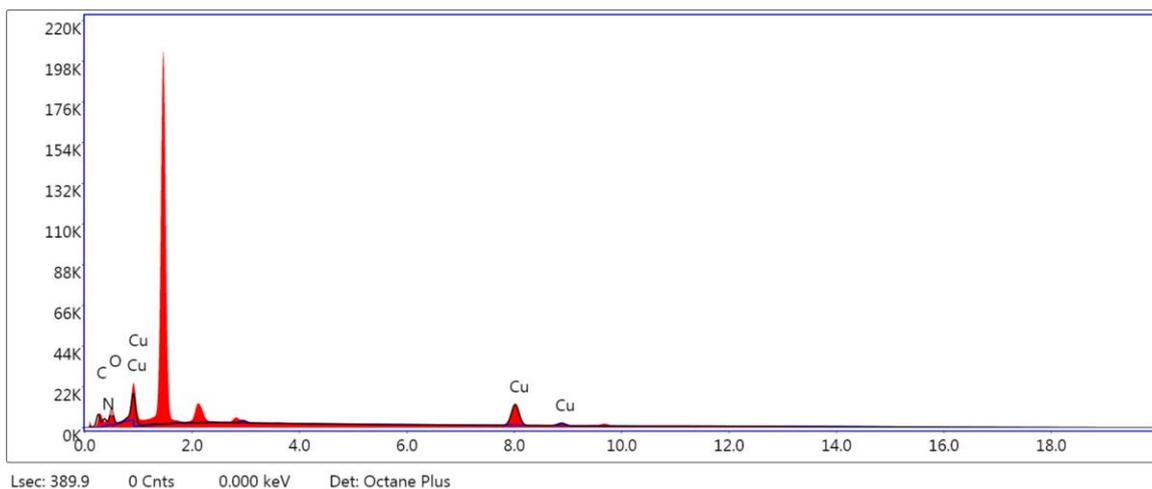


(b)

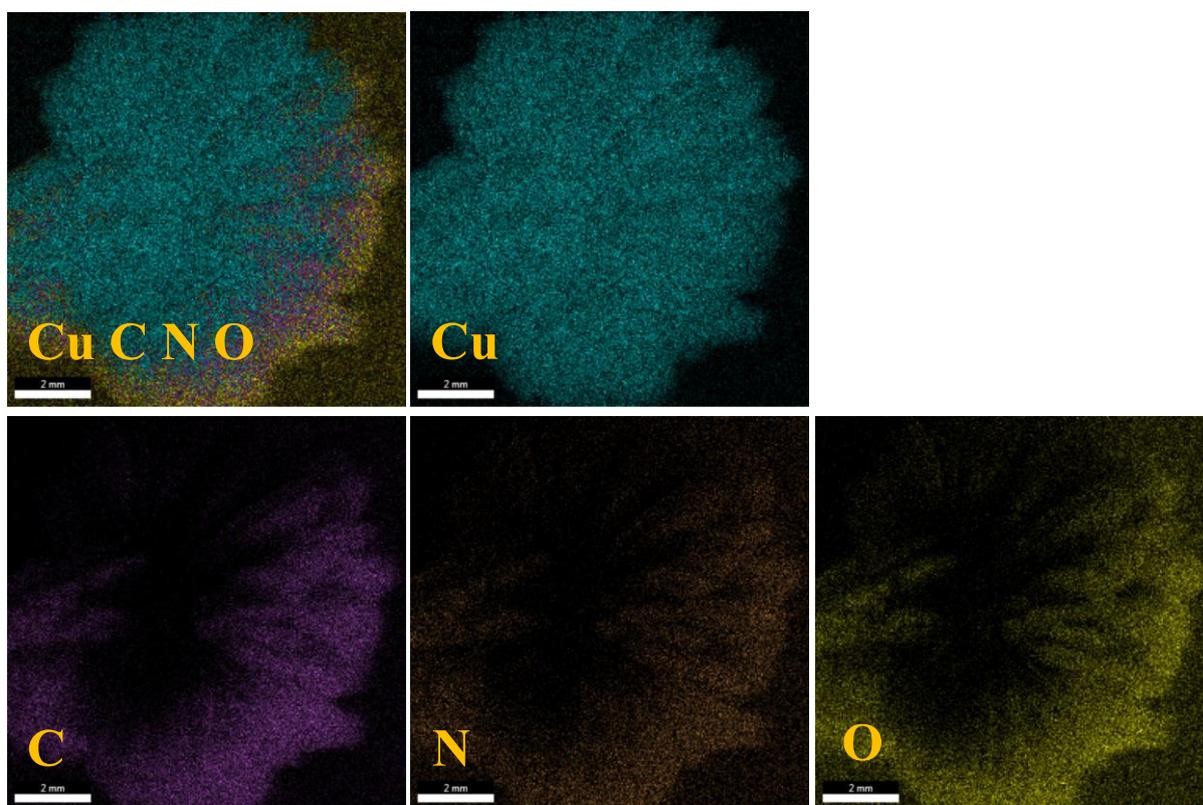


(c)

**Figure S10** FESEM (a), HRTEM (b), and AFM (c) images of Cu-MOG



Element	Weight %	Atomic %	Net Int.	Error %	Kratio	Z	A	F
C K	19.24	31.86	280.80	8.39	0.0636	1.1234	0.2942	1.0000
N K	18.15	25.78	182.80	9.60	0.0390	1.0994	0.1954	1.0000
O K	24.48	30.43	529.90	9.30	0.0501	1.0783	0.1897	1.0000
CuK	38.13	11.93	1032.60	2.11	0.3135	0.8008	1.0128	1.0135



**Figure S11** EDAX spectrum and elemental mapping of Cu-MOG

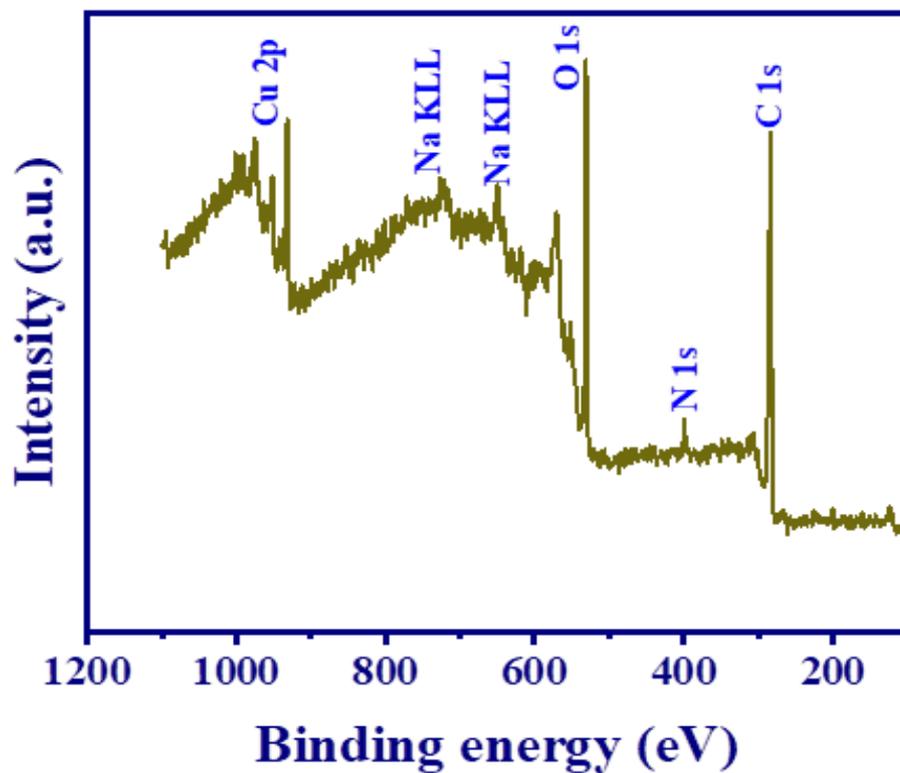


Figure S12 XPS survey scan of Cu-MOG

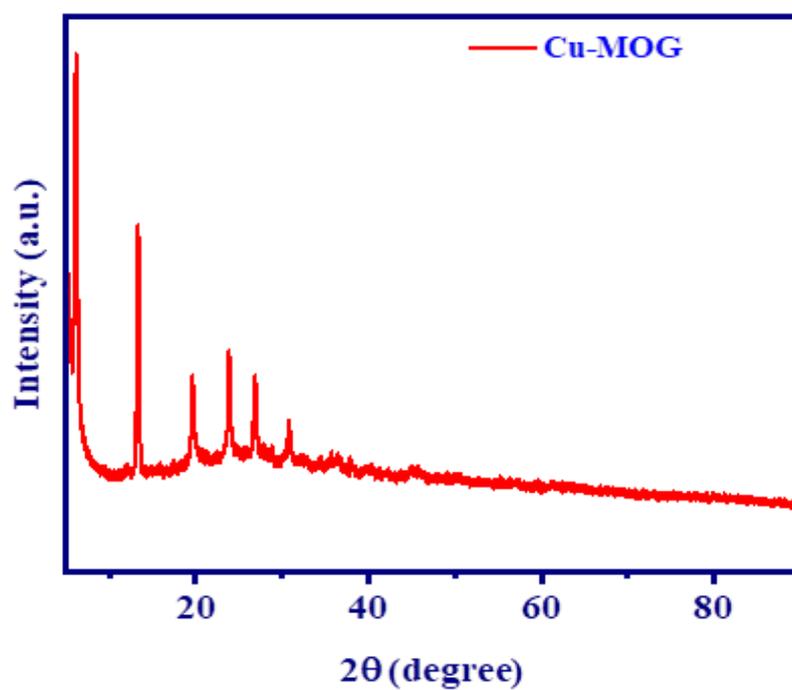


Figure S13 PXRD of the xerogel of Cu-MOG

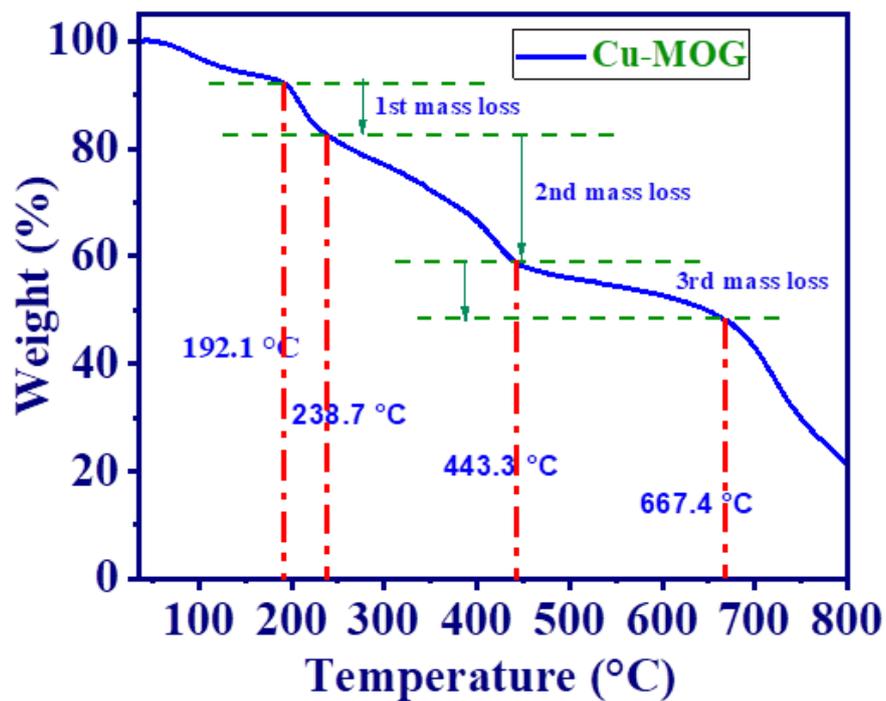


Figure S14 TGA curve of the xerogel of Cu-MOG

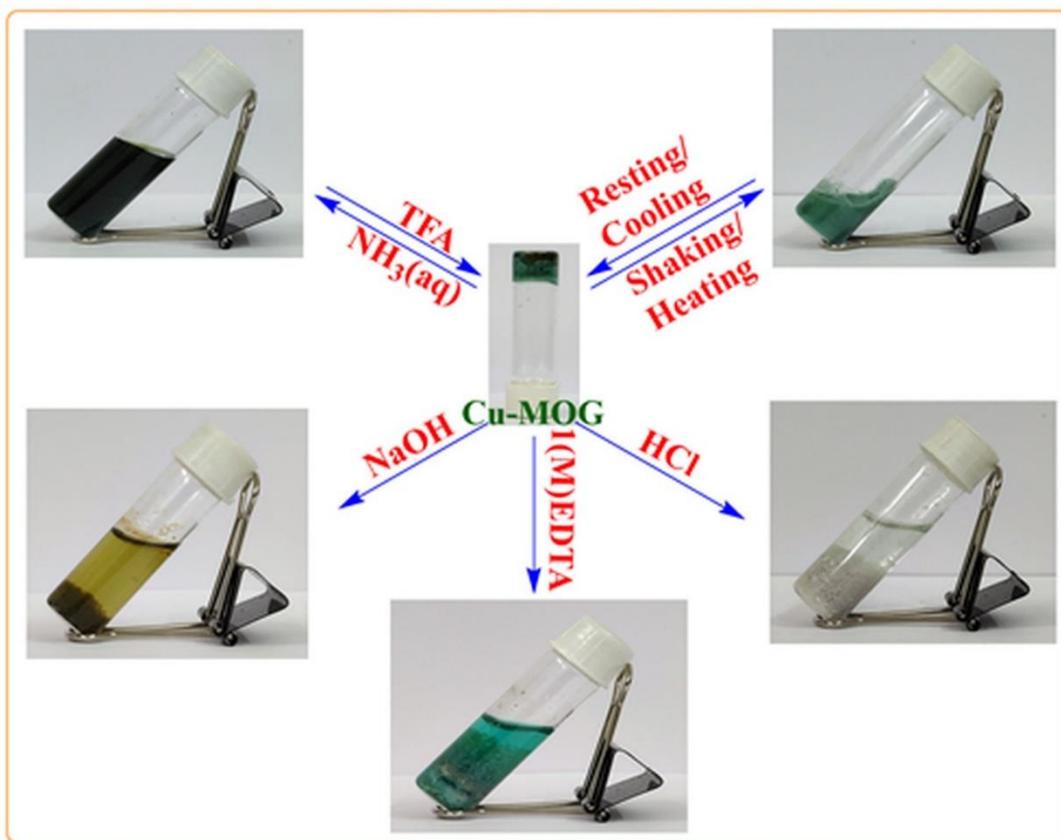
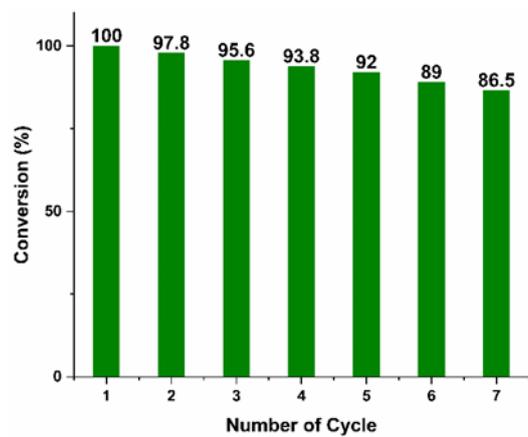
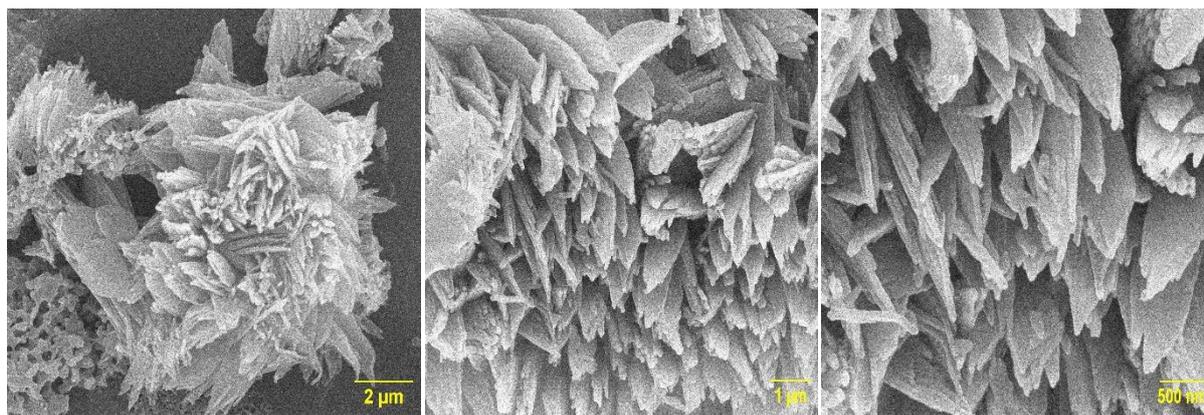


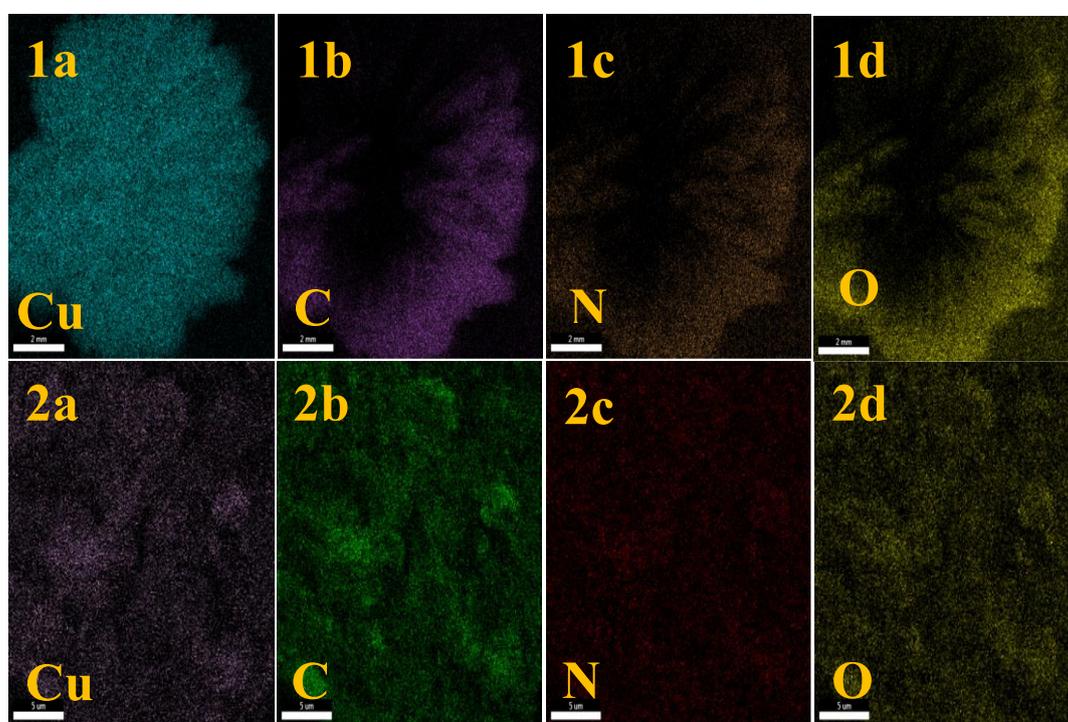
Figure S15 The multi-stimuli responsive nature of Cu-MOG



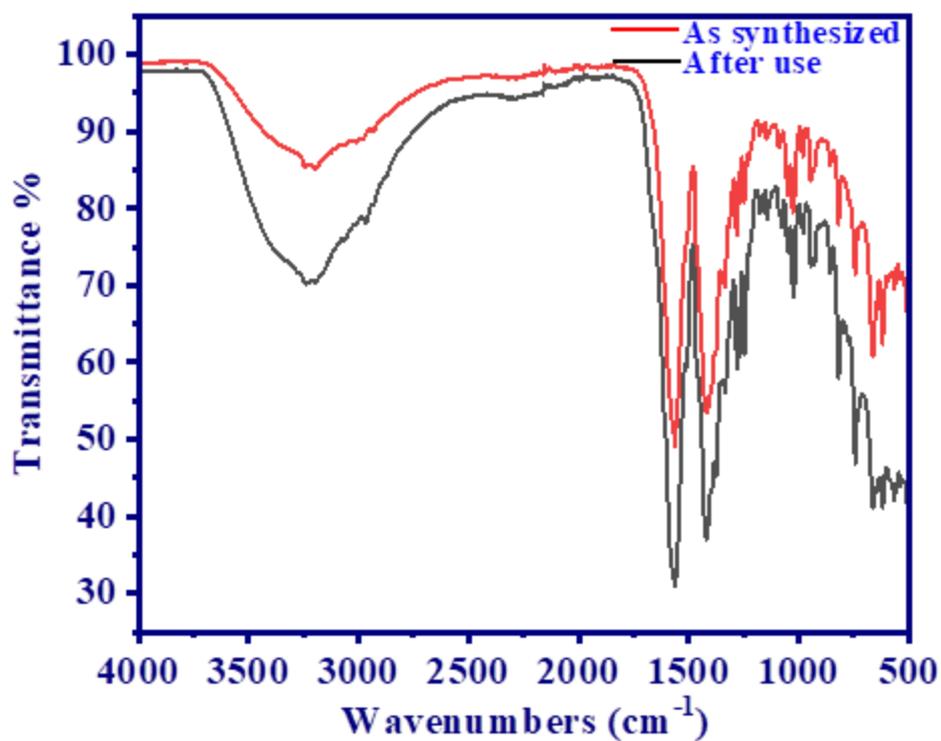
**Figure S16** Recyclability study of Cu-MOG



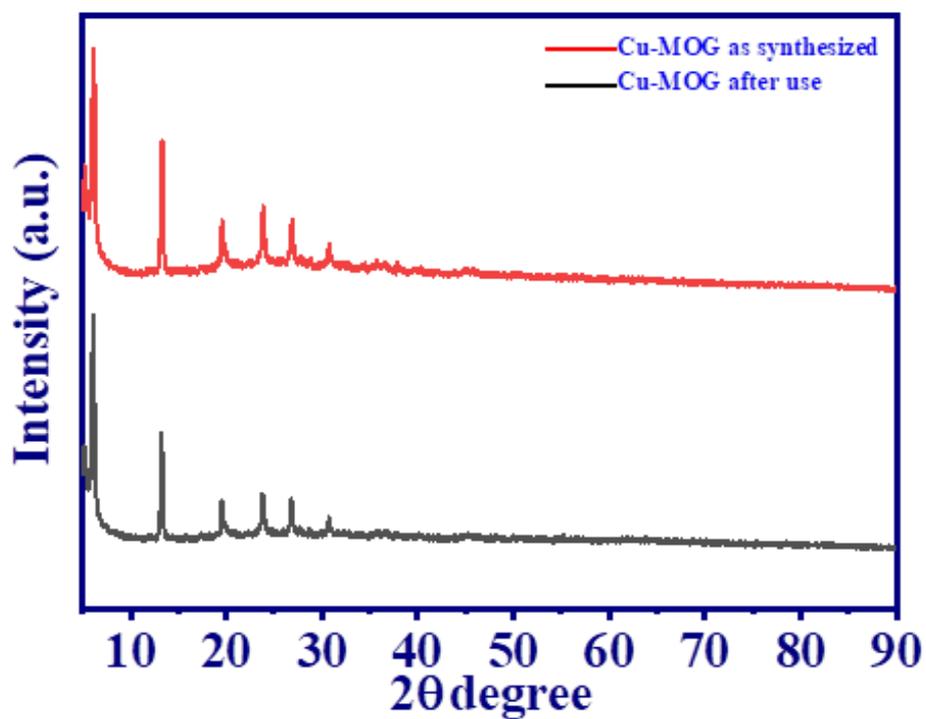
**Figure S17** FESEM images of recovered Cu-MOG after ring opening reaction



**Figure S18** Elemental mapping of Cu-MOG-3 before (1) and after (2) epoxide ring opening by amines



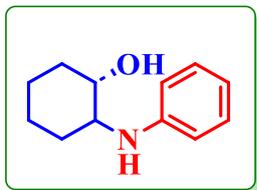
**Figure S19** FTIR spectra of Cu-MOG before and after epoxide ring opening reaction by amines



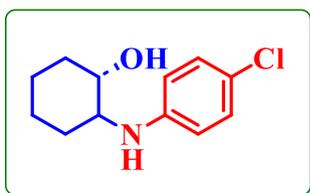
**Figure S20** PXRD pattern of Cu-MOG before and after epoxide ring opening reaction by amines

## Characterization Data of the Products

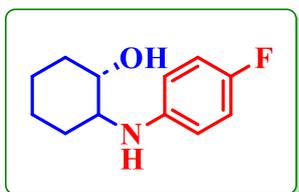
### NMR data ( $^1\text{H}$ , $^{13}\text{C}$ ) Products for Epoxide ring opening by amines.



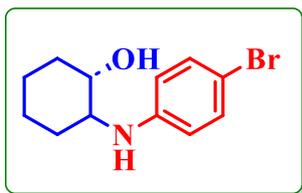
Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent ( Selectivity 100%, Yield 100%) (pale brownish solid) 2-(phenylamino)cyclohexan-1-ol<sup>S14</sup>:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.08 (t,  $J = 7.7$  Hz, 2H), 6.67-6.57 (m, 3H), 3.22 (td,  $J = 9.9, 4.3$  Hz, 1H), 3.08-2.97 (m, 2H), 2.00 (d,  $J = 12.2$  Hz, 2H), 1.63 (ddd,  $J = 9.7, 5.8, 3.5$  Hz, 2H), 1.31-1.16 (m, 3H), 0.92 (qd,  $J = 12.7, 3.3$  Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  147.92 (s), 129.37 (s), 118.28 (s), 114.41 (s), 74.43 (s), 60.11 (s), 33.30 (d,  $J = 1.9$  Hz), 31.60 (s), 25.01 (s), 24.34 (s).



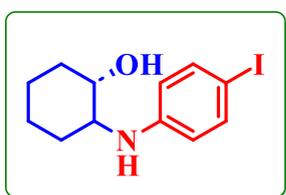
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent ( Selectivity 100%, Yield 98%) (white solid) 2-((4-chlorophenyl)amino)cyclohexan-1-ol<sup>S14</sup>:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.06-7.02 (m, 2H), 6.57-6.53 (m, 2H), 3.27 (td,  $J = 10.0, 4.3$  Hz, 1H), 3.03-2.96 (m, 1H), 2.06-1.97 (m, 2H), 1.73-1.60 (m, 2H), 1.36-1.19 (m, 3H), 0.97 (qd,  $J = 12.8, 3.7$  Hz, 1H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  146.44 (s), 129.14 (s), 122.81 (s), 115.43 (s), 74.51 (s), 60.35 (s), 33.28 (s), 31.51 (s), 24.93 (s), 24.24 (s).



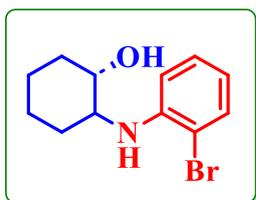
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent ( Selectivity 100%, Yield 97%) (white solid) 2-((4-fluorophenyl)amino)cyclohexan-1-ol<sup>S19</sup>:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  6.85-6.77 (m, 2H), 6.61-6.55 (m, 2H), 3.27 (td,  $J = 10.0, 4.4$  Hz, 1H), 2.99-2.92 (m, 1H), 2.11-1.97 (m, 2H), 1.67 (ddd,  $J = 26.8, 10.2, 4.6$  Hz, 2H), 1.36-1.16 (m, 3H), 1.01-0.90 (m, 1H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  157.55 (s), 155.20 (s), 144.01 (s), 115.85 (s), 115.63 (s), 74.46 (s), 61.23 (s), 33.20 (s), 31.51 (s), 25.02 (s), 24.27 (s).



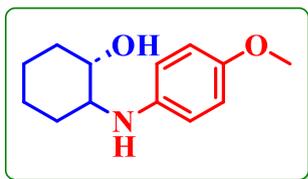
Purification by column chromatography with Hexane : Ethyl acetate (96:4) as eluent (Selectivity 100%, Yield 94%) (off white solid) 2-((4-fluorophenyl)amino)cyclohexan-1-ol<sup>S20</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):**  $\delta$  7.20-7.16 (m, 2H), 6.52 (d,  $J$  = 8.7 Hz, 2H), 3.28 (td,  $J$  = 9.9, 4.3 Hz, 1H), 3.04-2.97 (m, 1H), 2.00 (dd,  $J$  = 23.9, 11.5 Hz, 2H), 1.72-1.61 (m, 2H), 1.36-1.17 (m, 3H), 0.98 (qd,  $J$  = 12.7, 3.5 Hz, 1H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):**  $\delta$  146.71 (s), 132.04 (s), 116.00 (s), 110.01 (s), 74.46 (s), 60.33 (s), 33.29 (s), 31.44 (s), 24.91 (s), 24.23 (s).



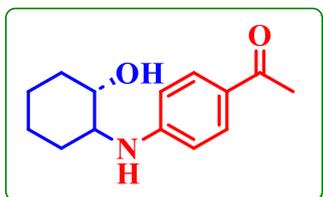
Purification by column chromatography with Hexane : Ethyl acetate (96:4) as eluent (Selectivity 100%, Yield 96%) (off white solid) 2-((2-bromophenyl)amino)cyclohexan-1-ol<sup>S19</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):**  $\delta$  7.35 (d,  $J$  = 8.6 Hz, 2H), 6.43 (d,  $J$  = 8.6 Hz, 2H), 3.29 (td,  $J$  = 9.9, 4.3 Hz, 1H), 3.01 (td,  $J$  = 11.4, 4.0 Hz, 1H), 2.01 (t,  $J$  = 12.7 Hz, 2H), 1.72 – 1.62 (m, 2H), 1.28 – 1.18 (m, 3H), 0.99 (qd,  $J$  = 12.6, 3.4 Hz, 1H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):**  $\delta$  147.27 (s), 137.93 (s), 116.61 (s), 79.17 (s), 74.46 (s), 60.17 (s), 33.29 (s), 31.42 (s), 24.89 (s), 24.22 (s).



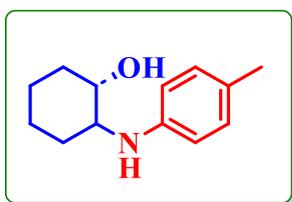
Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 91%) (pale brownish solid) 2-((2-bromophenyl)amino)cyclohexan-1-ol<sup>S20</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):**  $\delta$  7.28 (d,  $J$  = 7.9 Hz, 1H), 7.02 (t,  $J$  = 7.7 Hz, 1H), 6.68 (d,  $J$  = 8.1 Hz, 1H), 6.44 (t,  $J$  = 7.5 Hz, 1H), 3.31 – 3.23 (m, 1H), 3.06 – 3.00 (m, 1H), 1.93 (d,  $J$  = 11.5 Hz, 2H), 1.57 (d,  $J$  = 29.5 Hz, 2H), 1.18 (t,  $J$  = 16.8 Hz, 3H), 0.98 (dd,  $J$  = 15.7, 7.7 Hz, 1H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):**  $\delta$  144.90 (s), 132.56 (s), 128.54 (s), 118.39 (s), 112.95 (s), 110.90 (s), 74.19 (s), 59.78 (s), 33.36 (s), 31.58 (s), 24.82 (s), 24.23 (s).



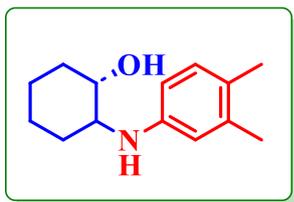
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent (Selectivity 100%, Yield 100%) (white solid) 2-((4-methoxyphenyl)amino)cyclohexan-1-ol<sup>S14</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):**  $\delta$  6.68 (d,  $J$  = 8.9 Hz, 2H), 6.58 (d,  $J$  = 8.8 Hz, 2H), 3.64 (s, 3H), 3.22 (td,  $J$  = 9.9, 4.3 Hz, 1H), 3.06 (s, 2H), 2.92 – 2.86 (m, 1H), 1.99 (dd,  $J$  = 8.3, 4.7 Hz, 2H), 1.62 (dd,  $J$  = 31.6, 9.3 Hz, 2H), 1.18 (t,  $J$  = 14.3 Hz, 3H), 0.90 (dd,  $J$  = 24.1, 12.4 Hz, 1H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):**  $\delta$  152.86 (s), 141.67 (s), 116.39 (s), 114.87 (s), 74.27 (s), 61.59 (s), 55.75 (s), 33.25 (s), 31.49 (s), 25.04 (s), 24.34 (s).



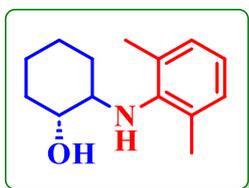
Purification by column chromatography with Hexane : Ethyl acetate (96:4) as eluent (Selectivity 100%, Yield 95%) (brown solid) 1-(4-(2-hydroxycyclohexyl)amino)phenyl ethan-1-one<sup>S18</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  7.71 (dd,  $J$  = 8.6, 1.9 Hz, 2H), 6.56 (dd,  $J$  = 10.0, 3.2 Hz, 2H), 3.35 (td,  $J$  = 9.8, 4.3 Hz, 1H), 3.24-3.13 (m, 1H), 2.40 (d,  $J$  = 3.4 Hz, 3H), 2.03 (d,  $J$  = 13.1 Hz, 2H), 1.75-1.62 (m, 2H), 1.28 (ddd,  $J$  = 32.2, 23.7, 13.7 Hz, 3H), 1.11-0.99 (m, 1H). **<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  196.56 (s), 152.26 (s), 130.84 (s), 126.90 (s), 112.31 (s), 74.46 (s), 59.05 (s), 33.57 (s), 31.57 (s), 26.02 (s), 24.71 (s), 24.22 (s).



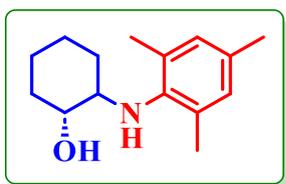
Purification by column chromatography with Hexane: Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 89%) (white solid) 2-(p-tolylamino)cyclohexan-1-ol<sup>S14</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  6.86 (d,  $J$  = 8.2 Hz, 2H), 6.49 (d,  $J$  = 8.4 Hz, 2H), 3.22 – 3.11 (m, 3H), 2.91 (ddd,  $J$  = 11.1, 9.4, 3.9 Hz, 1H), 2.12 (s, 3H), 1.94 (d,  $J$  = 11.2 Hz, 2H), 1.63 – 1.51 (m, 2H), 1.23 – 1.09 (m, 3H), 0.88 – 0.78 (m, 1H). **<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  144.54 (s), 128.72 (s), 126.23 (s), 113.61 (s), 73.10 (s), 59.34 (s), 32.28 (s), 30.40 (s), 23.88 (s), 23.28 (s), 19.36 (s).



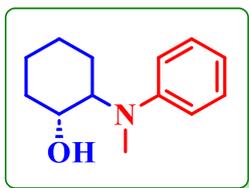
Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 87%) (off white solid) 2-((3,4-dimethylphenyl)amino)cyclohexan-1-ol<sup>S20</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  6.84 (d,  $J$  = 8.0 Hz, 1H), 6.44 – 6.35 (m, 2H), 3.19 (td,  $J$  = 10.0, 4.3 Hz, 1H), 3.00 – 2.91 (m, 3H), 2.08 (d,  $J$  = 15.8 Hz, 6H), 1.99 (dd,  $J$  = 13.2, 2.1 Hz, 2H), 1.62 (ddd,  $J$  = 27.7, 6.5, 3.6 Hz, 2H), 1.28 – 1.14 (m, 3H), 0.89 (qd,  $J$  = 12.8, 3.7 Hz, 1H). **<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  145.97 (s), 137.44 (s), 130.41 (s), 126.48 (s), 116.51 (s), 112.15 (s), 74.44 (s), 60.63 (s), 33.22 (s), 31.65 (s), 25.12 (s), 24.39 (s), 20.08 (s), 18.77 (s).



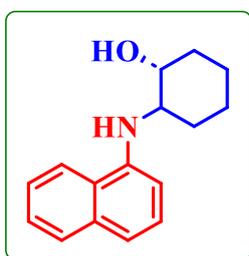
Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 91%) (colourless oil) 2-((2,6-dimethylphenyl)amino)cyclohexan-1-ol<sup>S16</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):**  $\delta$  6.89 (d,  $J$  = 7.5 Hz, 2H), 6.73 (t,  $J$  = 7.5 Hz, 1H), 3.31 (td,  $J$  = 10.0, 4.4 Hz, 1H), 2.81 – 2.73 (m, 1H), 2.19 (s, 6H), 2.06 – 2.00 (m, 1H), 1.74 – 1.51 (m, 3H), 1.32 – 1.14 (m, 3H), 1.05 – 0.92 (m, 2H). **<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>):**  $\delta$  144.10 (s), 129.83 (s), 129.13 (s), 122.29 (s), 75.09 (s), 63.41 (s), 33.18 (s), 32.45 (s), 25.27 (s), 24.35 (s), 19.21 (s).



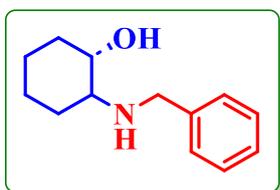
Purification by column chromatography with Hexane : Ethyl acetate (98:2) as eluent (Selectivity 100%, Yield 91%) (light Brown oil) 2-((2,6-dimethylphenyl)amino)cyclohexan-1-ol<sup>S25</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  6.89 (s, 2H), 3.53 – 3.45 (m, 1H), 2.93 – 2.87 (m, 1H), 2.34 (d,  $J$  = 13.8 Hz, 9H), 1.93 – 1.59 (m, 4H), 1.50 – 1.36 (m, 2H), 1.21 – 1.09 (m, 2H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**  $\delta$  141.62 (s), 131.33 (s), 130.10 (s), 129.78 (s), 74.98 (s), 63.68 (s), 33.49 (s), 32.30 (s), 25.35 (s), 24.50 (s), 20.64 (s), 19.06 (s).



Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 79%) (light brown oil) 2-(methyl(phenyl)amino)cyclohexan-1-ol<sup>S20</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):**  $\delta$  7.34-7.18 (m, 2H), 7.00-6.72 (m, 3H), 3.68-3.18 (m, 3H), 2.79-2.60 (m, 3H), 2.22 (d,  $J$  = 10.3 Hz, 1H), 1.81-1.66 (m, 3H), 1.47-1.21 (m, 5H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>):**  $\delta$  151.40 (s), 129.10 (s), 118.06 (s), 115.18 (s), 70.08 (s), 66.49 (s), 33.87 (s), 31.03 (s), 26.46 (s), 25.60 (s), 24.56 (s).

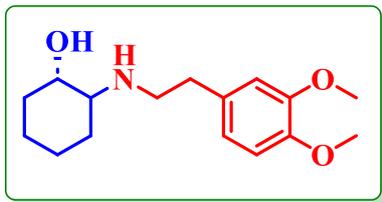


Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 100%) (White solid) 2-(naphthalen-1-ylamino)cyclohexan-1-ol<sup>S20</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.79 – 7.68 (m, 2H), 7.41 – 7.33 (m, 2H), 7.26 (t,  $J$  = 7.8 Hz, 1H), 7.21 – 7.16 (m, 1H), 6.72 (d,  $J$  = 7.4 Hz, 1H), 3.45 (td,  $J$  = 10.0, 4.2 Hz, 1H), 3.29 (td,  $J$  = 11.2, 3.8 Hz, 1H), 2.68 (s, 1H), 2.13 (dd,  $J$  = 33.2, 13.2 Hz, 2H), 1.76 – 1.63 (m, 2H), 1.43 – 1.23 (m, 3H), 1.11 – 0.99 (m, 1H). **<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**  $\delta$  142.86 (s), 134.52 (s), 128.82 (s), 126.49 (s), 125.81 (s), 124.92 (s), 124.28 (s), 120.00 (s), 118.37 (s), 106.45 (s), 74.65 (s), 59.90 (s), 33.46 (s), 31.29 (s), 24.97 (s), 24.36 (s).

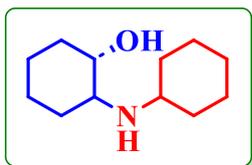


Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 100%) (brown solid) 2-(benzylamino)cyclohexan-1-ol<sup>S14</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>):**  $\delta$  7.29-7.15 (m, 5H), 3.89 (d,  $J$  = 12.9 Hz, 1H), 3.62 (d,  $J$  = 12.9 Hz, 1H), 3.14 (td,  $J$  = 9.8, 4.6 Hz, 1H), 2.27-2.20 (m, 1H), 2.11-2.05 (m, 1H), 1.95 (dd,  $J$  = 10.1, 3.4 Hz, 1H), 1.65 (dd,  $J$  = 7.9, 4.7 Hz, 2H), 1.17 (d,  $J$  = 10.6 Hz, 3H), 0.94 (dt,  $J$  = 12.4, 9.2 Hz, 1H).

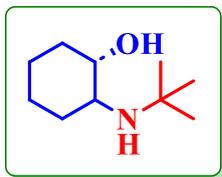
**$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):**  $\delta$  140.24 (s), 128.47 (s), 128.18 (s), 127.10 (s), 73.73 (s), 63.11 (s), 50.71 (s), 33.33 (s), 30.43 (s), 25.14 (s), 24.33 (s).



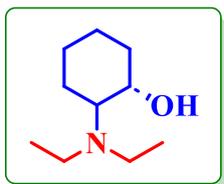
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent (Selectivity 100%, Yield 73%) (brown sticky solid) 2-((3,4-dimethoxyphenethyl)amino)cyclohexan-1-ol:  **$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):**  $\delta$  6.72 – 6.63 (m, 3H), 3.76 (d,  $J$  = 12.4 Hz, 6H), 3.30 – 3.10 (m, 3H), 2.93 (s, 1H), 2.66 (s, 2H), 2.20 (s, 1H), 1.98 – 1.87 (m, 2H), 1.61 (d,  $J$  = 6.9 Hz, 2H), 1.19 – 1.11 (m, 3H), 0.90 (dd,  $J$  = 23.8, 12.1 Hz, 1H).  **$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):**  $\delta$  147.89 (s), 146.44 (s), 131.37 (s), 119.56 (s), 111.00 (s), 110.34 (s), 72.26 (s), 62.43 (s), 54.87 (s), 47.00 (s), 35.01 (s), 32.68 (s), 29.07 (s), 23.93 (s), 23.37 (s).



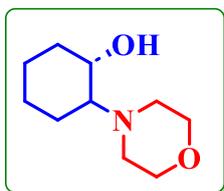
Purification by Purification by work up only (Selectivity 100%, Yield 96%) (brown solid) 2-(cyclohexylamino)cyclohexan-1-ol<sup>S24</sup>:  **$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):**  $\delta$  3.06 (s, 1H), 2.59 (d,  $J$  = 43.8 Hz, 3H), 2.26 (t,  $J$  = 8.1 Hz, 1H), 1.97 (s, 2H), 1.85 (d,  $J$  = 11.3 Hz, 1H), 1.71 – 1.51 (m, 5H), 1.17 (d,  $J$  = 20.8 Hz, 6H), 1.00 – 0.82 (m, 2H).  **$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):**  $\delta$  73.64 (s), 60.46 (s), 53.51 (s), 34.85 (s), 33.21 (s), 31.18 (s), 25.98 (s), 25.21 (d,  $J$  = 19.4 Hz), 24.63 (s), 24.30 (s).



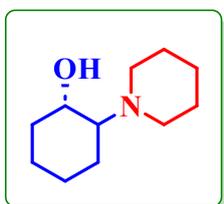
Purification by Purification by work up only (Selectivity 100%, Yield 96%) (brown solid) 2-(tert-butylamino)cyclohexan-1-ol<sup>S27</sup>:  **$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )**  $\delta$  3.02 – 2.95 (m, 1H), 2.90 (d,  $J$  = 0.7 Hz, 2H), 2.29 – 2.22 (m, 1H), 2.03 – 1.92 (m, 2H), 1.68 – 1.60 (m, 2H), 1.21 (t,  $J$  = 10.2 Hz, 3H), 1.09 (s, 9H).  **$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):**  $\delta$  73.74 (s), 58.58 (s), 51.99 (s), 34.23 (s), 33.01 (s), 30.09 (d,  $J$  = 7.6 Hz), 25.71 (s), 24.42 (s).



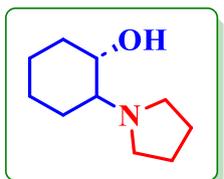
Purification by Purification by work up only (Selectivity 100%, Yield 96%) (brown oil) 2-(diethylamino)cyclohexan-1-ol<sup>S28</sup>: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 3.81 (s, 1H), 3.30 – 3.23 (m, 1H), 2.21 (d, *J* = 5.0 Hz, 6H), 2.13 (d, *J* = 9.9 Hz, 1H), 2.02 (d, *J* = 2.0 Hz, 1H), 1.78 – 1.54 (m, 4H), 1.20 – 1.00 (m, 5H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 69.56 (s), 69.28 (s), 52.02 (s), 40.13 (s), 33.27 (s), 25.23 (s), 24.11 (s), 20.44 (s), 19.35 (s).



Purification by work up only (Selectivity 100%, Yield 98%) (red oily liquid) 2-morpholinocyclohexan-1-ol<sup>S18</sup>: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 3.63 (ddd, *J* = 10.8, 8.6, 5.2 Hz, 4H), 3.30 (dd, *J* = 11.0, 7.7 Hz, 1H), 2.64 (s, 2H), 2.36 (dd, *J* = 8.3, 5.5 Hz, 2H), 2.15-1.97 (m, 2H), 1.71 (dd, *J* = 42.3, 25.6 Hz, 3H), 1.22-1.05 (m, 4H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 70.19 (s), 68.13 (s), 67.15 (s), 48.54 (s), 33.06 (s), 25.22 (s), 23.82 (s), 22.05 (s).

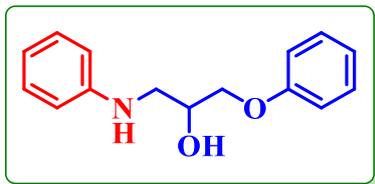


Purification by work up only (Selectivity 100%, Yield 93%) (brown oily liquid) 2-(piperidin-1-yl)cyclohexan-1-ol<sup>S14</sup>: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): 3.90 (s, 1H), 3.28 (dd, *J* = 11.6, 7.4 Hz, 1H), 2.68 – 2.56 (m, 2H), 2.27 (s, 2H), 2.13 – 1.99 (m, 2H), 1.71 (d, *J* = 11.8 Hz, 2H), 1.63 – 1.35 (m, 6H), 1.20 – 1.04 (m, 4H). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>): δ 70.92 (s), 68.42 (s), 49.66 (s), 33.22 (s), 26.58 (s), 25.52 (s), 24.71 (s), 24.02 (s), 22.10 (s).

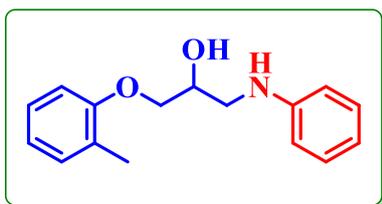


Purification by work up only (Selectivity 100%, Yield 95%) (brown oily liquid) 2-(pyrrolidin-1-yl)cyclohexan-1-ol<sup>S14</sup>: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>): δ 3.69-3.63 (m, 1H), 3.47-3.42 (m, 1H), 2.56 (s, 1H), 2.20-1.99 (m, 3H), 1.71-1.53 (m, 4H), 1.37-1.14 (m, 8H). <sup>13</sup>C NMR (126

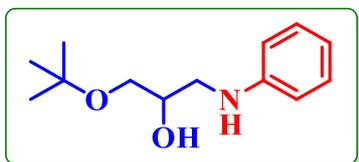
**MHz, CDCl<sub>3</sub>**):  $\delta$  74.30 (s), 66.43 (s), 34.14 (s), 32.14 (s), 28.68 (s), 24.62 (s), 22.95 (s), -0.00 (s).



Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent ( Selectivity 100%, Yield 96%) (colourless oil) 1-phenoxy-3-(phenylamino)propan-2-ol<sup>S14</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**:  $\delta$  7.46-7.39 (m, 2H), 7.37-7.29 (m, 2H), 7.16-7.09 (m, 1H), 7.07-7.00 (m, 2H), 6.93-6.87 (m, 1H), 6.82-6.75 (m, 2H), 4.35-4.27 (m, 1H), 4.10 (t,  $J = 4.8$  Hz, 2H), 3.75 (s, 2H), 3.49 (dd,  $J = 12.9, 3.6$  Hz, 1H), 3.34 (dd,  $J = 12.8, 7.3$  Hz, 1H). **<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)**:  $\delta$  158.60 (s), 148.25 (s), 129.78 (s), 129.53 (s), 121.45 (s), 118.20 (s), 114.76 (s), 113.56 (s), 70.21 (s), 68.88 (s), 46.85 (s).

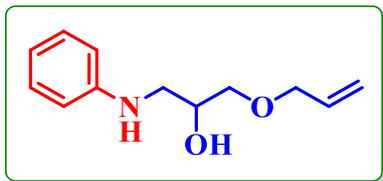


Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent ( Selectivity 100%, Yield 91%) (brown oil) 1-(phenylamino)-3-(o-tolyloxy)propan-2-ol<sup>S22</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**:  $\delta$  7.41 (ddd,  $J = 24.6, 17.1, 10.0$  Hz, 4H), 7.14 (dt,  $J = 14.0, 6.0$  Hz, 1H), 7.05-6.80 (m, 4H), 4.39 (d,  $J = 2.7$  Hz, 1H), 4.15 (d,  $J = 2.3$  Hz, 2H), 3.89 (d,  $J = 0.8$  Hz, 2H), 3.62 (dd,  $J = 9.6, 5.5$  Hz, 1H), 3.49-3.39 (m, 1H), 2.56-2.44 (m, 3H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**: 156.81 (s), 148.39 (s), 131.10 (s), 129.63 (s), 127.22 (s), 126.89 (s), 121.24 (s), 118.28 (s), 113.64 (s), 111.47 (s), 69.04 (s), 47.21 (s), 16.58 (s).

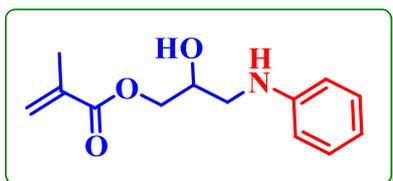


Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent (Selectivity 100%, Yield 93%) (light brown oil) 1-(tert-butoxy)-3-(phenylamino)propan-2-ol<sup>S15</sup>: **<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**  $\delta$  7.26 (dd,  $J = 8.4, 7.5$  Hz, 2H), 6.81 (t,  $J = 7.3$  Hz, 1H), 6.73 (d,  $J = 7.7$  Hz, 2H), 4.02 (dt,  $J = 10.6, 5.3$  Hz, 1H), 3.52-3.44 (m, 2H), 3.34 (dd,  $J = 12.7, 4.2$  Hz, 1H), 3.18 (dd,  $J = 12.7, 7.2$  Hz, 1H), 1.28 (d,  $J = 15.5$  Hz, 9H). **<sup>13</sup>C NMR (101 MHz,**

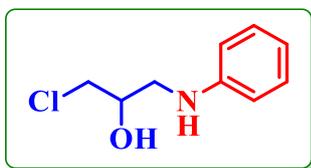
$\text{CDCl}_3$ )  $\delta$  148.62 (s), 129.30 (s), 117.60 (s), 113.37 (s), 73.43 (s), 69.42 (s), 64.47 (s), 47.20 (s), 27.59 (s).



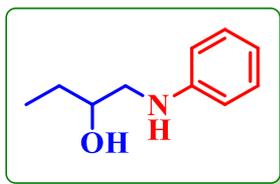
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent (Selectivity 100%, Yield 100%) (brown oil) 1-(allyloxy)-3-(phenylamino)propan-2-ol<sup>S21</sup>:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.14 (t,  $J$  = 7.2 Hz, 2H), 6.69 (t,  $J$  = 6.7 Hz, 1H), 6.60 (d,  $J$  = 8.3 Hz, 2H), 5.88 (ddd,  $J$  = 16.0, 10.8, 5.6 Hz, 1H), 5.29 – 5.14 (m, 2H), 3.97 (d,  $J$  = 5.2 Hz, 3H), 3.50 – 3.40 (m, 3H), 3.23 (dd,  $J$  = 12.8, 3.7 Hz, 1H), 3.08 (dd,  $J$  = 12.8, 7.4 Hz, 1H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  148.34 (s), 134.46 (s), 129.33 (s), 117.80 (s), 117.52 (s), 113.33 (s), 72.61 (s), 72.40 (s), 69.08 (s), 46.88 (s).



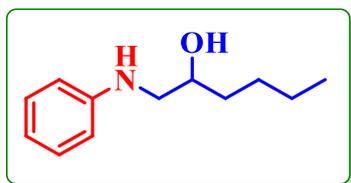
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent (Selectivity 100%, Yield 82%) (brown oil) 2-hydroxy-3-(phenylamino)propyl methacrylate<sup>S21</sup>:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.02 (t,  $J$  = 7.3 Hz, 2H), 6.58 (t,  $J$  = 7.4 Hz, 1H), 6.49 (t,  $J$  = 8.9 Hz, 2H), 6.00 (d,  $J$  = 13.0 Hz, 1H), 5.44 (d,  $J$  = 10.6 Hz, 1H), 4.06 (d,  $J$  = 5.0 Hz, 2H), 3.92 (s, 1H), 3.61 (d,  $J$  = 4.2 Hz, 2H), 3.25 – 2.95 (m, 2H), 1.80 (d,  $J$  = 12.6 Hz, 3H).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.74 (s), 148.12 (s), 135.97 (s), 129.40 (s), 126.50 (s), 118.01 (s), 113.37 (s), 113.14 (s), 68.29 (s), 66.79 (s), 46.77 (s), 18.34 (s).



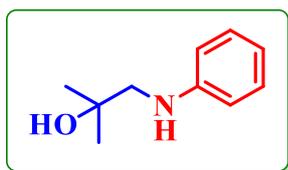
Purification by column chromatography with Hexane : Ethyl acetate (95:5) as eluent (Selectivity 100%, Yield 93%) (brown oil) 1-chloro-3-(phenylamino)propan-2-ol<sup>S14</sup>:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.11-7.04 (m, 2H), 6.68-6.63 (m, 1H), 6.56-6.51 (m, 2H), 3.89 (ddt,  $J$  = 9.1, 7.4, 4.5 Hz, 1H), 3.54-3.39 (m, 4H), 3.20 (dd,  $J$  = 13.3, 4.3 Hz, 1H), 3.03 (dd,  $J$  = 13.3, 7.4 Hz, 1H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ):  $\delta$  147.70 (s), 129.50 (s), 118.48 (s), 113.59 (s), 69.83 (s), 47.59 (s), 47.29 (s).



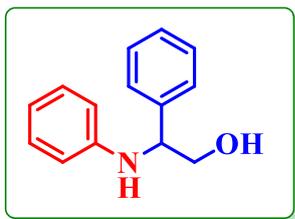
Purification by column chromatography with Hexane : Ethyl acetate (97:3) as eluent (Selectivity 100%, Yield 91%) (colourless oil) 1-(phenylamino)butan-2-ol<sup>S21</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  7.16 – 7.08 (m, 2H), 6.73 – 6.64 (m, 1H), 6.58 (d,  $J = 7.1$  Hz, 2H), 3.61 (dd,  $J = 15.9, 4.3$  Hz, 1H), 3.36 (ddd,  $J = 15.8, 10.0, 4.7$  Hz, 2H), 3.14 (d,  $J = 12.8$  Hz, 1H), 2.88 (dd,  $J = 12.6, 8.7$  Hz, 1H), 1.53 – 1.40 (m, 2H), 0.91 (dt,  $J = 15.2, 6.9$  Hz, 3H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**  $\delta$  148.47 (s), 129.38 (s), 117.90 (s), 113.46 (s), 71.68 (s), 49.88 (s), 28.04 (s), 10.10 (s).



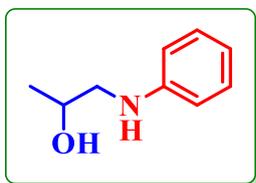
**Figure S85.** Purification by column chromatography with Hexane : Ethyl acetate (98:2) as eluent (Selectivity 100%, Yield 97%) (brown oil) 1-(phenylamino)hexan-2-ol<sup>S17</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**:  $\delta$  7.17 (t,  $J = 7.7$  Hz, 2H), 6.72 (t,  $J = 7.3$  Hz, 1H), 6.64 (d,  $J = 7.8$  Hz, 2H), 3.84 – 3.76 (m, 1H), 3.24 (dd,  $J = 12.8, 1.6$  Hz, 1H), 2.97 (dd,  $J = 12.5, 8.8$  Hz, 1H), 1.55 – 1.29 (m, 7H), 0.92 (t,  $J = 6.3$  Hz, 3H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**:  $\delta$  148.31 (s), 129.31 (s), 117.92 (s), 113.35 (s), 70.39 (s), 50.36 (s), 34.83 (s), 27.82 (s), 22.76 (s), 14.04 (s).



Purification by column chromatography with Hexane: Ethyl acetate (93:7) as eluent (Selectivity 100%, Yield 96%) (colourless oil) 2-methyl-1-(phenylamino)propan-2-ol<sup>S26</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  7.06 (dd,  $J = 9.1, 7.9$  Hz, 2H), 6.60 (dd,  $J = 7.7, 6.6$  Hz, 1H), 6.53 (d,  $J = 7.7$  Hz, 2H), 2.94 (s, 2H), 1.15 (s, 6H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**  $\delta$  148.90 (s), 129.35 (s), 117.68 (s), 113.29 (s), 70.87 (s), 55.08 (s), 27.60 (s).



Purification by column chromatography with Hexane: Ethyl acetate (92:8) as eluent (Selectivity 100%, Yield 96%) (colourless oil) 2-phenyl-2-(phenylamino)ethan-1-ol<sup>S14</sup>: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**  $\delta$  7.54 – 7.43 (m, 5H), 7.37 – 7.28 (m, 2H), 6.93 (dd,  $J = 12.1, 5.1$  Hz, 1H), 6.79 (dd,  $J = 7.4, 4.4$  Hz, 2H), 4.63 (dd,  $J = 7.1, 3.6$  Hz, 1H), 4.03 – 3.96 (m, 1H), 3.83 – 3.77 (m, 1H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**:  $\delta$  147.66 (s), 140.61 (s), 129.48 (s), 128.97 (s), 127.74 (s), 127.03 (s), 118.11 (s), 114.24 (s), 67.38 (s), 60.16 (s).



Purification by column chromatography with Hexane: Ethyl acetate (95:5) as eluent (79.16:20.84, Yield 80%) (colourless oil) 1-(phenylamino)propan-2-ol, and 2-(phenylamino)propan-1-ol: **<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)**:  $\delta$  7.09 – 7.03 (m, 2H), 6.62 (d,  $J = 6.3$  Hz, 1H), 6.53 – 6.47 (m, 2H), 3.83 (ddd,  $J = 9.5, 6.2, 3.4$  Hz, 1H), 3.50 (dd,  $J = 10.7, 4.3$  Hz, 0.28H), 3.46 – 3.40 (m, 0.45H), 3.33 (dd,  $J = 10.1, 5.6$  Hz, 0.53H), 3.03 (dd,  $J = 12.9, 3.4$  Hz, 1H), 2.81 (dd,  $J = 12.8, 8.4$  Hz, 1H), 1.08 (d,  $J = 6.3$  Hz, 2.38H), 1.02 (d,  $J = 6.4$  Hz, 0.62H). **<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)**:  $\delta$  148.36 (s), 117.91 (s), 113.97 (s), 113.39 (s), 66.37 (s), 51.67 (s), 20.94 (s), 17.49 (s).

## NMR spectra ( $^1\text{H}$ , $^{13}\text{C}$ ) of products

mb.msr423  
mb / ms / r - 423

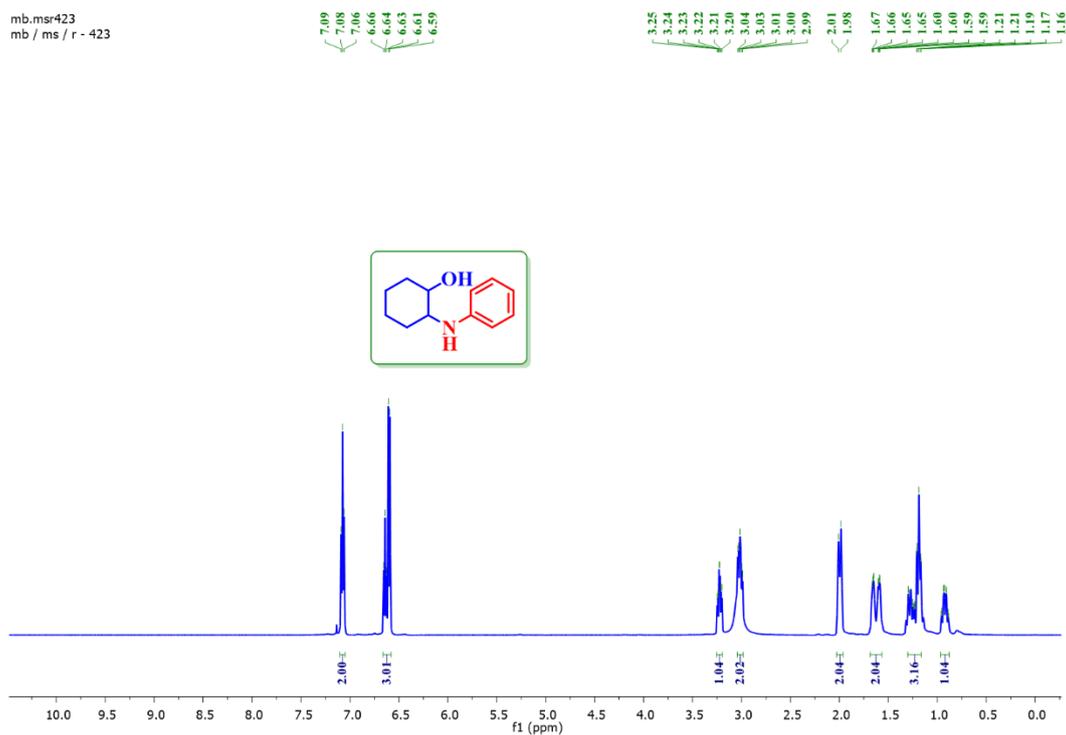


Figure S22  $^1\text{H}$  NMR spectrum of **3a**

mb.msr423  
mb / ms / r - 423

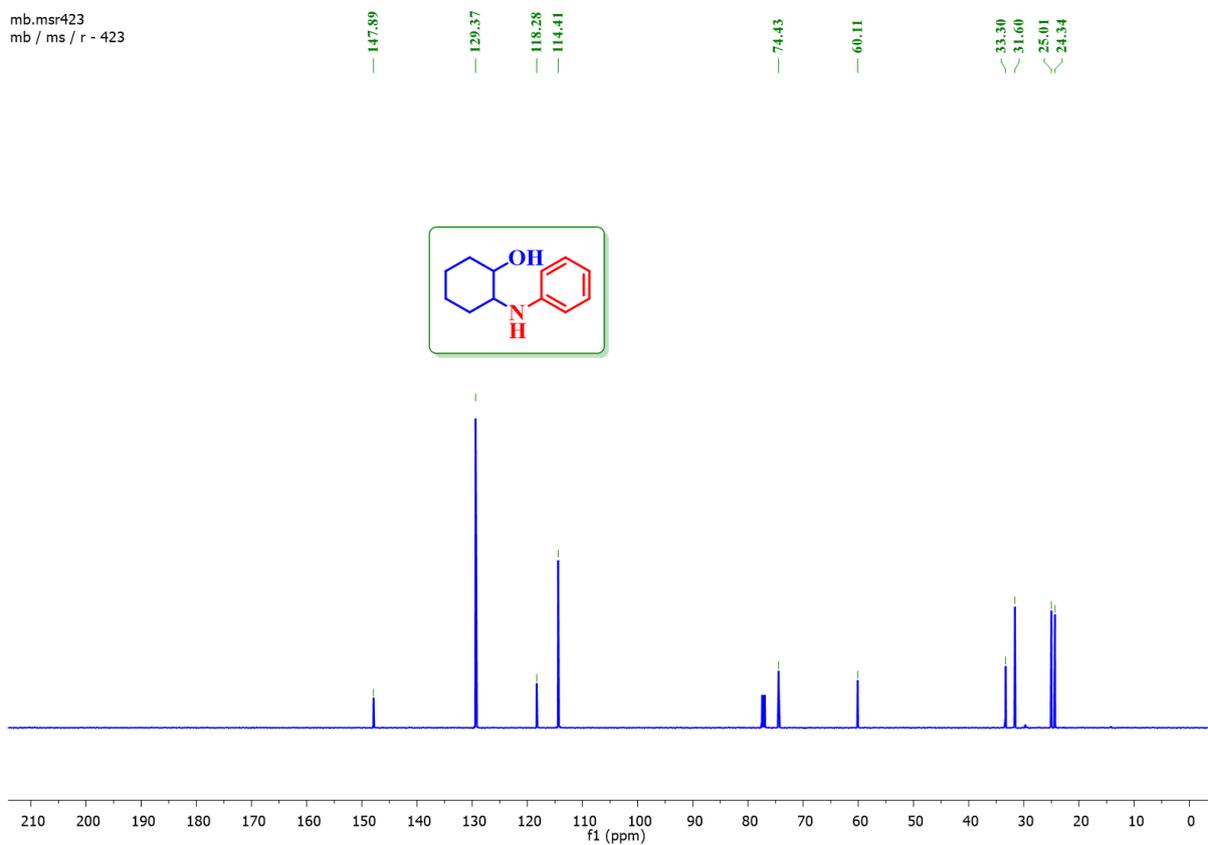


Figure S23  $^{13}\text{C}$  NMR spectrum of **3a**

mb.msr424  
mb.msr427-1h-400mhz

7.06  
7.05  
7.03  
7.02  
6.57  
6.56  
6.54  
6.53

3.30  
3.29  
3.27  
3.26  
3.25  
3.24  
3.03  
3.02  
3.00  
2.99  
2.97  
2.96

2.04  
2.02  
2.01  
1.99  
1.71  
1.70  
1.69  
1.65  
1.64  
1.64  
1.25  
1.24  
1.22  
1.20  
1.19

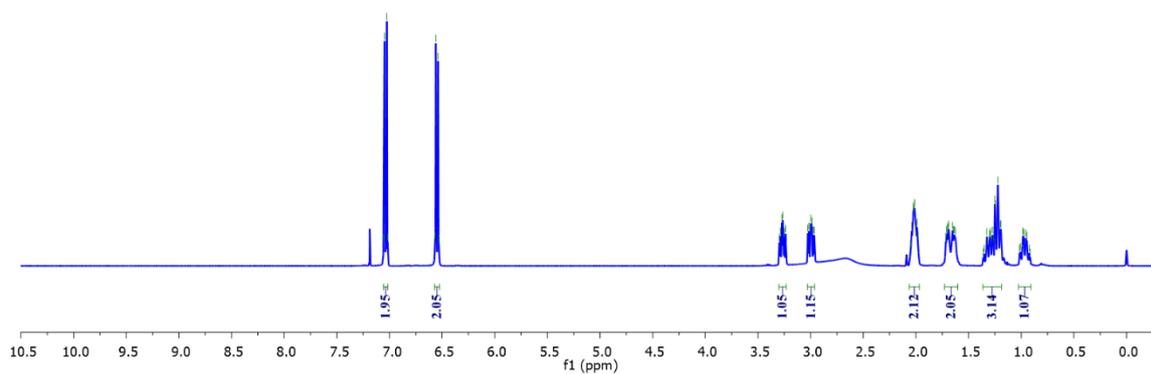


Figure S23  $^1\text{H}$  NMR spectrum of **3b**

mb.msr424  
mb.msr427-13c-400mhz

146.44  
129.14  
122.81  
115.43

74.51  
60.35

33.28  
31.51  
24.93  
24.24

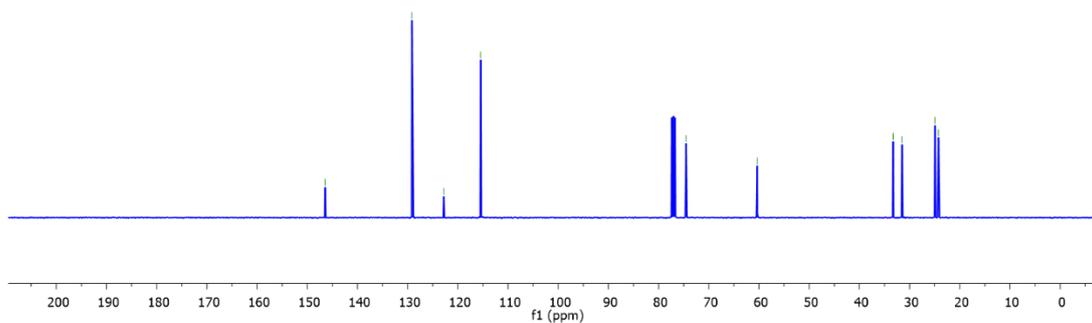
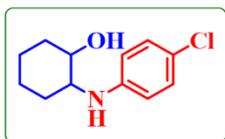
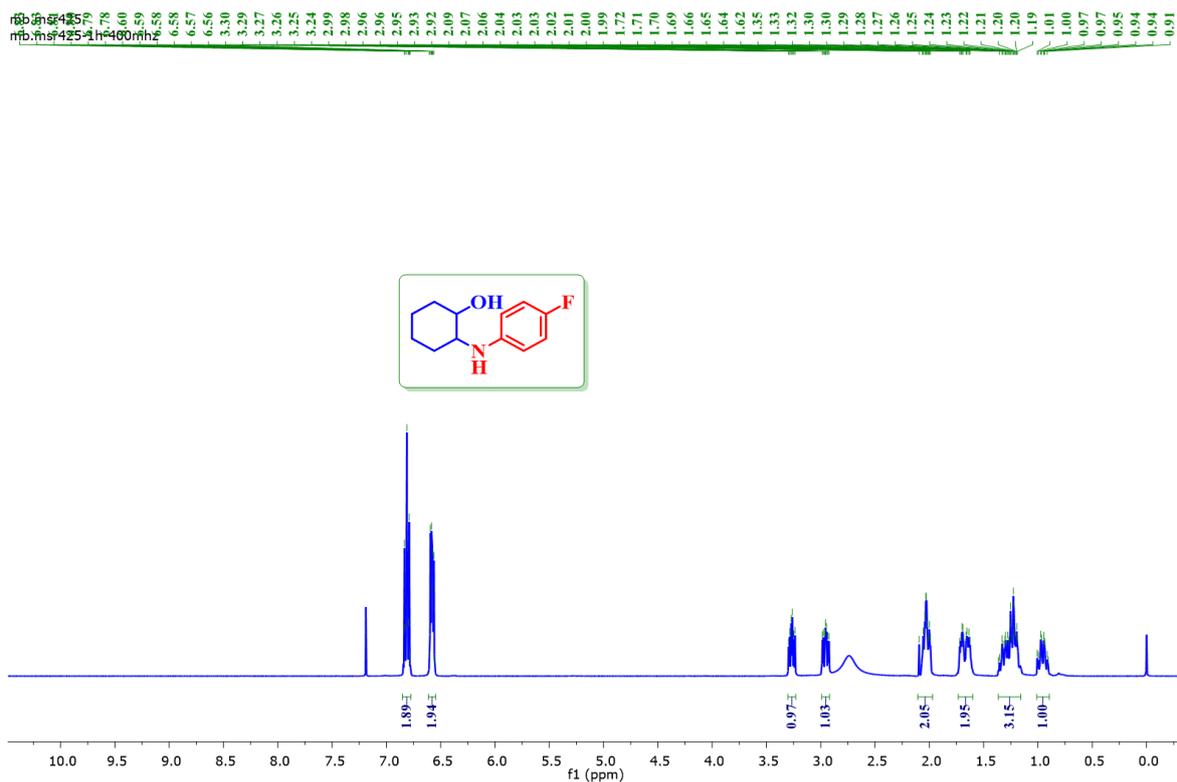
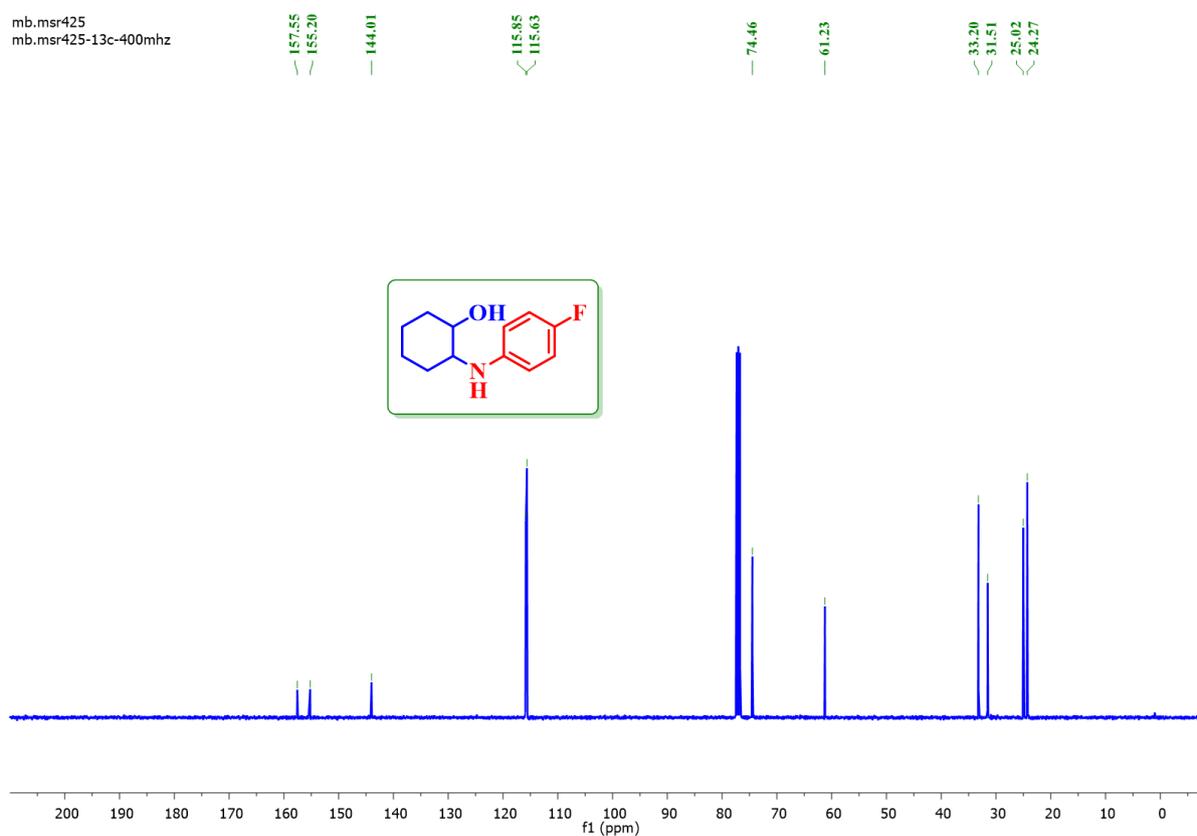


Figure S24  $^{13}\text{C}$  NMR spectrum of **3b**



mb.msr425  
 mb.msr425-13c-400mhz



mb.msr428  
mb / ms / r - 428

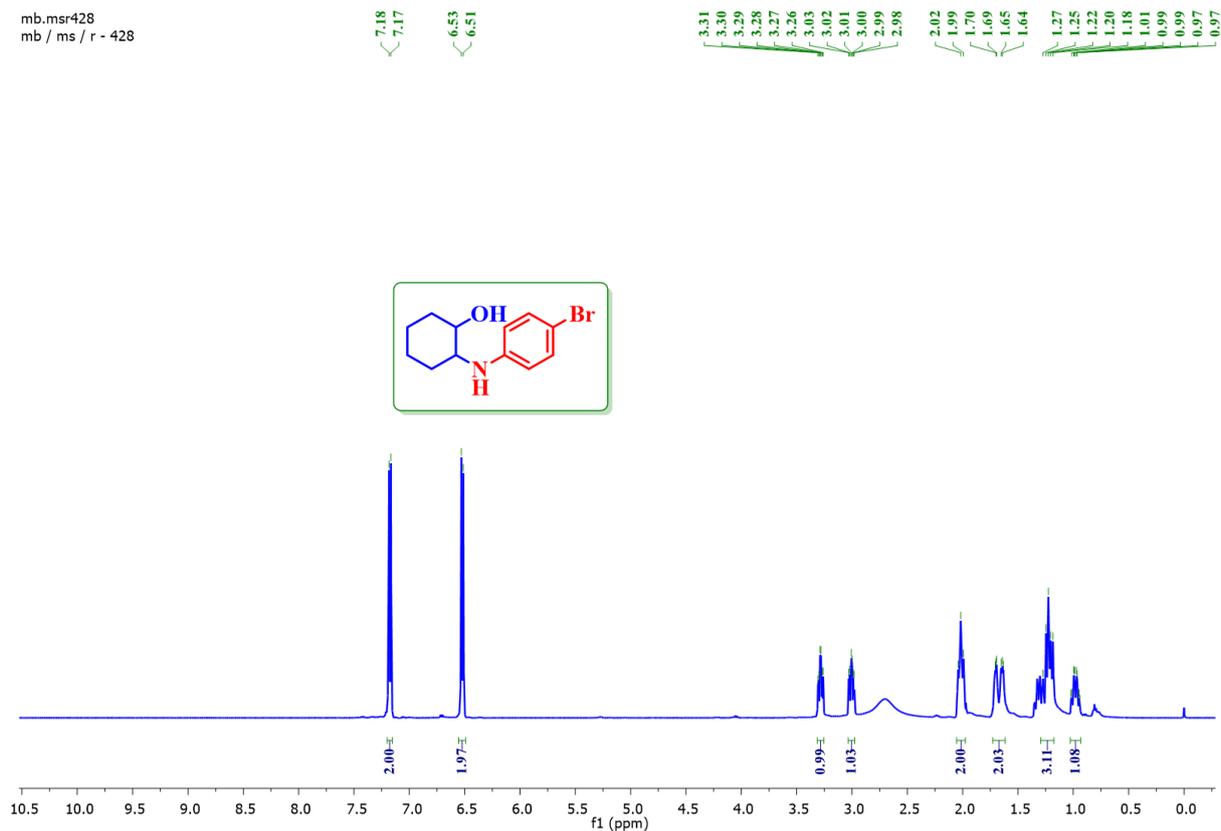


Figure S27  $^1\text{H}$  NMR spectrum of **3d**

mb.msr428  
mb / ms / r - 428

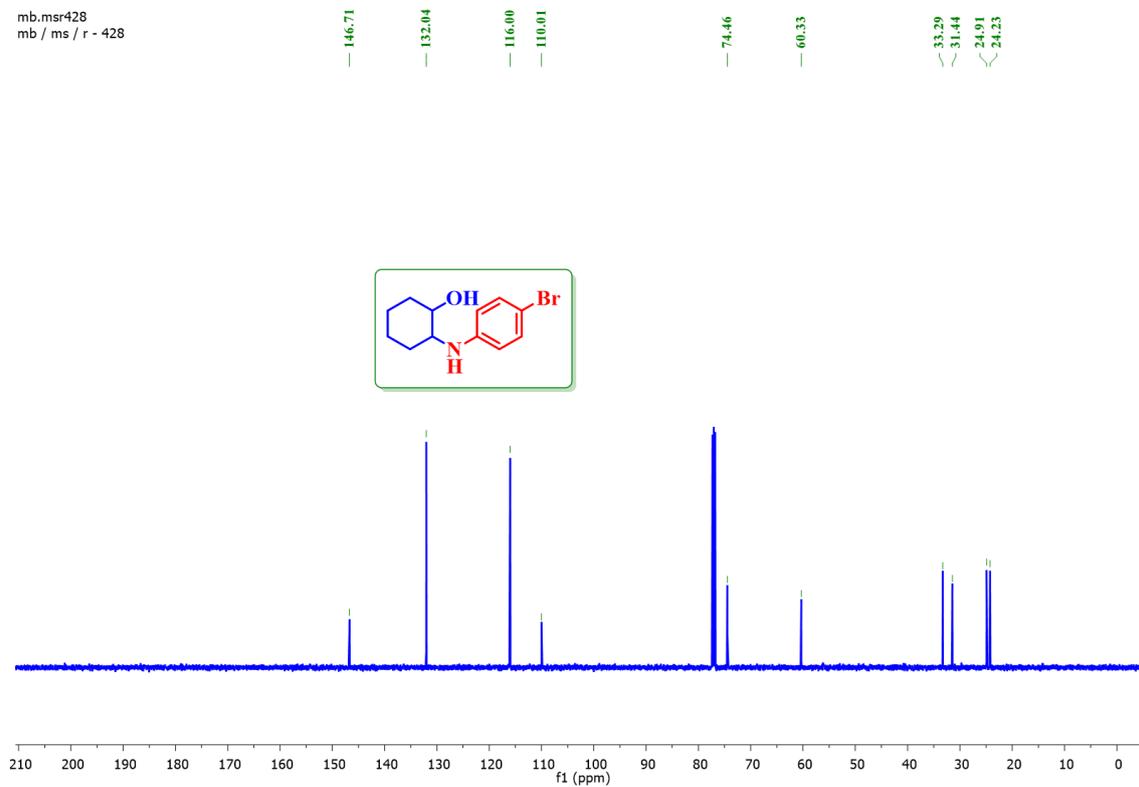


Figure S28  $^{13}\text{C}$  NMR spectrum of **3d**

mb.msr431pi  
mb / ms / r - 431 pi

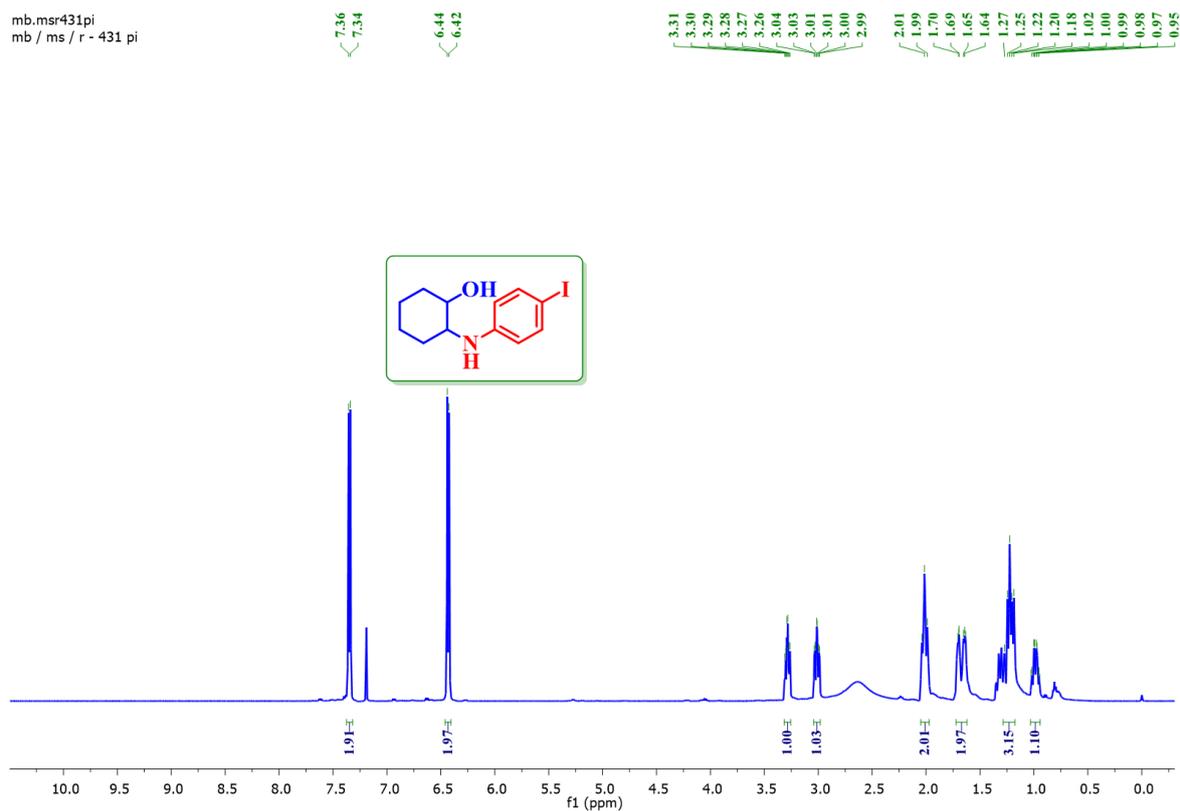


Figure S29  $^1\text{H}$  NMR spectrum of **3e**

mb.msr431pi  
mb / ms / r - 431 pi

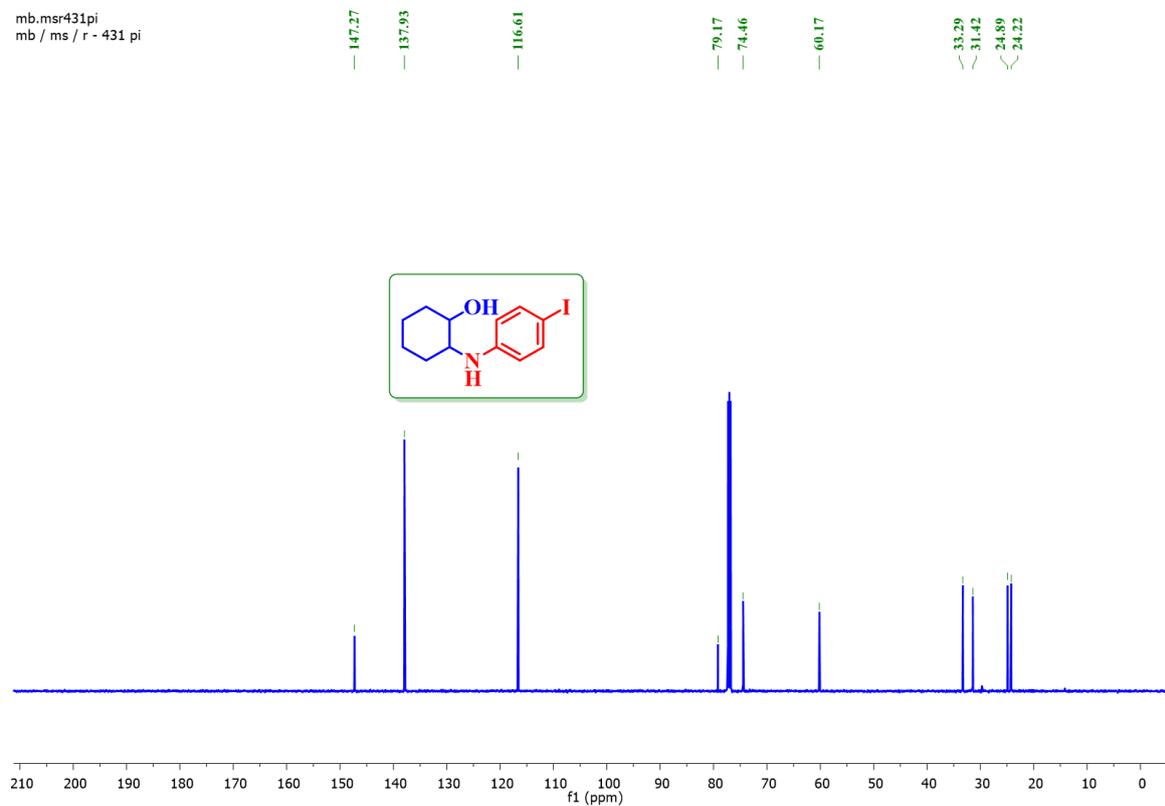


Figure S30  $^{13}\text{C}$  NMR spectrum of **3e**

mb.msr432  
mb / ms / r - 432

7.29  
7.27  
7.03  
7.02  
7.00  
6.69  
6.45  
6.44  
6.42  
3.28  
3.27  
3.26  
3.05  
3.03  
3.01  
1.95  
1.92  
1.60  
1.55  
1.22  
1.17  
1.15  
1.01  
0.98  
0.97  
0.96

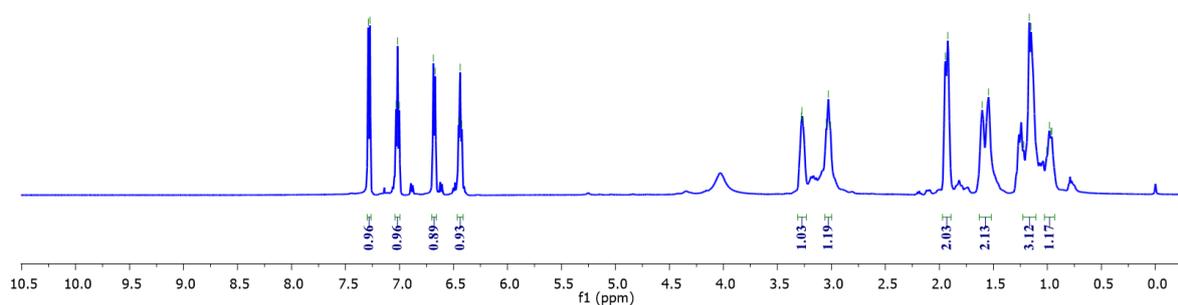
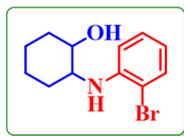


Figure S31  $^1\text{H}$  NMR spectrum of **3f**

mb.msr432  
mb / ms / r - 432

144.90  
132.56  
128.54  
118.39  
112.95  
110.92  
74.19  
59.78  
33.36  
31.58  
24.82  
24.23

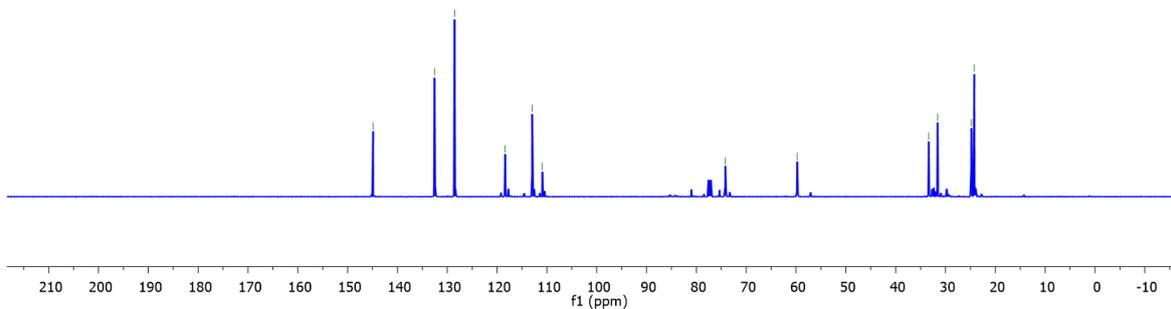
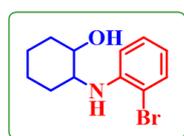


Figure S32  $^{13}\text{C}$  NMR spectrum of **3f**

mb.msr441  
mb / ms / r - 441

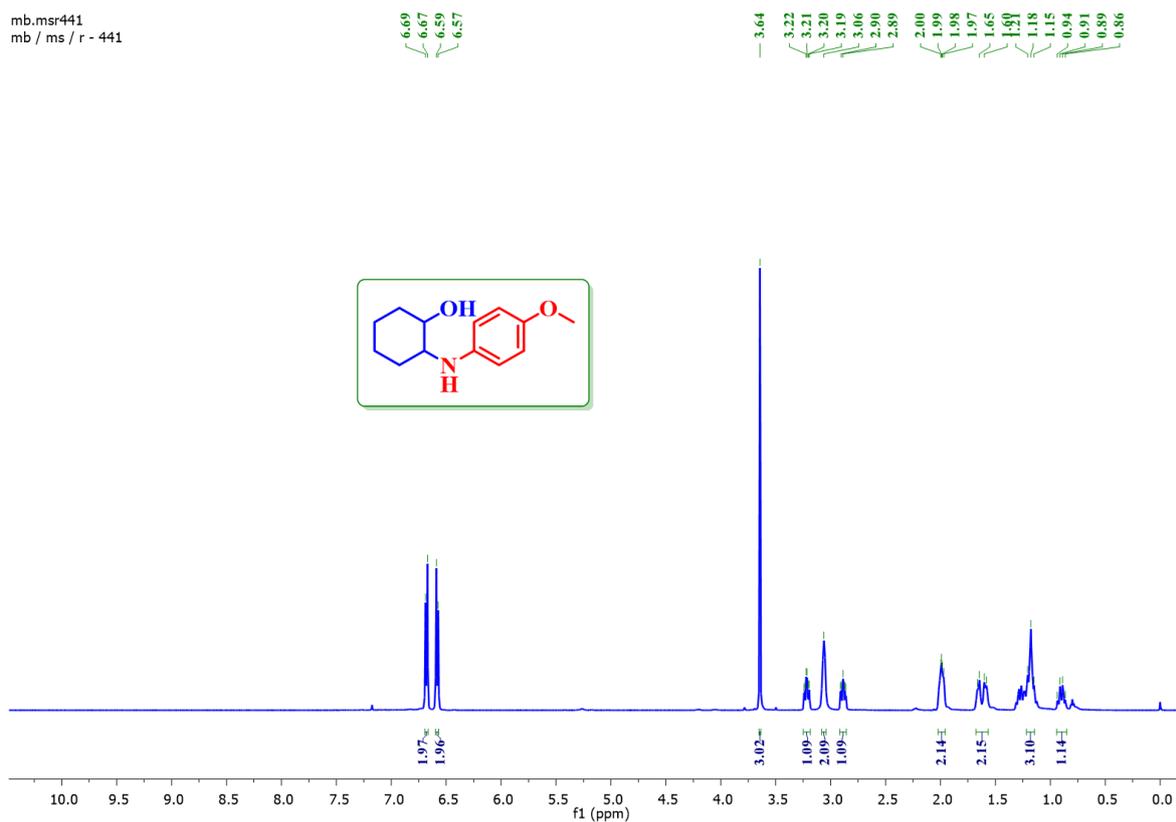


Figure S33  $^1\text{H}$  NMR spectrum of **3g**

mb.msr441  
mb / ms / r - 441

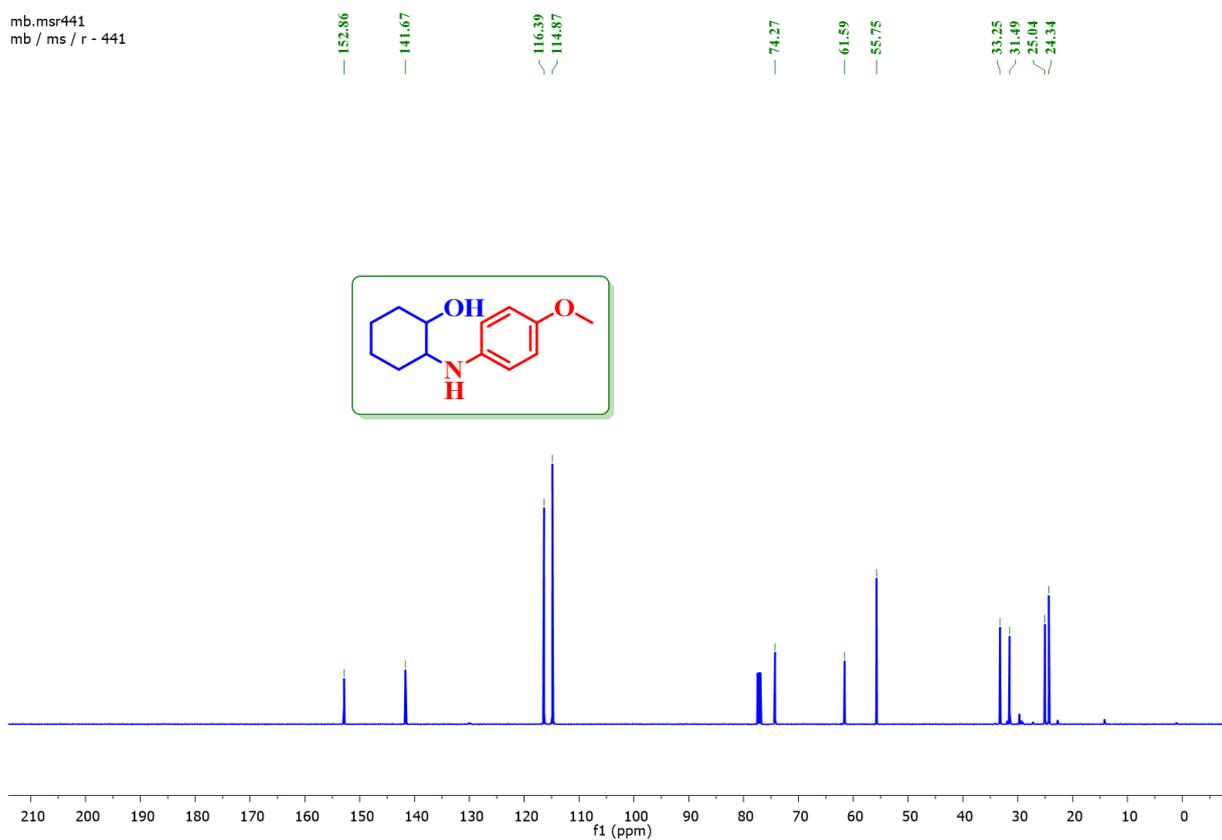


Figure S34  $^{13}\text{C}$  NMR spectrum of **3g**

mb.msr427  
mb.msr427-1h-400mhz

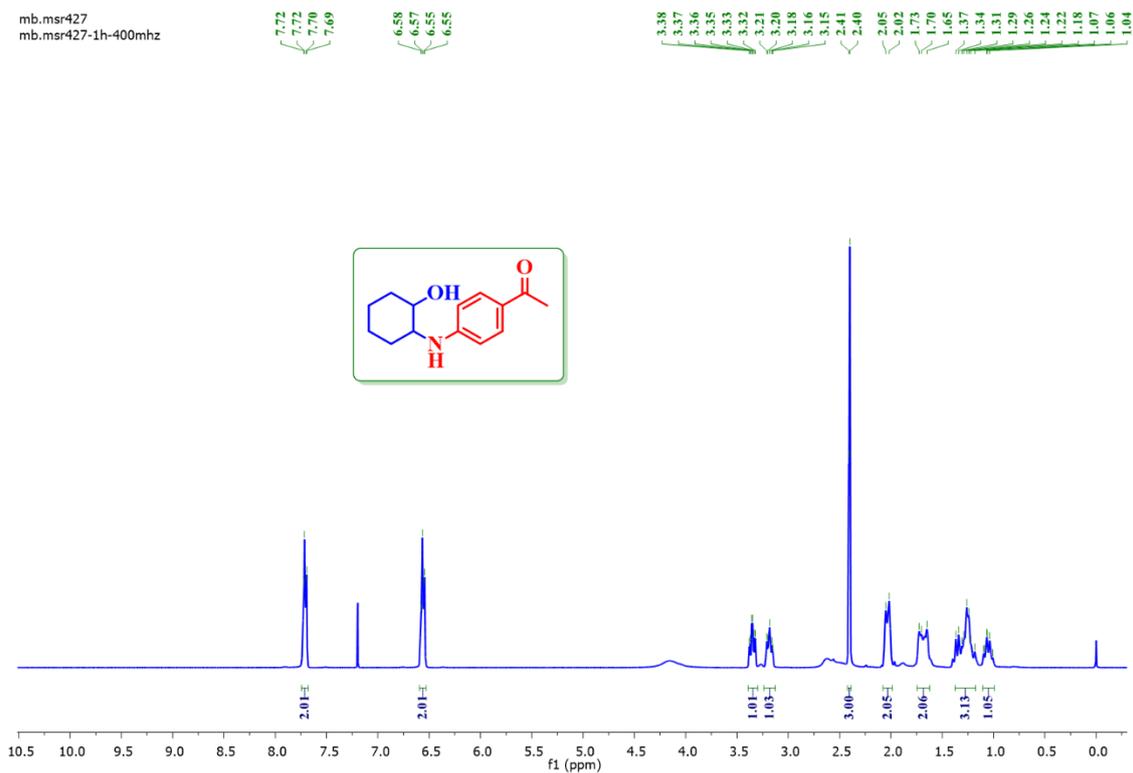


Figure S35  $^1\text{H}$  NMR spectrum of 3h

mb.msr427  
mb.msr427-13c-400mhz

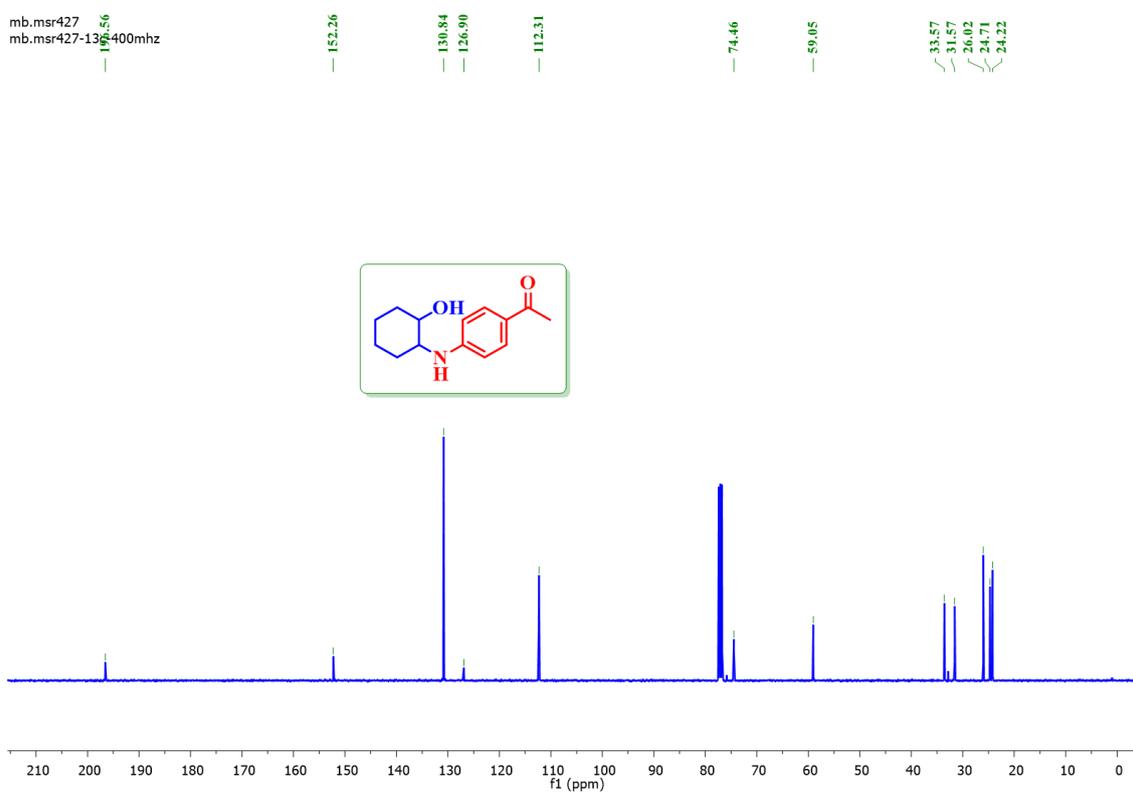


Figure S36  $^{13}\text{C}$  NMR spectrum of 3h

mb.msr426  
mb.msr426-1h-400mhz

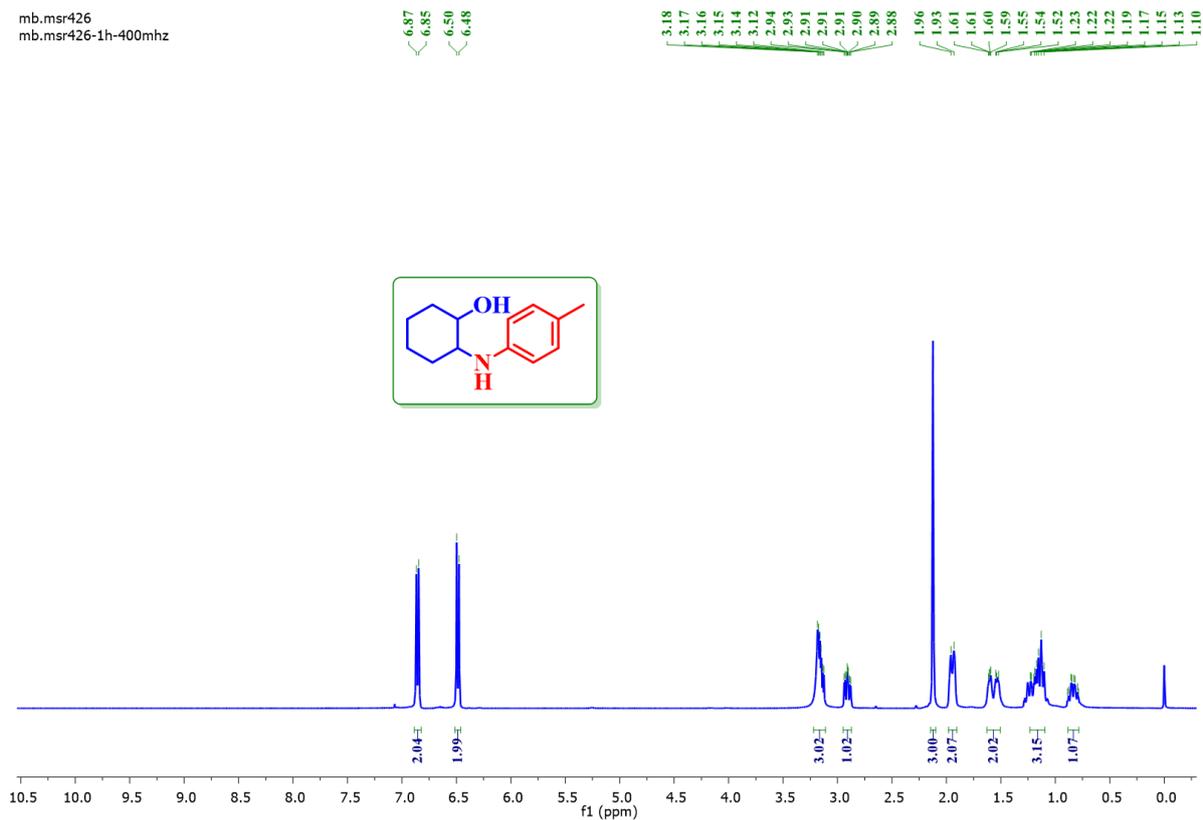


Figure S37 <sup>1</sup>H NMR spectrum of **3i**

mb.msr426  
mb.msr426-13c-400mhz

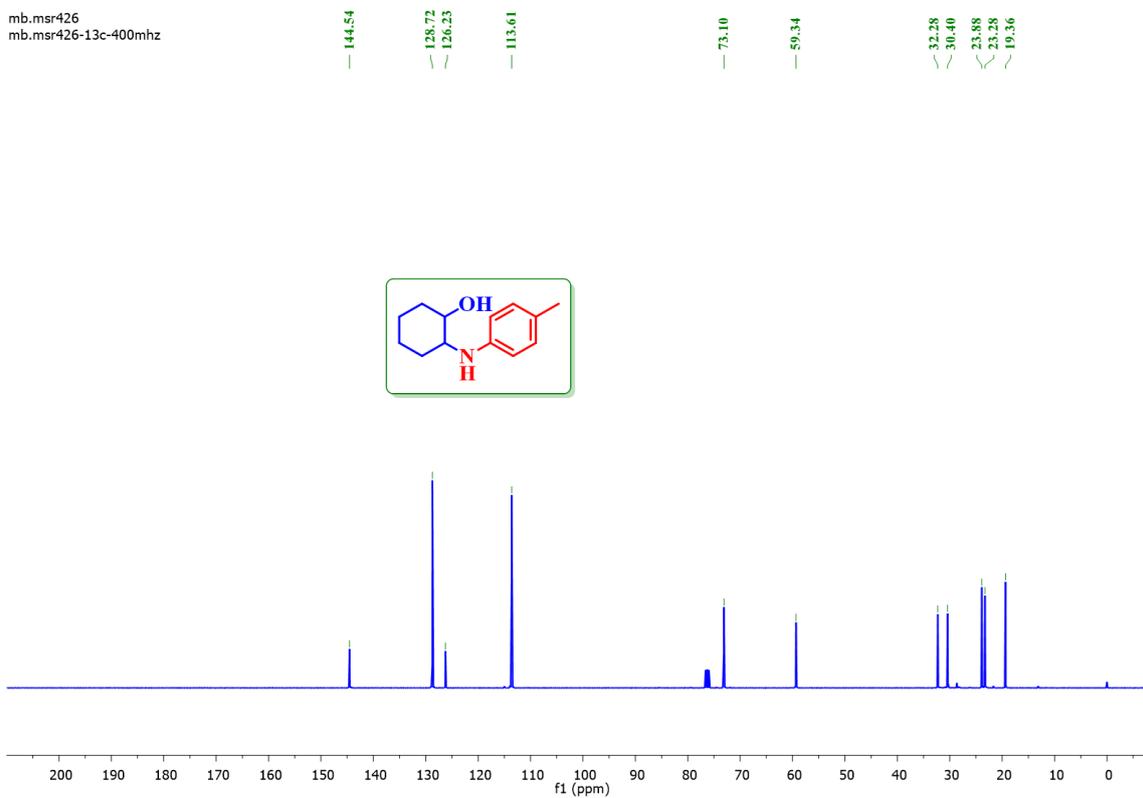


Figure S38 <sup>13</sup>C NMR spectrum of **3i**

mb.msr429  
mb.msr429-1h-400mhz

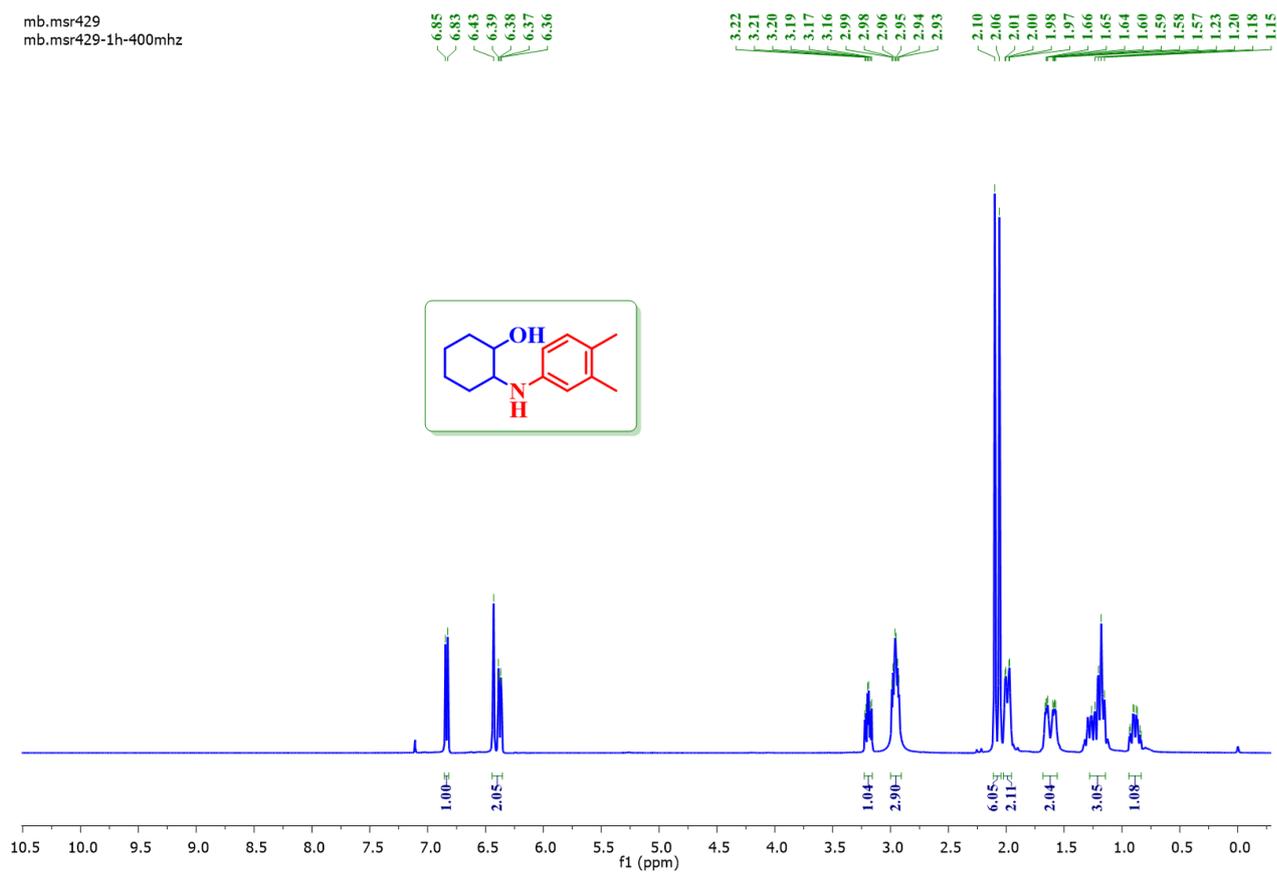


Figure S39  $^1\text{H}$  NMR spectrum of **3j**

mb.msr429  
mb.msr429-13c-400mhz

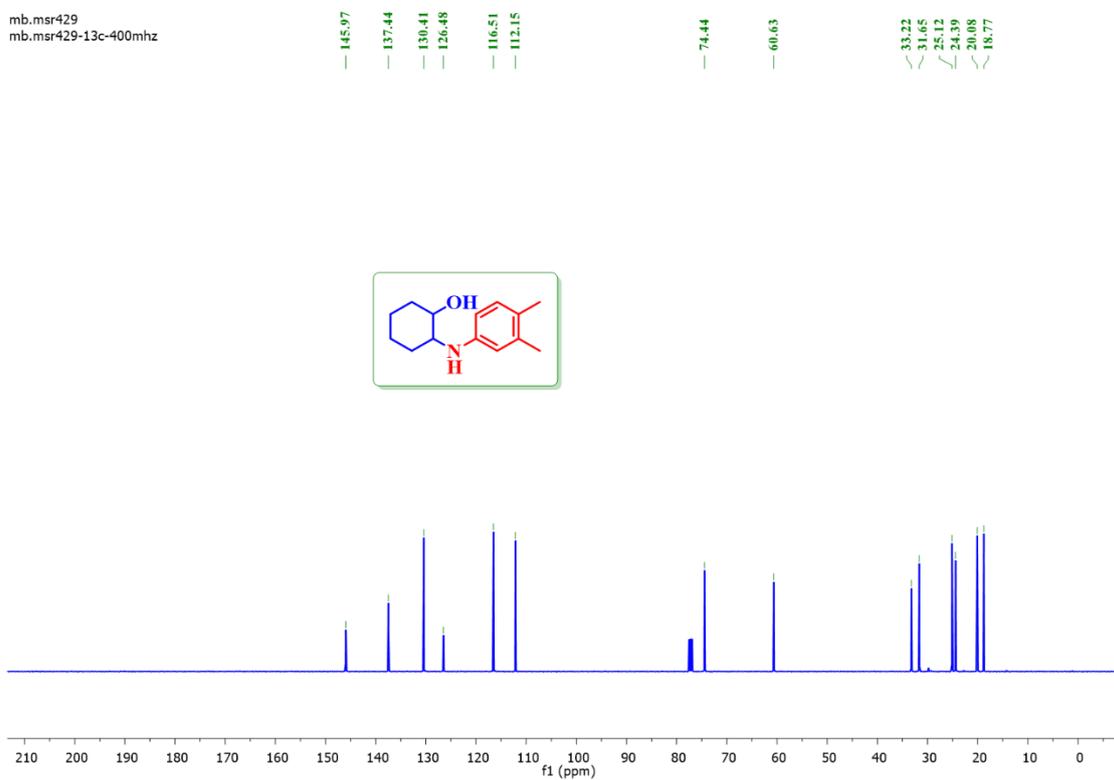
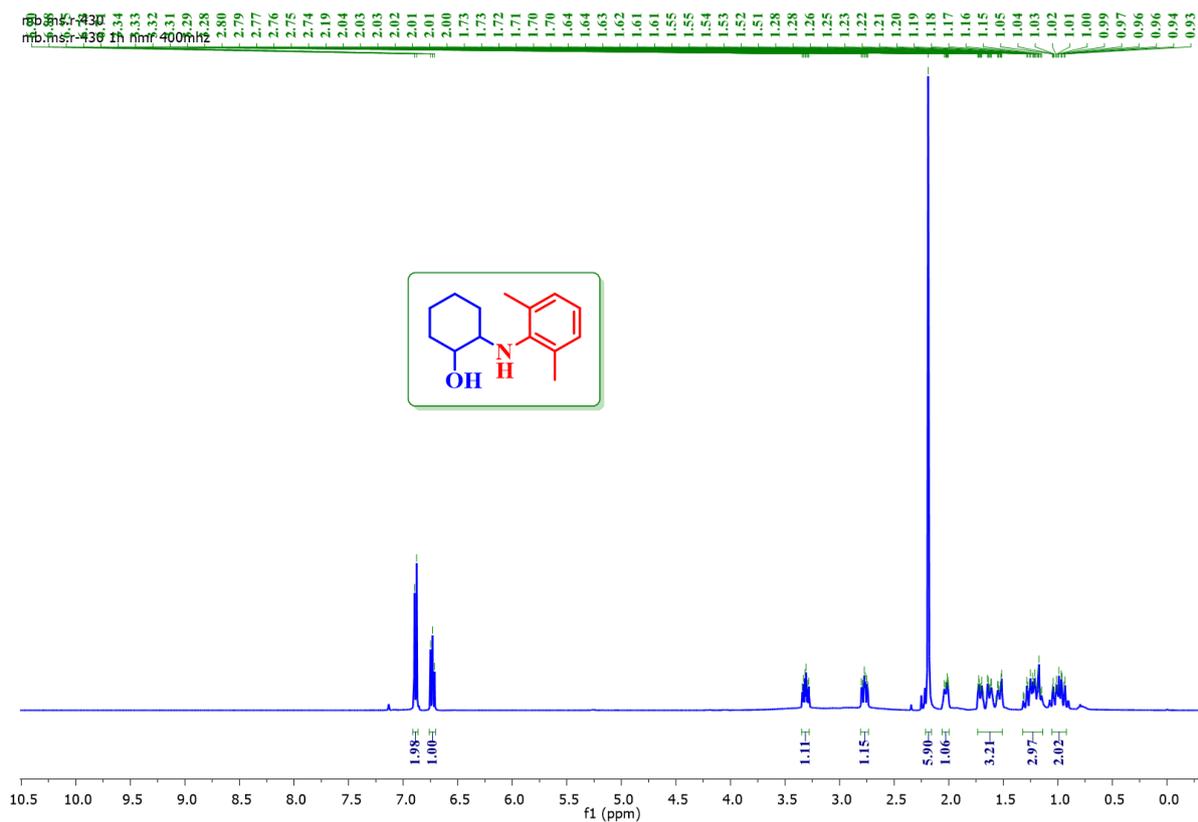
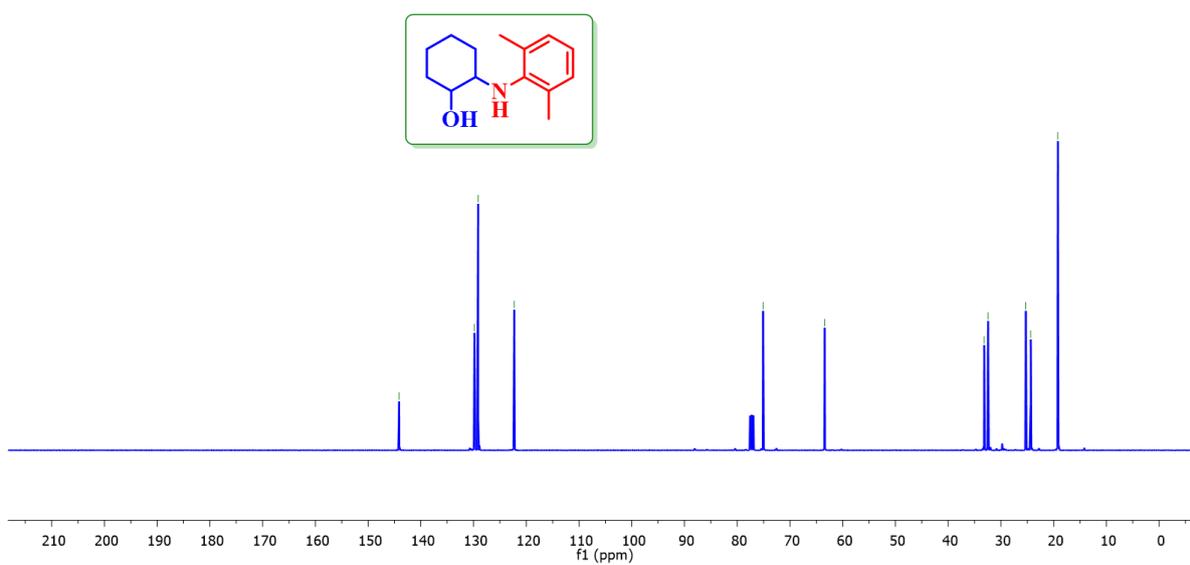


Figure S40  $^{13}\text{C}$  NMR spectrum of **3j**



mb.ms.r-430  
mb.ms.r-430 13c nmr 400mhz

144.10  
129.83  
129.13  
122.29  
75.09  
63.41  
33.18  
32.45  
25.27  
24.35  
19.21



mb.msr456  
mb / ms / r - 456

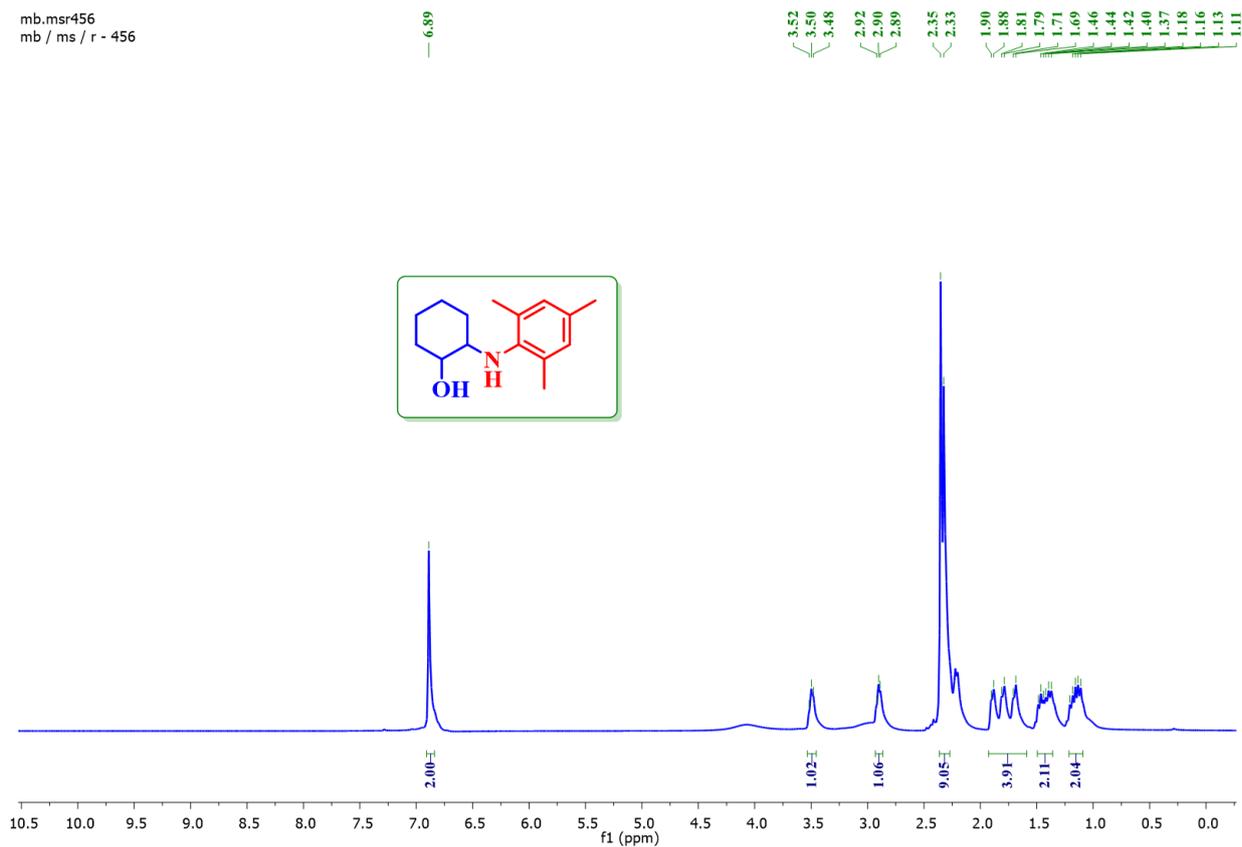


Figure S43  $^1\text{H}$  NMR spectrum of **31**

mb.msr456  
mb / ms / r - 456

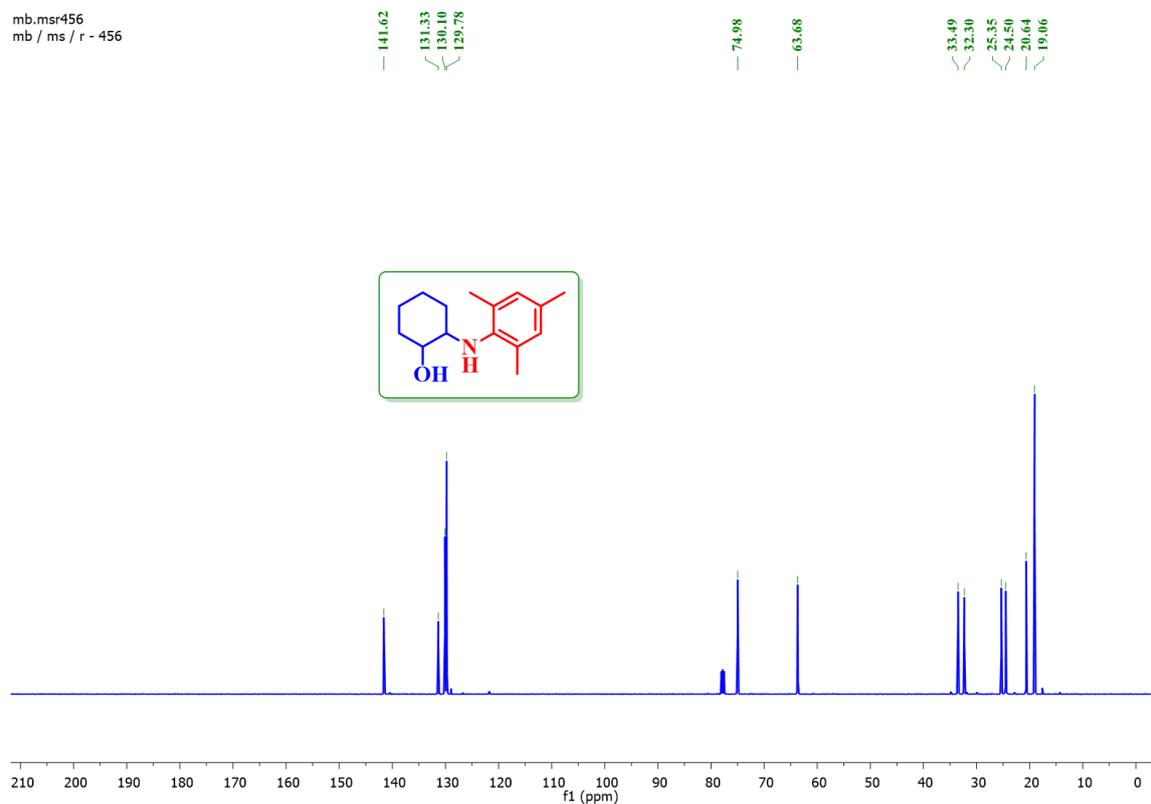


Figure S44  $^{13}\text{C}$  NMR spectrum of **31**

mb.msr442  
mb / ms / r - 442

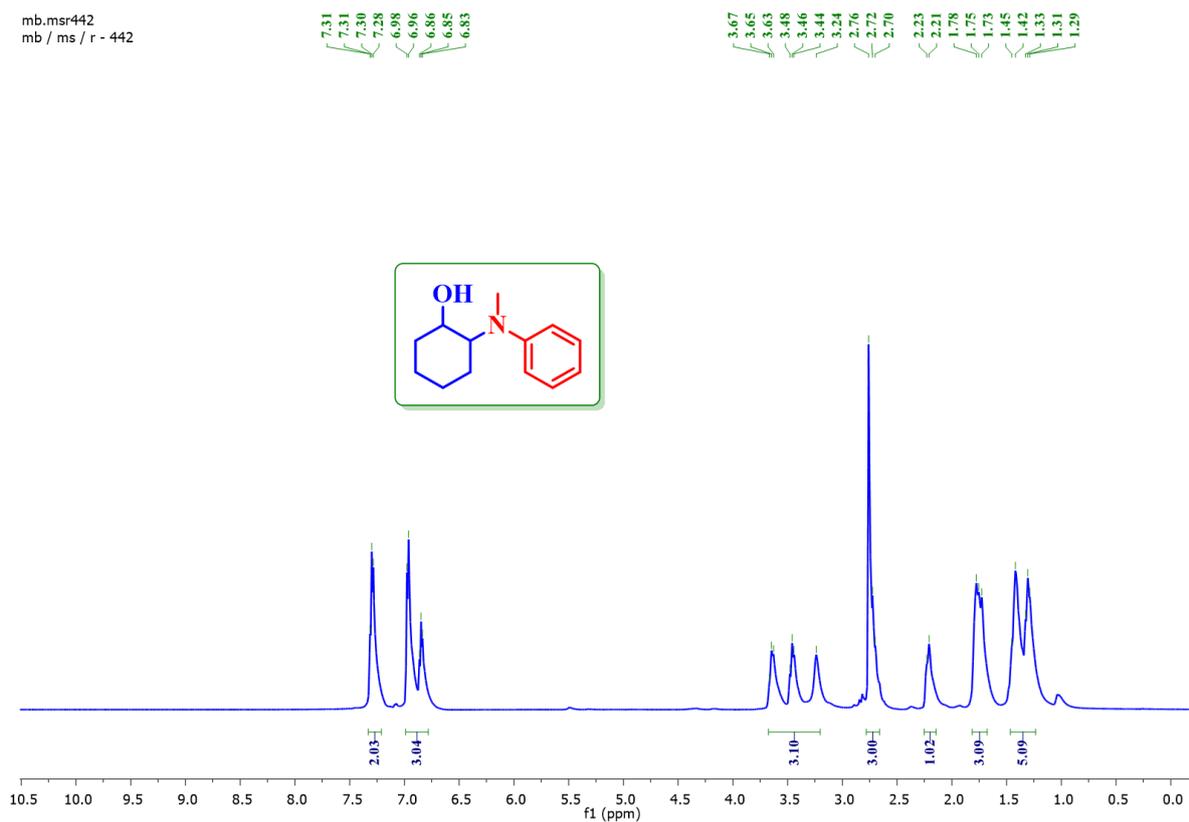


Figure S45 <sup>1</sup>H NMR spectrum of **3m**

mb.msr442  
mb / ms / r - 442

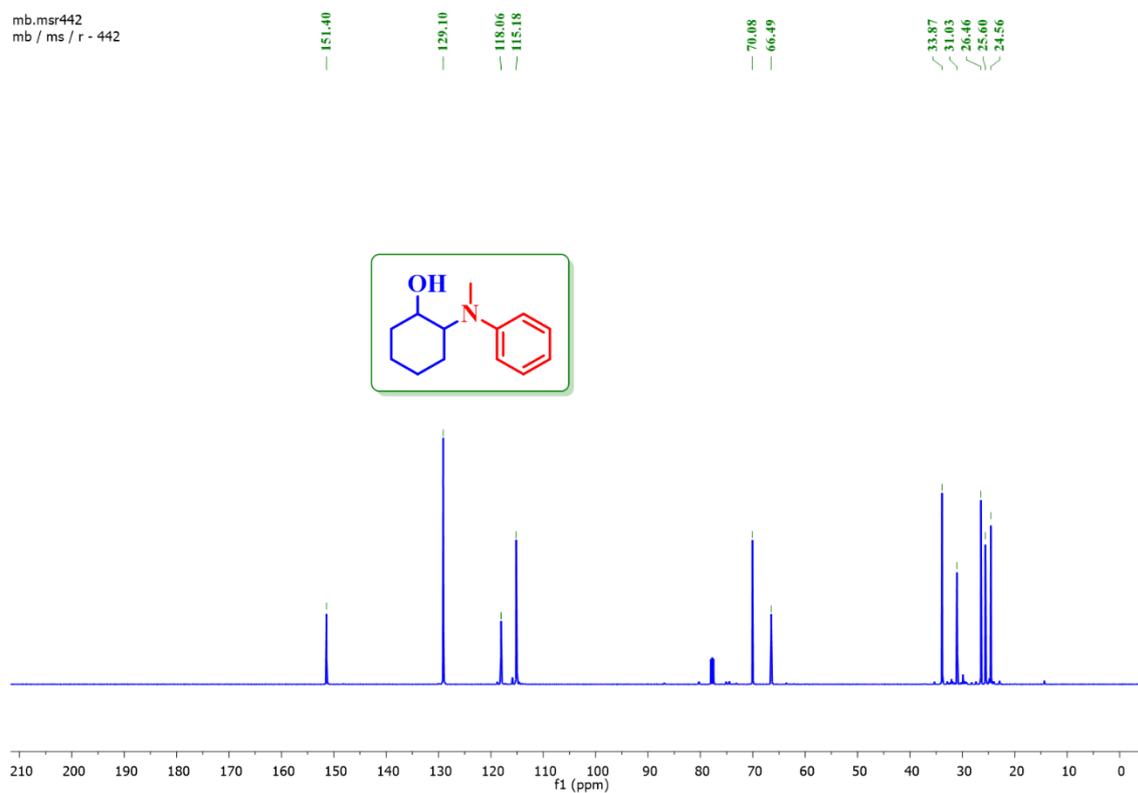


Figure S46 <sup>13</sup>C NMR spectrum of **3m**

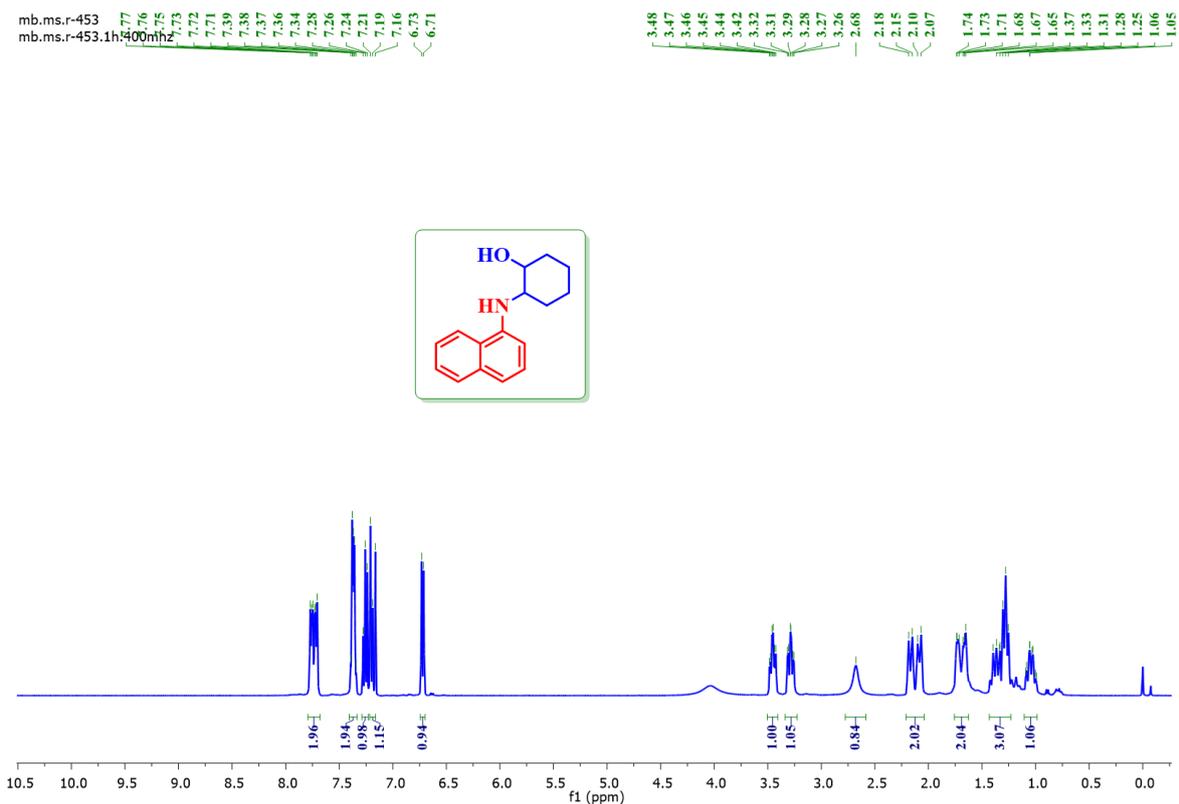


Figure S47  $^1\text{H}$  NMR spectrum of **3n**

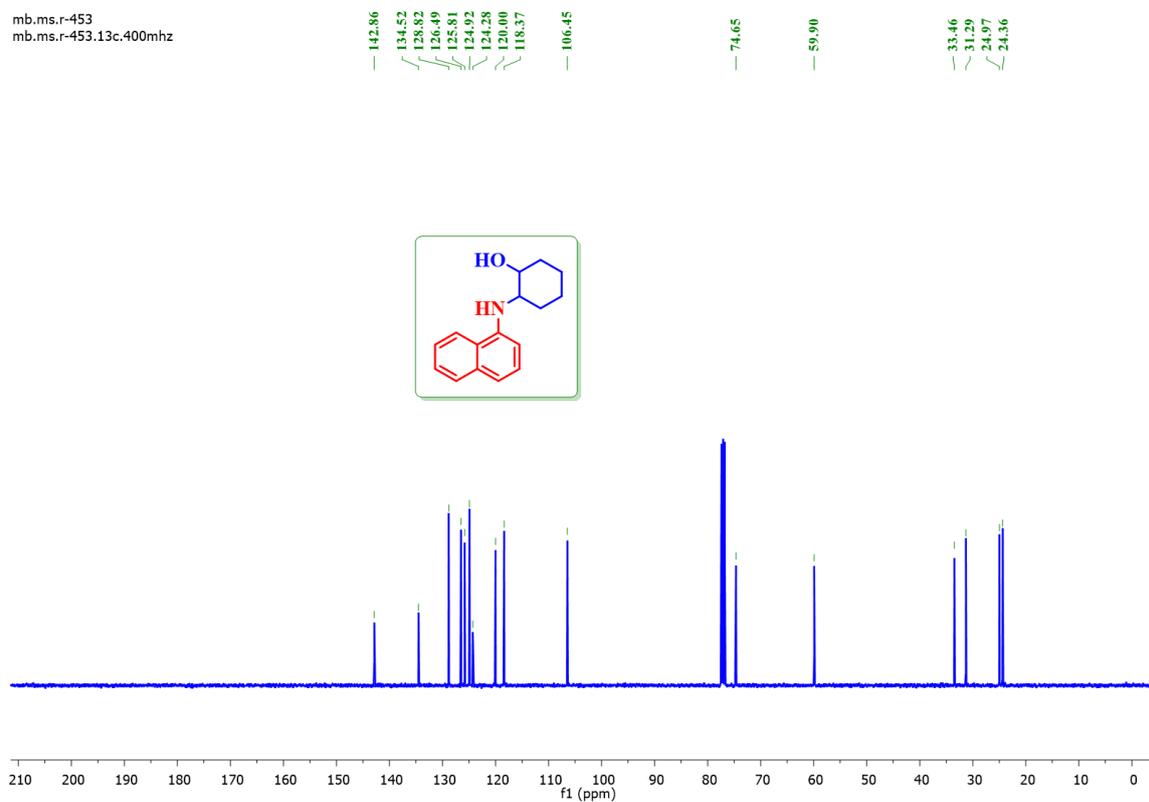


Figure S48  $^{13}\text{C}$  NMR spectrum of **3n**

mb.msc439  
mb / ms / c - 439

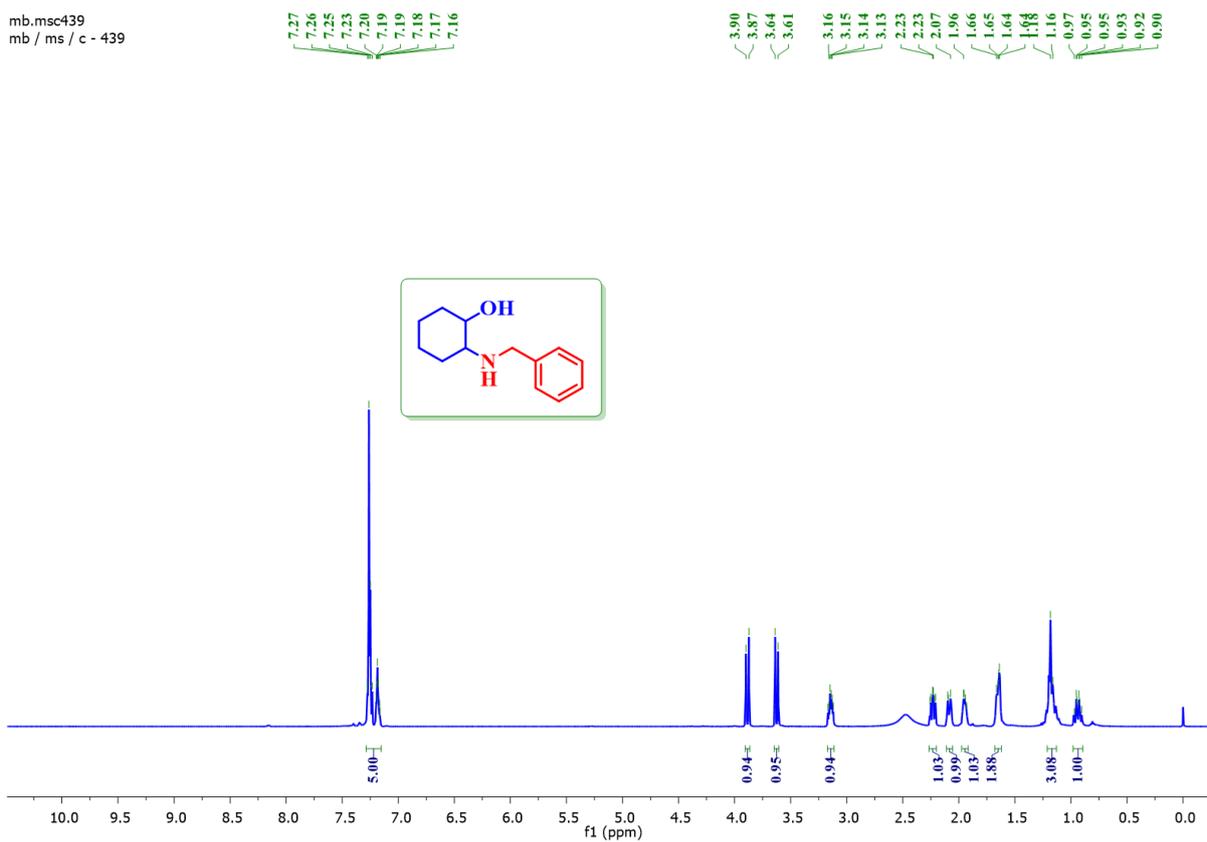


Figure S49  $^1\text{H}$  NMR spectrum of **3o**

mb.msc439  
mb / ms / c - 439

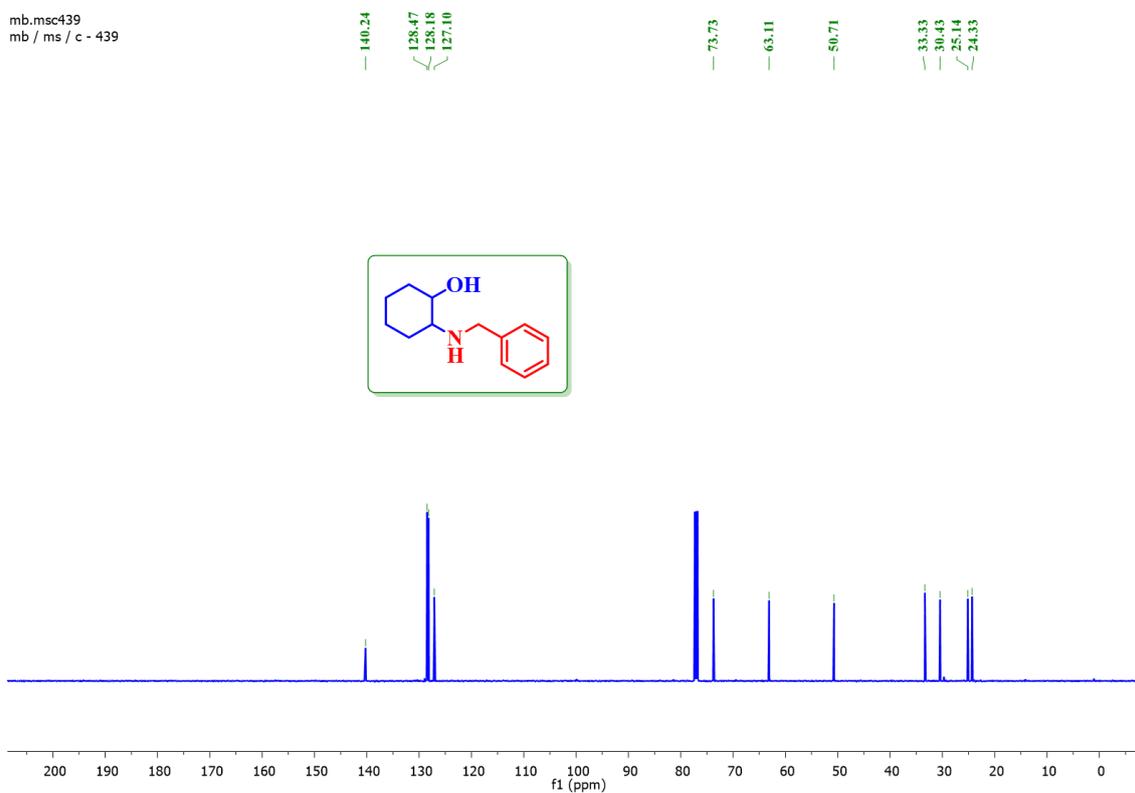


Figure S50  $^{13}\text{C}$  NMR spectrum of **3o**

mb.msr433a  
mb / ms / r - 433a

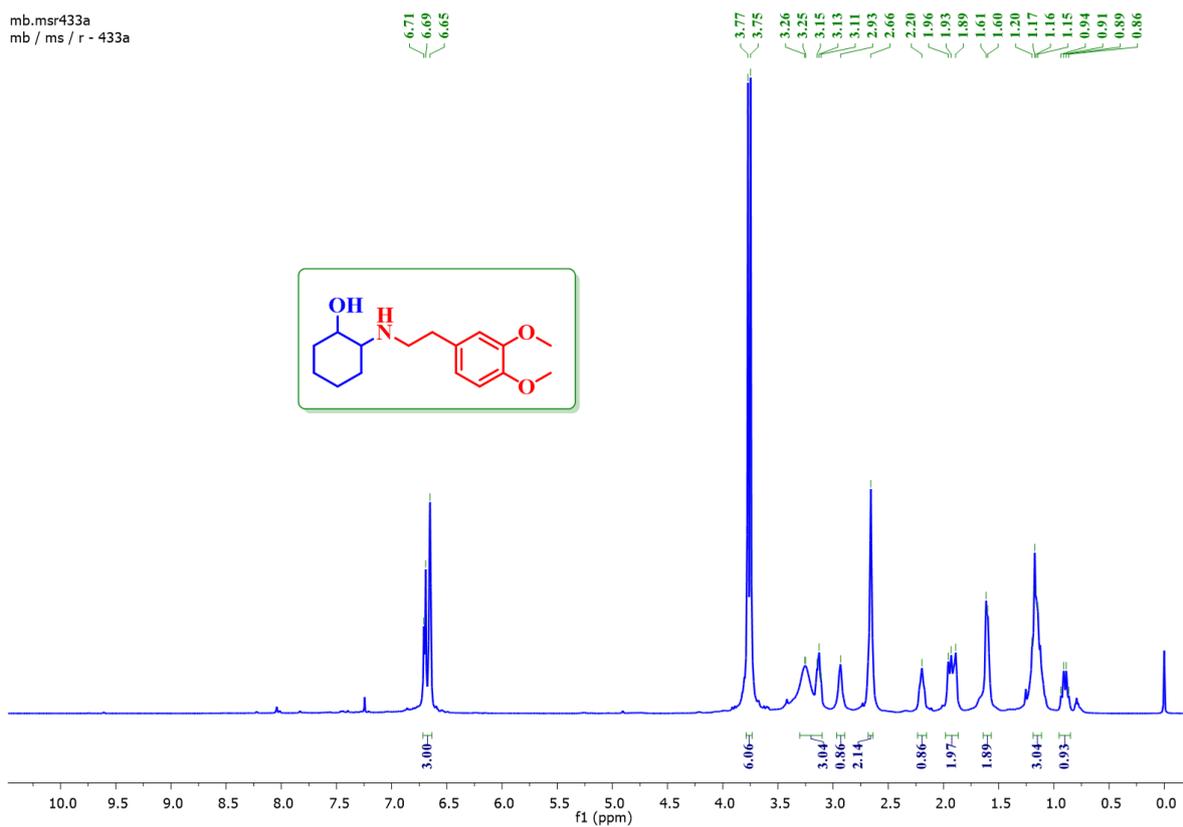


Figure S51  $^1\text{H}$  NMR spectrum of **3p**

mb.msr433a  
mb / ms / r - 433a

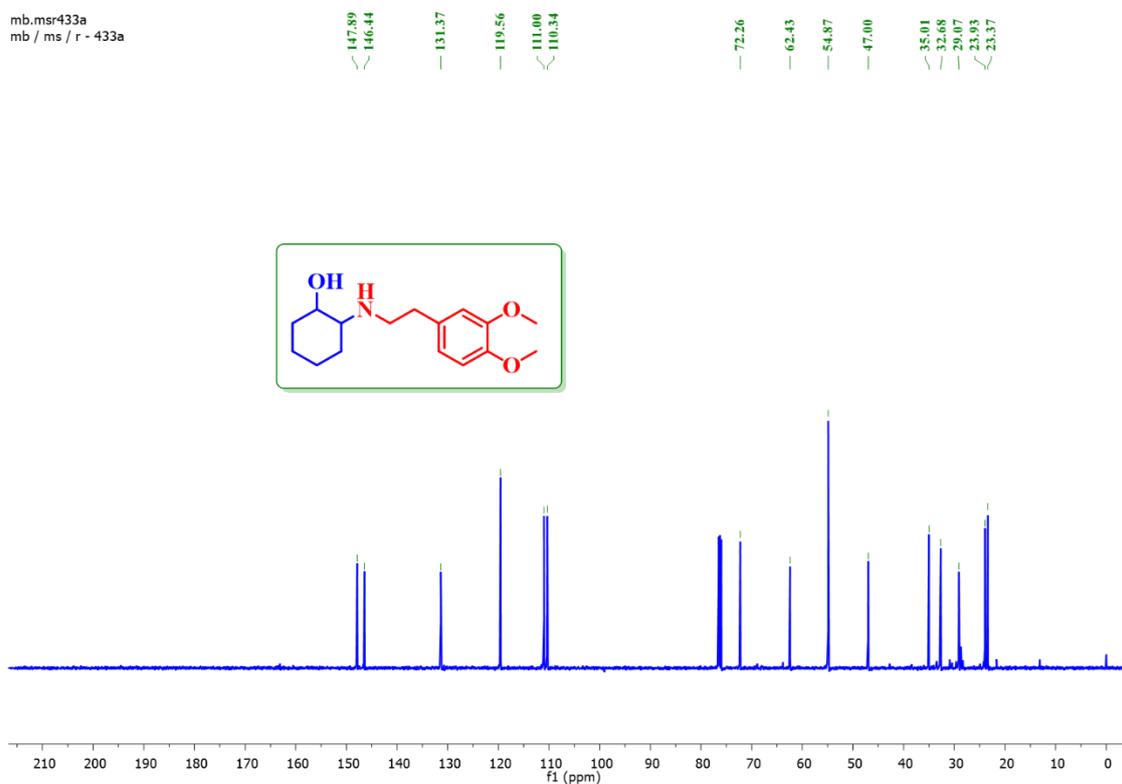
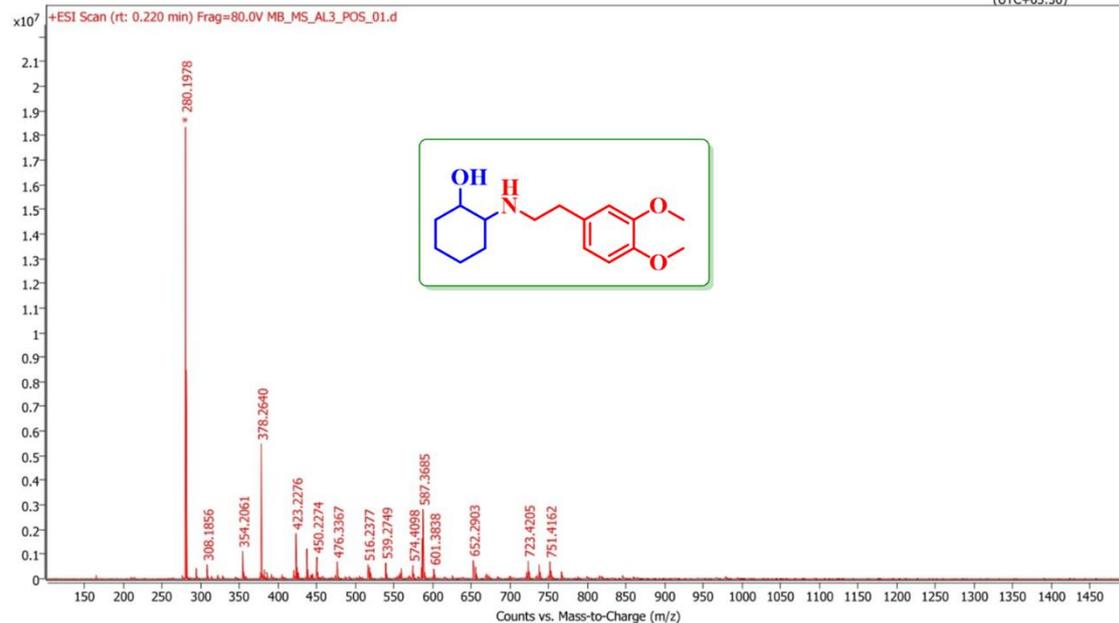


Figure S52  $^{13}\text{C}$  NMR spectrum of **3p**

# Spectrum Plot Report



Name: MB\_MS\_AL3 Rack Pos.: Plate Pos. Instrument: IITKGP Operator: Success  
 Inj. Vol. (ul): 2 Method (Acq): MS SCAN POS\_01.m Comment: Acq. Time (Local): 7/23/2025 4:53:19 PM (UTC+05:30)  
 Data File: MB\_MS\_AL3\_POS\_01.d



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Generated at 4:58 PM on 7/24/2025

Figure S53 HRMS spectrum of 3p

mb.ms.r-452  
 mb.ms.r-452.1h.400mhz

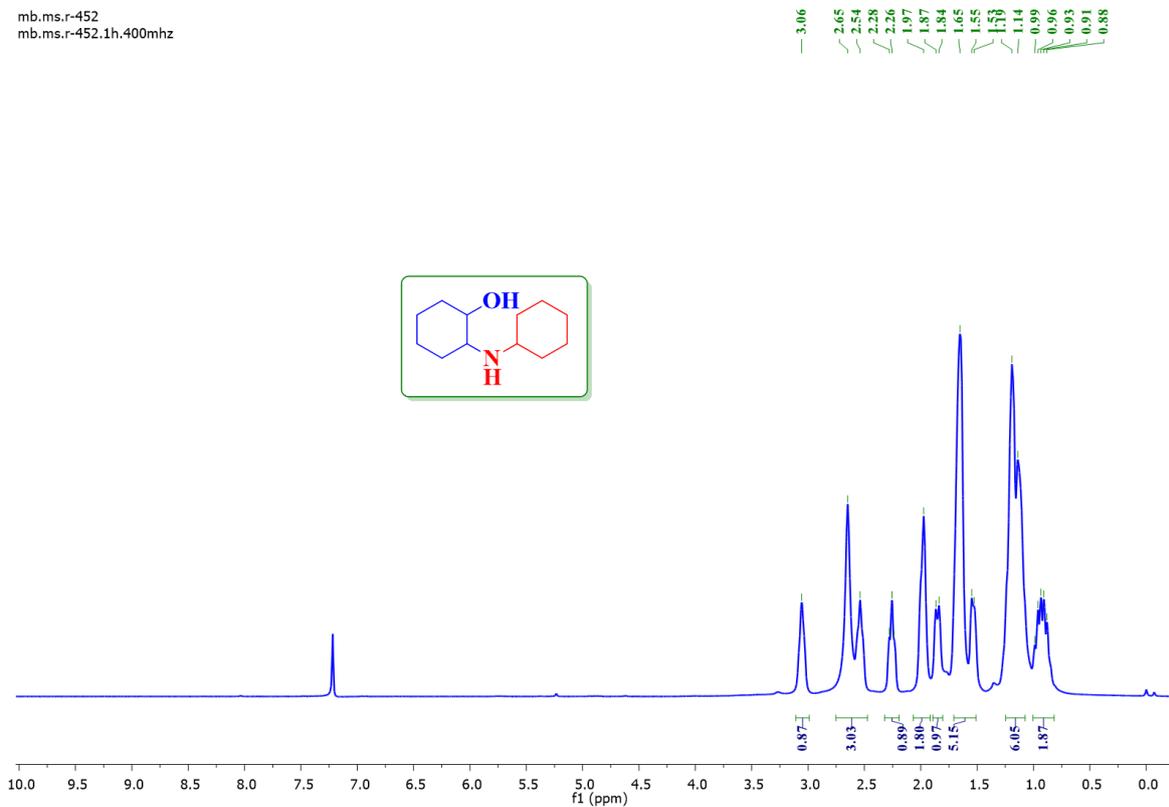


Figure S54 <sup>1</sup>H NMR spectrum of 3q

mb.ms.r-452  
mb.ms.r-452.13c.400mhz

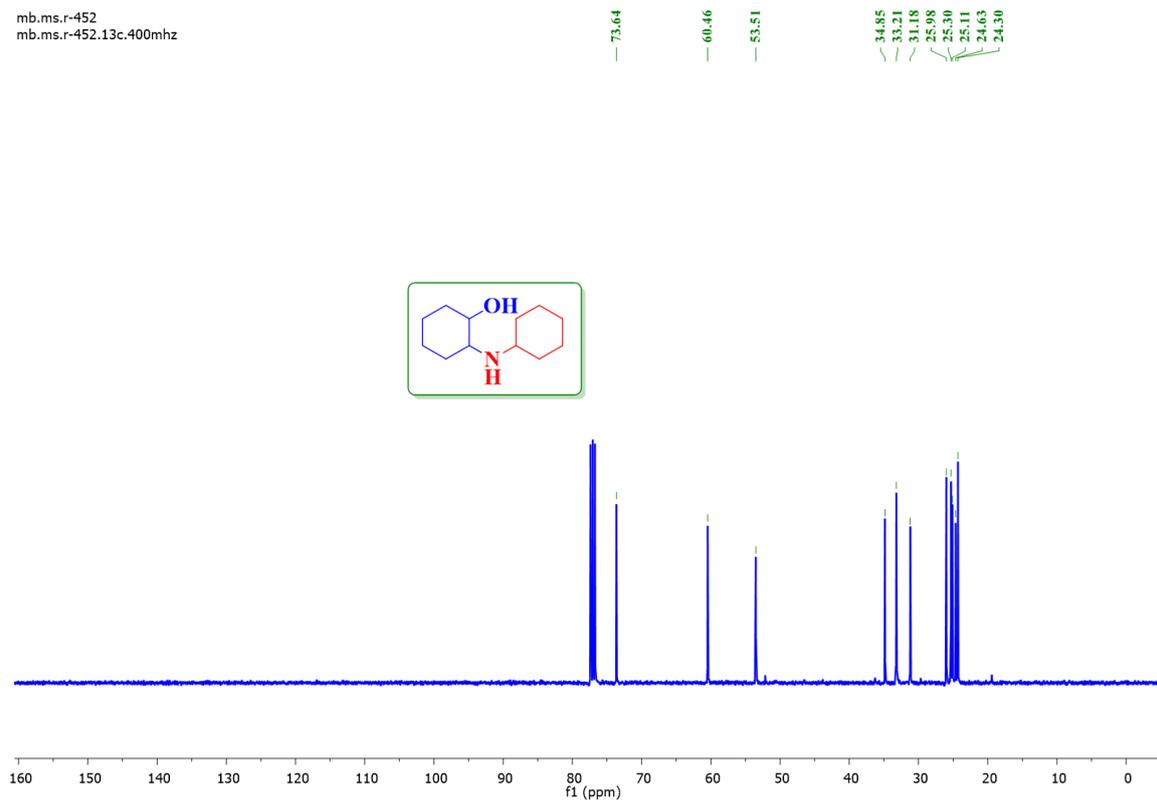


Figure S55 <sup>13</sup>C NMR spectrum of 3q

mb.msr462  
mb / ms / r - 462

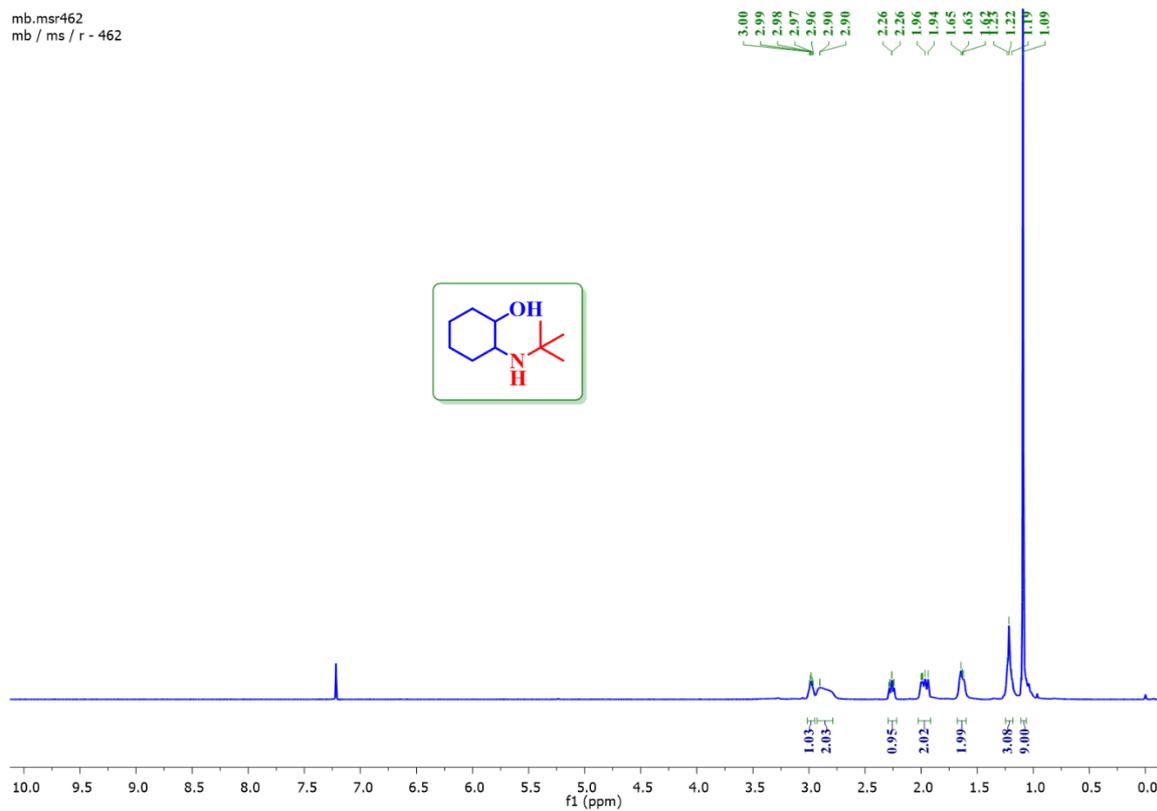


Figure S56 <sup>1</sup>H NMR spectrum of 3r

mb.msr462  
mb / ms / r - 462

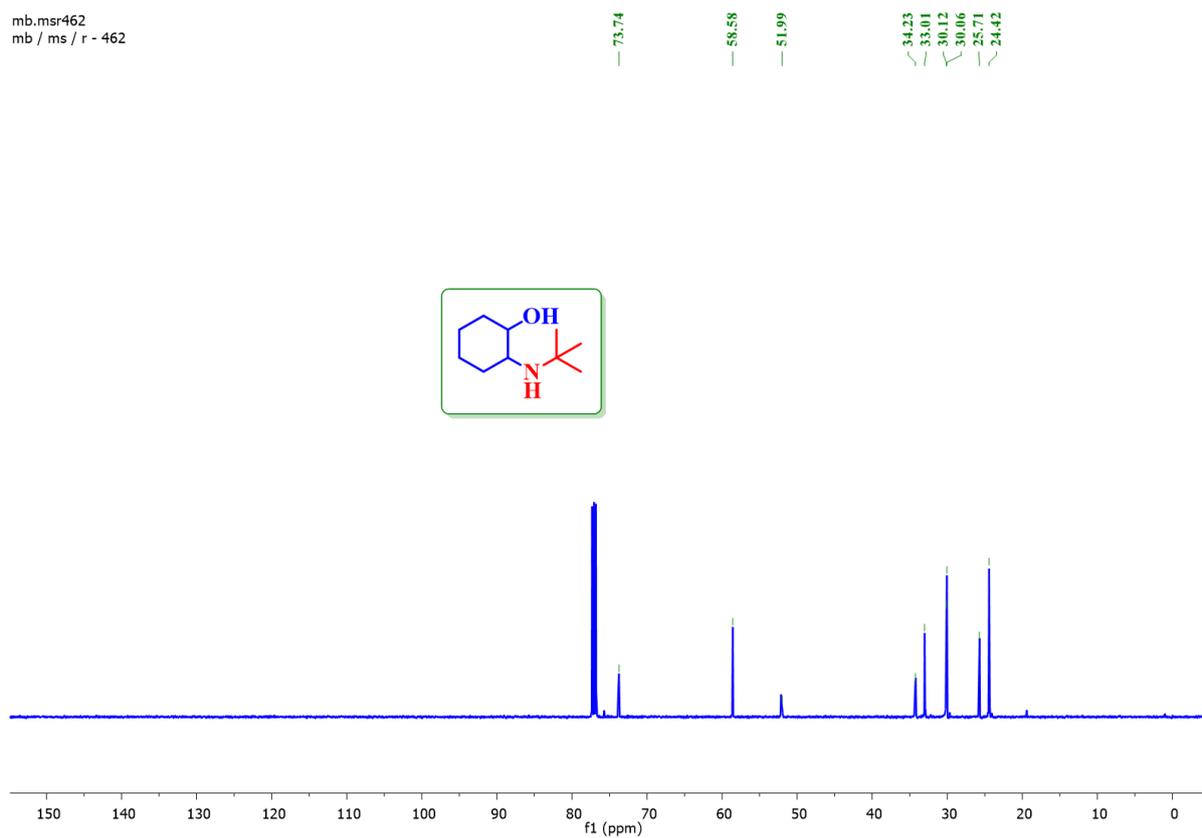


Figure S57 <sup>13</sup>C NMR spectrum of 3r

mb.msr461c  
mb / ms / r - 461c - 1h

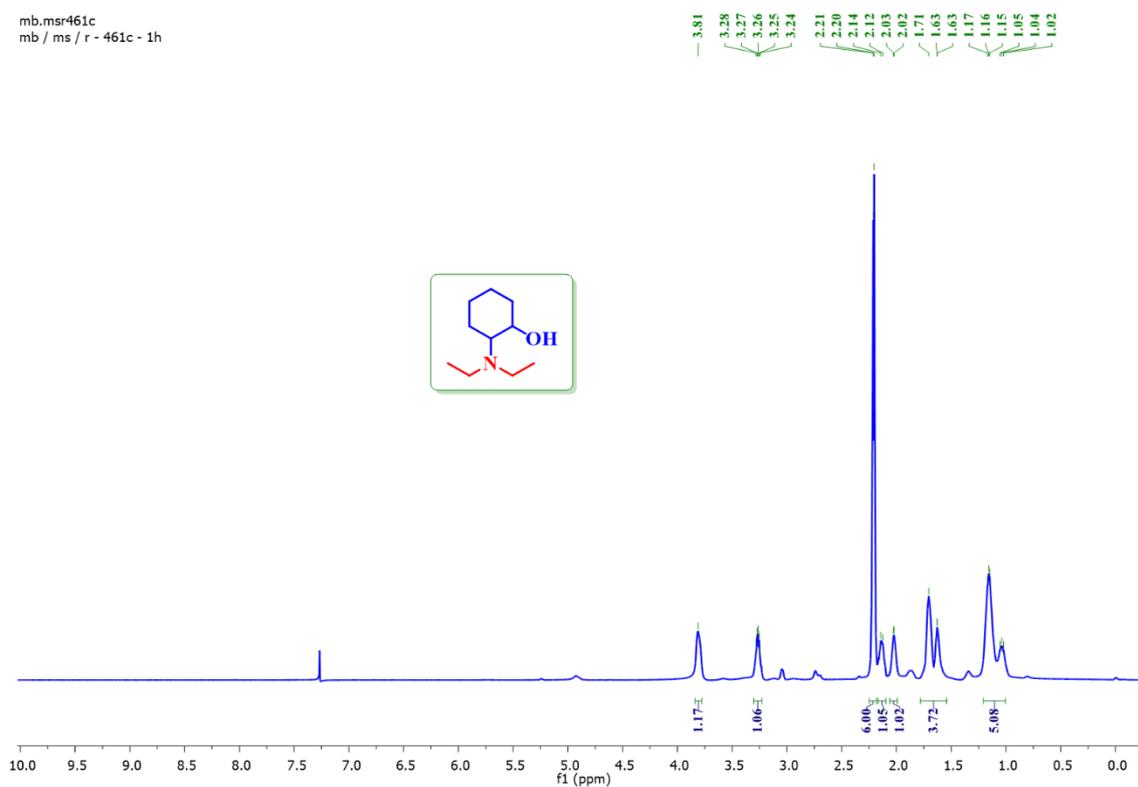


Figure S58 <sup>1</sup>H NMR spectrum of 3s

mb.msr461c  
mb / ms / r - 461c - 1h

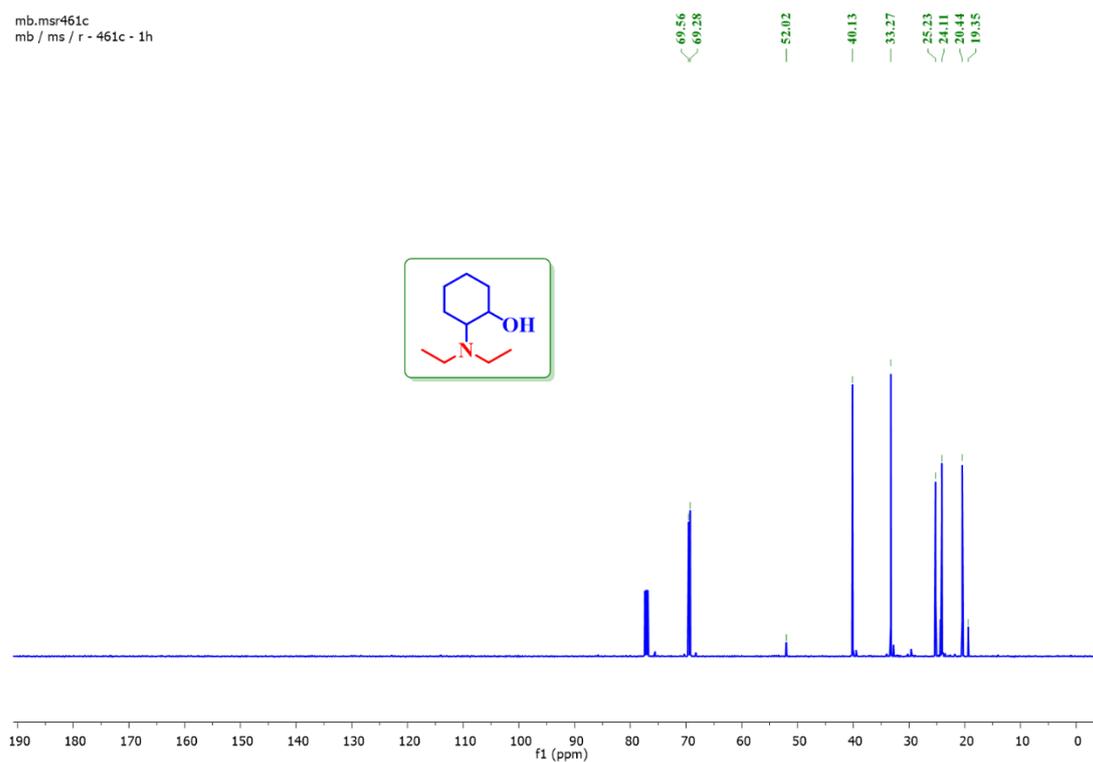


Figure S59 <sup>13</sup>C NMR spectrum of 3s

mb.msr434  
mb / ms / r - 434

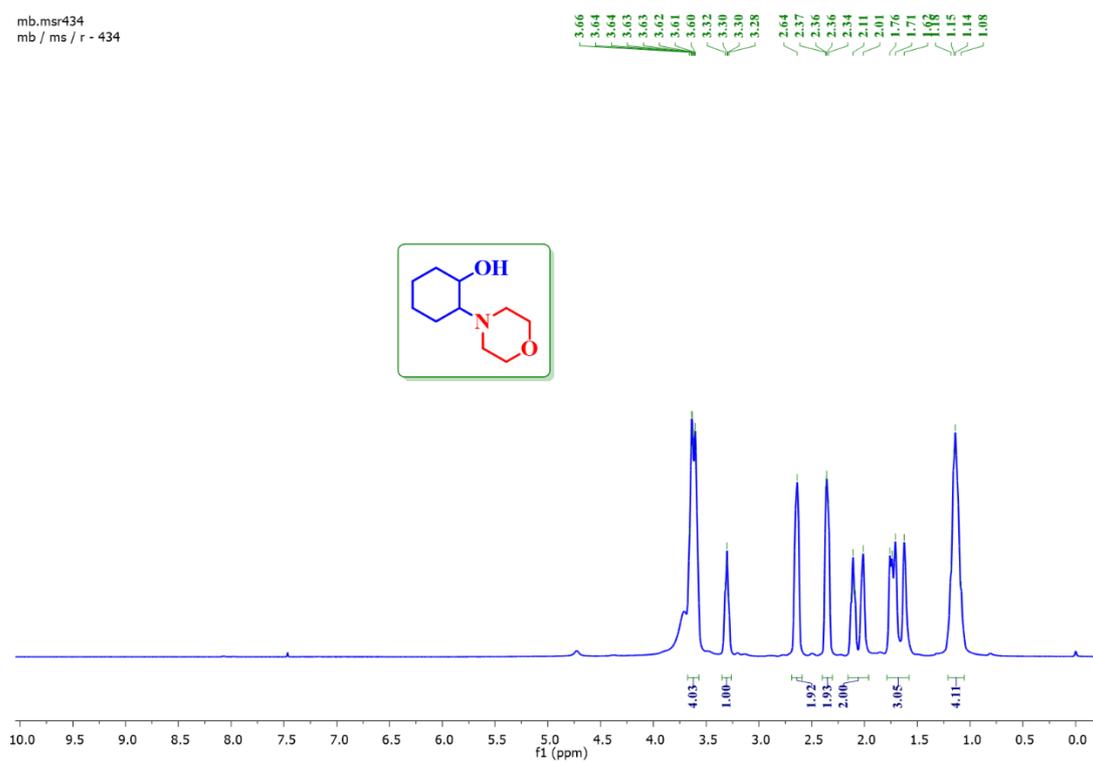


Figure S60 <sup>1</sup>H NMR spectrum of 3t

mb.msr434  
mb / ms / r - 434

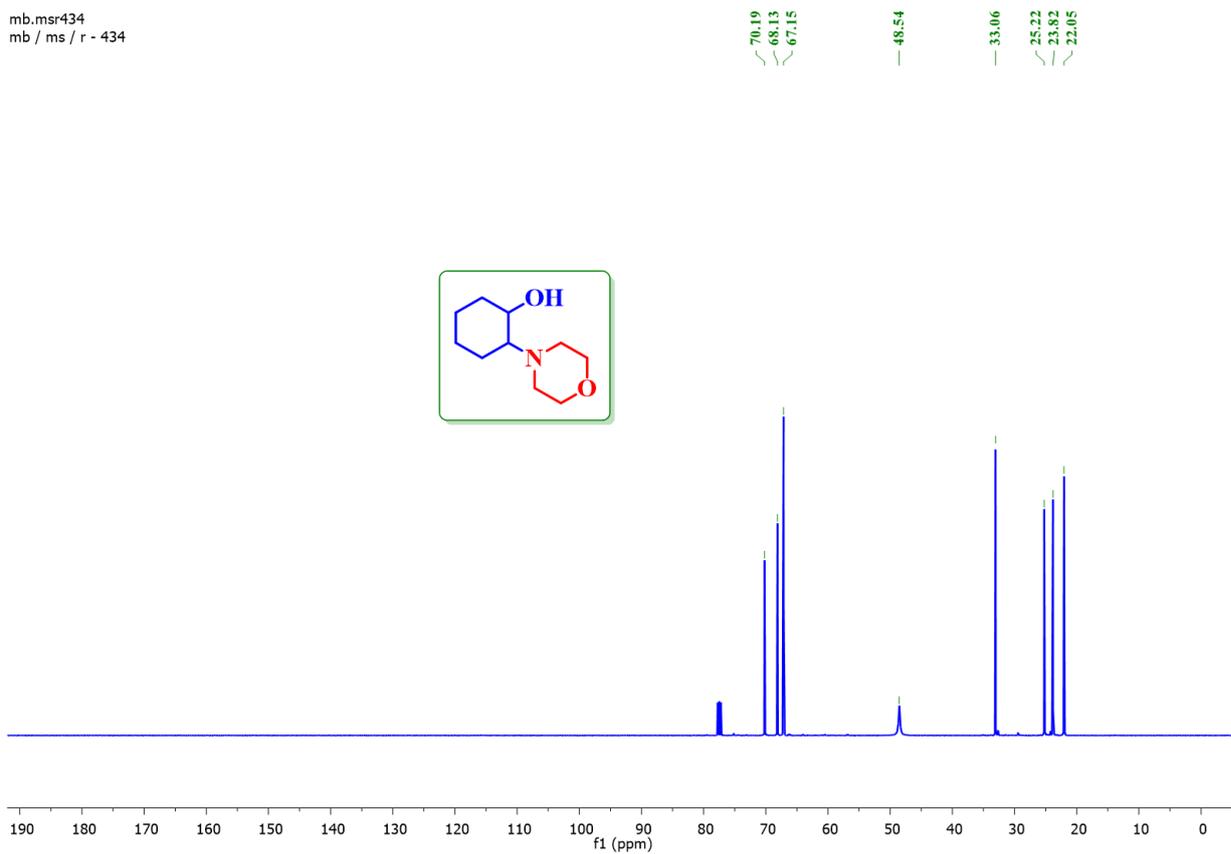


Figure S61  $^{13}\text{C}$  NMR spectrum of **3t**

mb.msr437pr  
mb / ms / r - 437pr

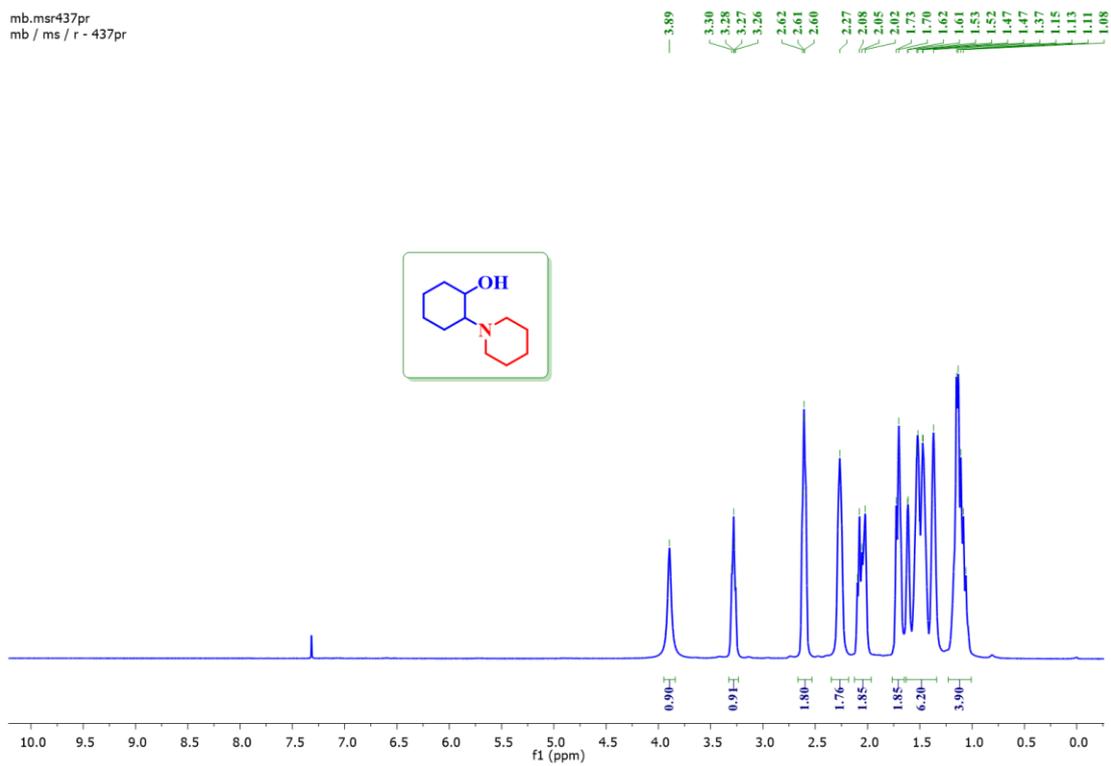


Figure S62  $^1\text{H}$  NMR spectrum of **3u**

mb.msr437pr  
mb / ms / r - 437pr

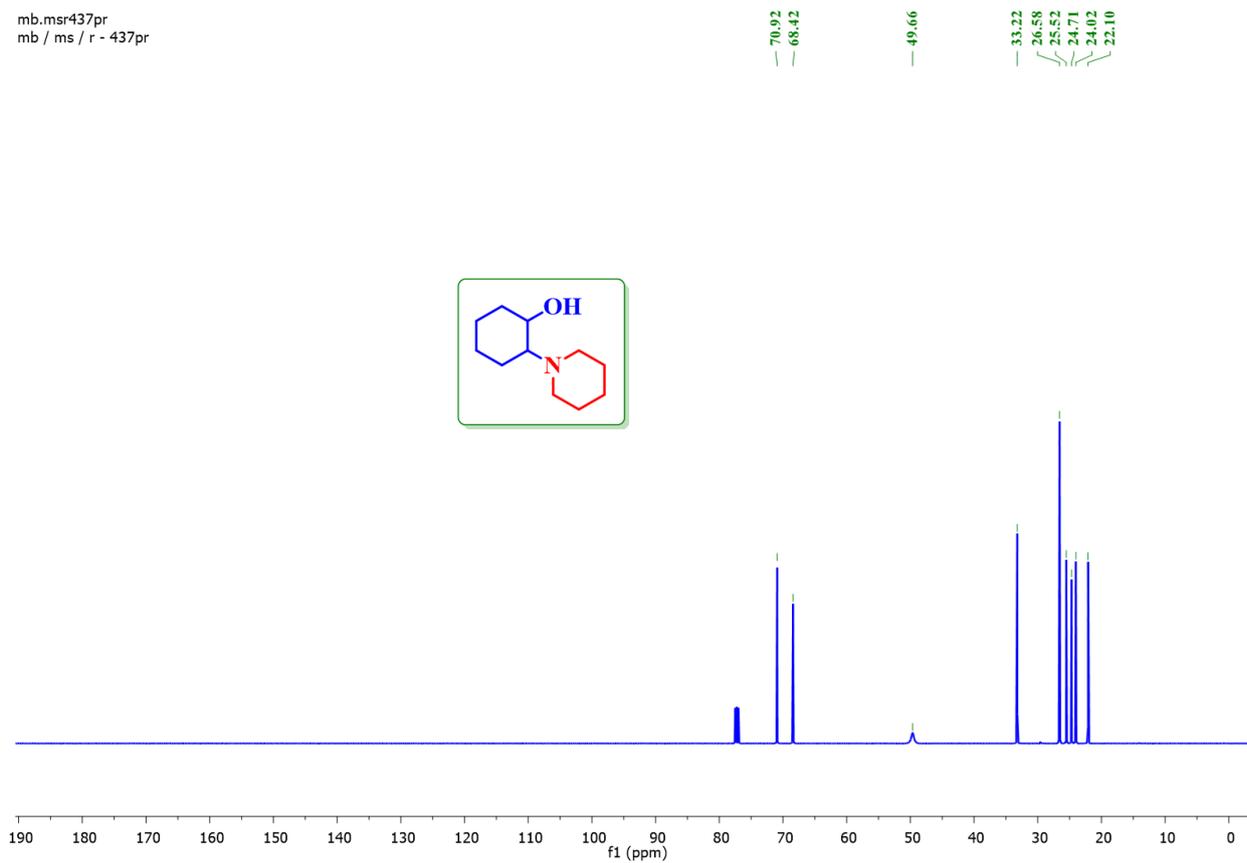


Figure S63  $^{13}\text{C}$  NMR spectrum of **3u**

mb.msr435np2  
mb / ms / r - 435 np2

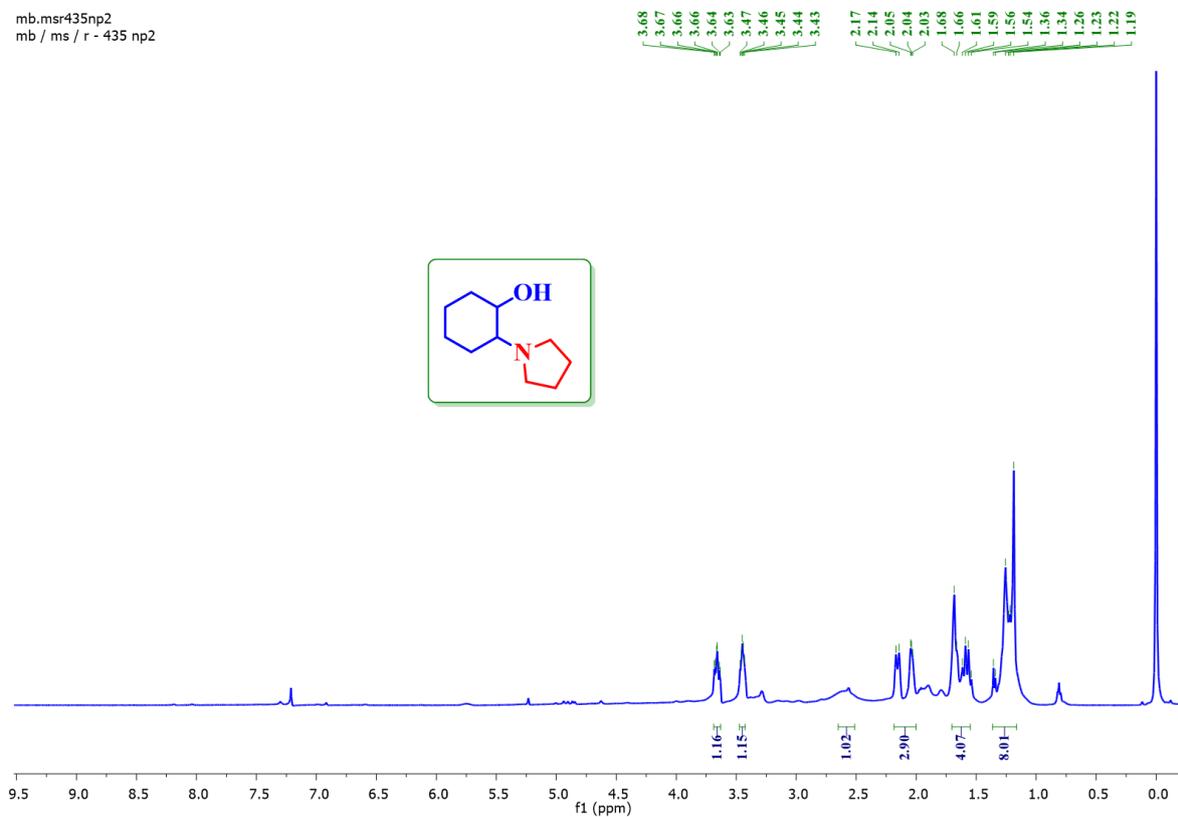


Figure S64  $^1\text{H}$  NMR spectrum of **3v**

mb.msr435np2  
mb / ms / r - 435 np2

74.30

66.43

34.14

32.14

28.68

24.62

22.95

-0.00

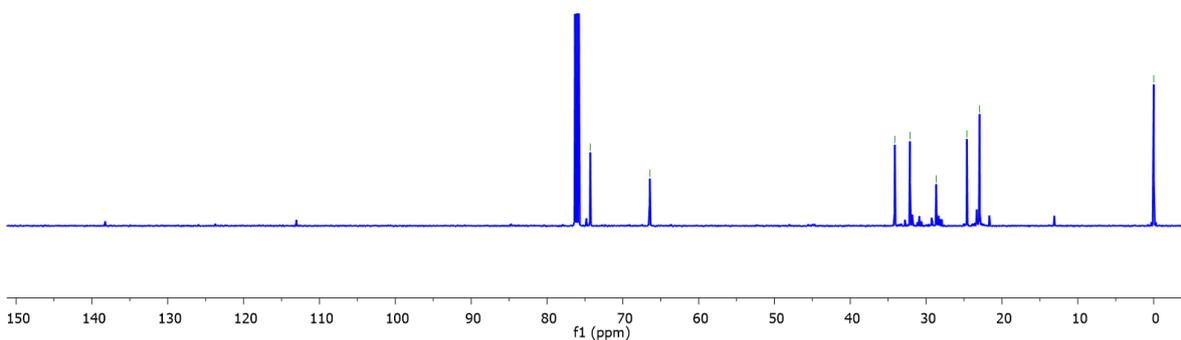
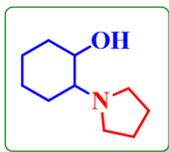


Figure S65 <sup>13</sup>C NMR spectrum of 3v

mb.msr409-1h-400mhz  
mb.msr409-1h-400mhz  
7.40  
7.35  
7.35  
7.33  
7.33  
7.33  
7.32  
7.32  
7.31  
7.31  
7.30  
7.13  
7.13  
7.13  
7.13  
7.12  
7.12  
6.91  
6.89  
6.80  
6.80  
6.79  
6.78  
6.78  
6.77  
4.33  
4.32  
4.32  
4.31  
4.31  
4.30  
4.30  
4.11  
4.10  
4.09  
3.75  
3.51  
3.50  
3.48  
3.47  
3.37  
3.35  
3.34  
3.32

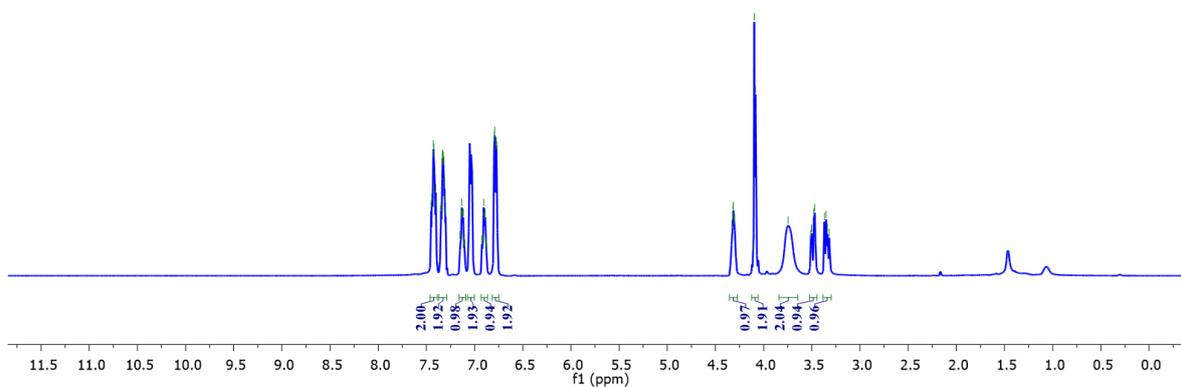
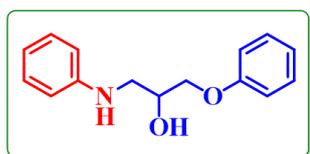


Figure S66 <sup>1</sup>H NMR spectrum of 3v

mb.ms,r-409  
mb.ms,r-409-13c-400mhz

158.60  
148.25  
129.78  
129.53  
121.45  
118.20  
114.76  
113.56  
70.21  
68.88  
46.85

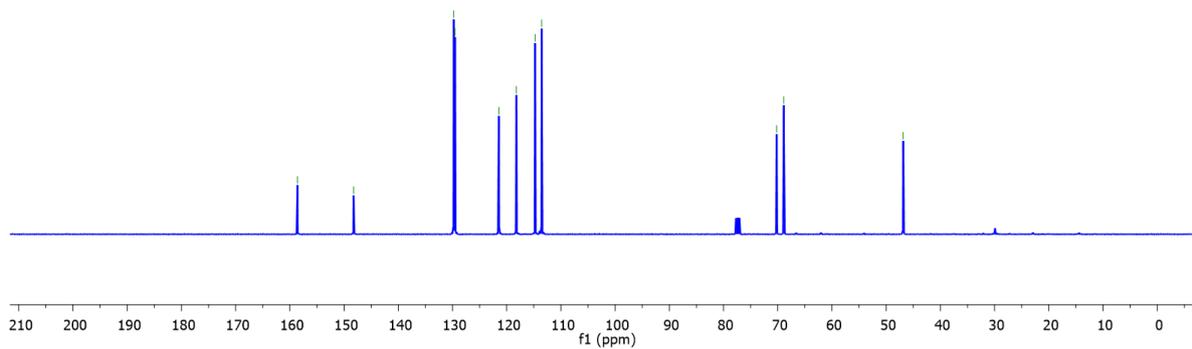
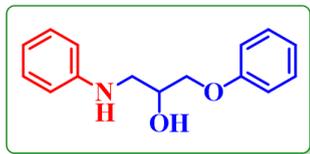


Figure S67  $^{13}\text{C}$  NMR spectrum of **3w**

mb.msr436a1  
mb / ms / r - 436 a1

7.44  
7.42  
7.42  
7.40  
7.39  
7.36  
7.16  
7.15  
7.14  
7.12  
7.11  
6.98  
6.97  
6.89  
6.88  
6.87  
6.86  
4.39  
4.39  
4.15  
4.15  
3.90  
3.89  
3.63  
3.62  
3.61  
3.60  
3.45  
3.44  
3.43  
2.53  
2.53  
2.51  
2.50  
2.49

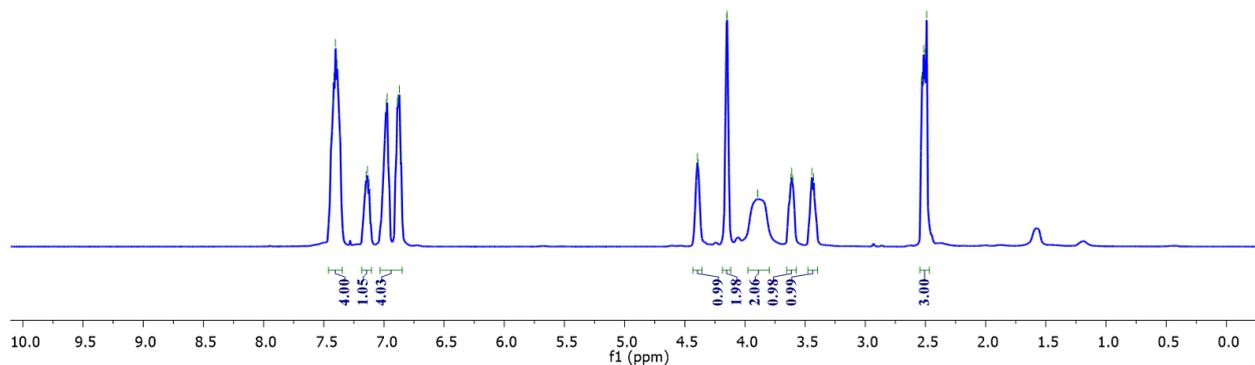
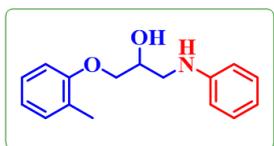


Figure S68  $^1\text{H}$  NMR spectrum of **3x**

mb.msr436a1  
mb / ms / r - 436 a1

156.81  
148.39  
131.10  
129.63  
127.22  
126.89  
121.24  
118.28  
113.64  
111.47  
70.06  
69.04  
47.21  
16.58

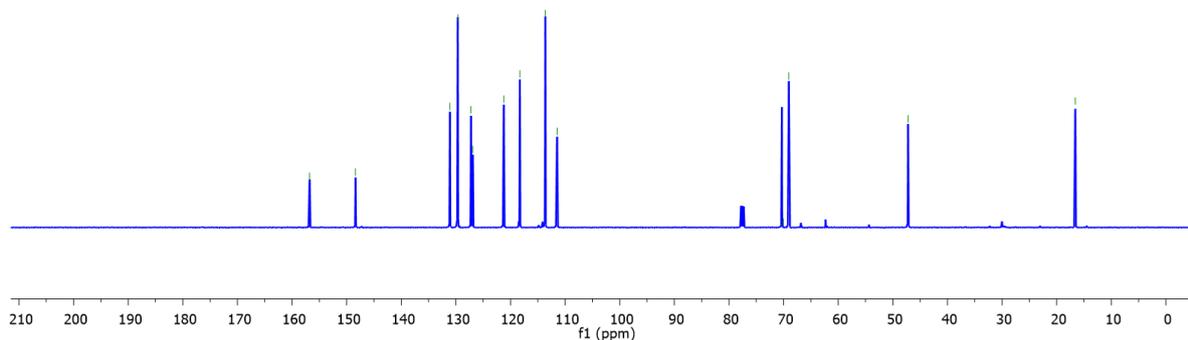
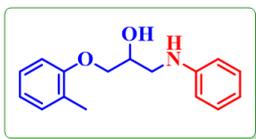


Figure S69 <sup>13</sup>C NMR spectrum of 3x

mb.ms,r-405  
mb.ms,r-405-1h-400mhz

7.28  
7.26  
7.26  
7.24  
6.83  
6.81  
6.79  
6.74  
6.72  
4.04  
4.03  
4.02  
4.00  
3.99  
3.49  
3.48  
3.46  
3.46  
3.36  
3.35  
3.33  
3.32  
3.20  
3.19  
3.17  
3.15  
1.50  
1.26

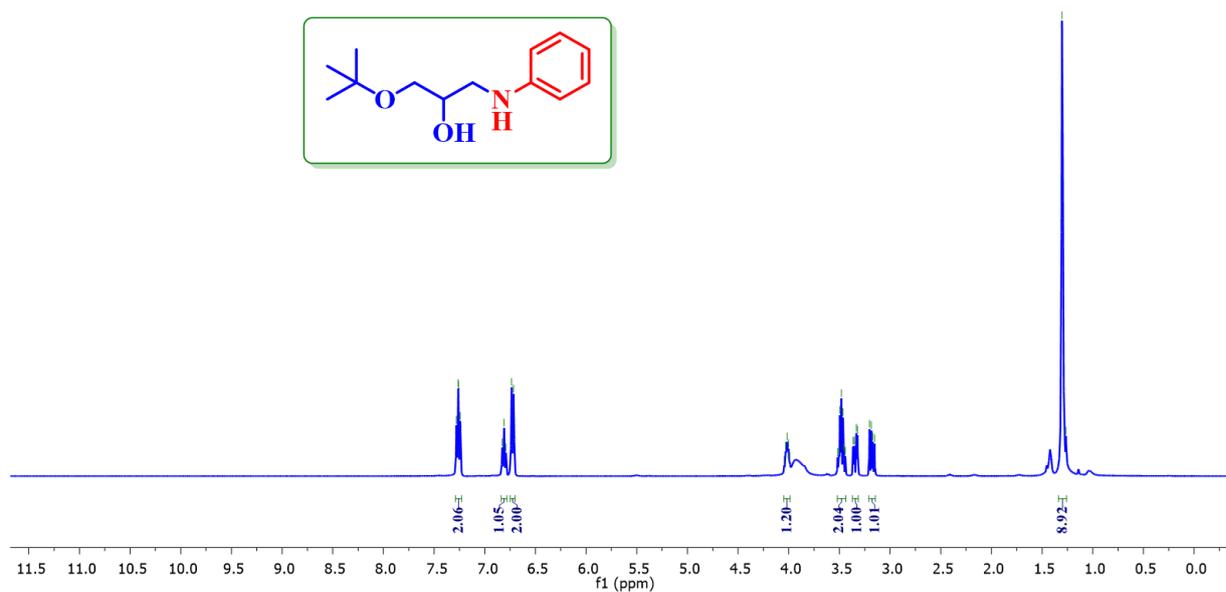
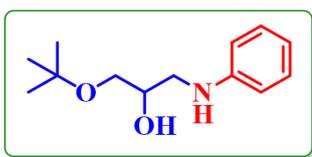


Figure S70 <sup>1</sup>H NMR spectrum of 3y

mb.ms,r-405  
mb.ms,r-405-13c-400mhz

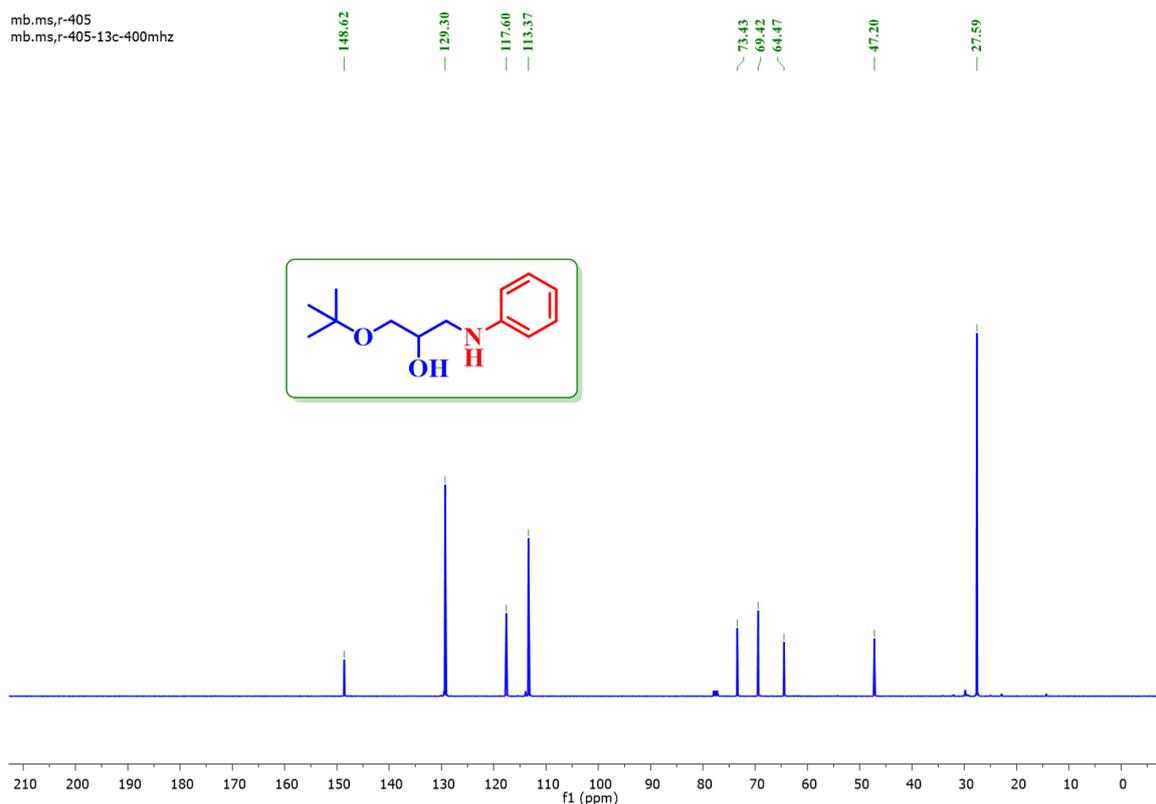


Figure S71 <sup>13</sup>C NMR spectrum of 3y

mb.msr449a  
mb / ms / r - 449a

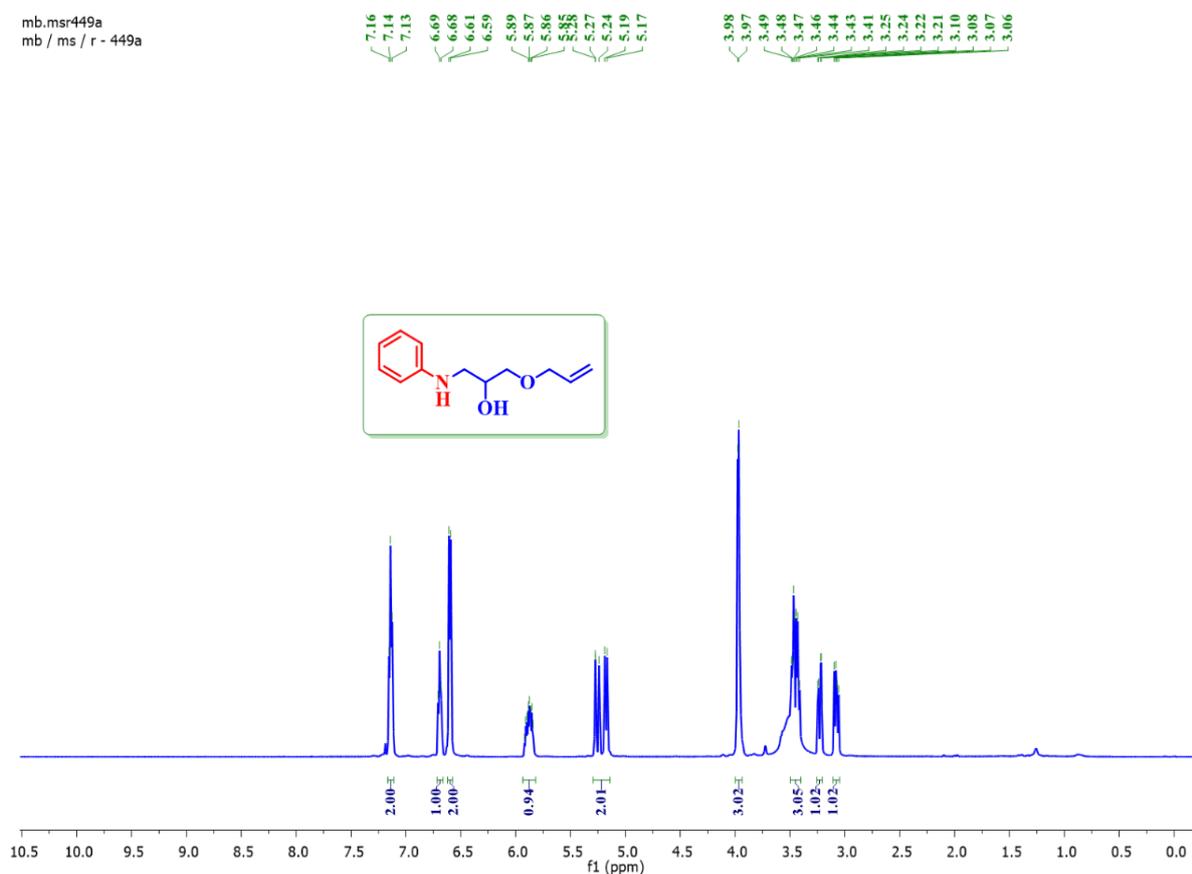


Figure S72 <sup>1</sup>H NMR spectrum of 3z

mb.msr449a  
mb / ms / r - 449a

148.34  
134.46  
129.33  
117.80  
117.52  
113.33  
72.61  
72.40  
69.08  
46.88

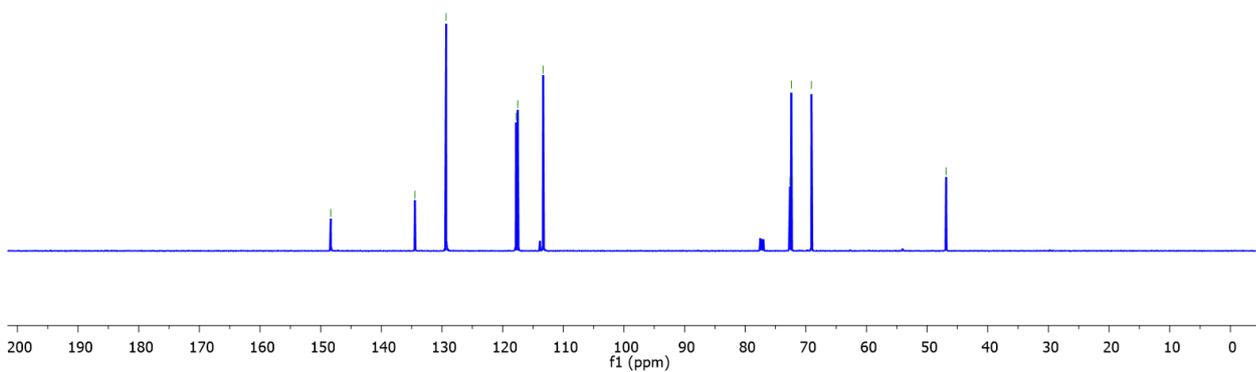
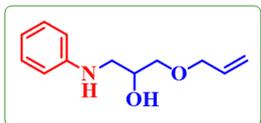


Figure S73 <sup>13</sup>C NMR spectrum of 3z

mb.msr446a  
mb / ms / r - 446a

7.04  
7.02  
7.01  
6.58  
6.57  
6.49  
6.48  
6.48  
5.99  
5.45  
5.43  
4.06  
4.05  
3.92  
3.62  
3.61  
3.23  
3.14  
3.14  
3.12  
3.11  
3.00  
2.98  
2.97  
1.86  
1.79

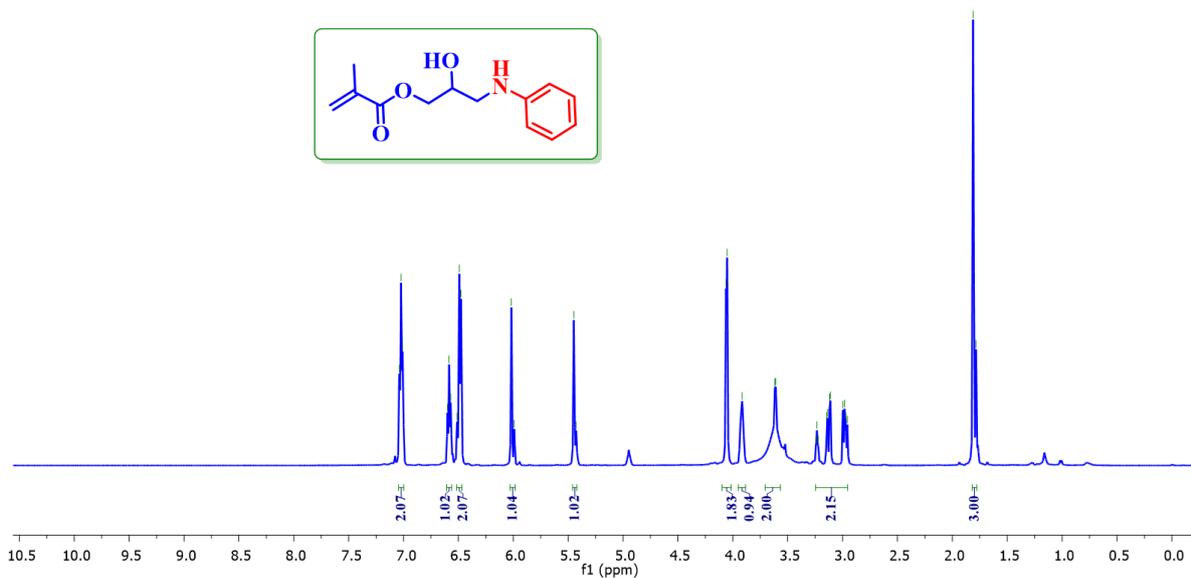
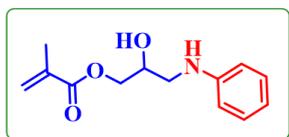


Figure S74 <sup>1</sup>H NMR spectrum of 3aa

mb.msr446a  
mb / ms / r - 446a

167.74  
148.12  
135.97  
129.40  
126.50  
118.01  
113.37  
113.14  
68.29  
66.79  
46.77  
18.34

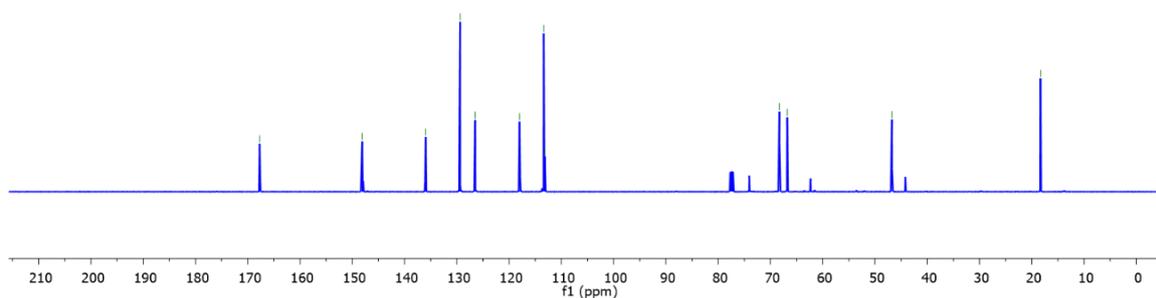
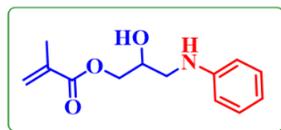


Figure S75 <sup>13</sup>C NMR spectrum of 3aa

mb.ms.r-407  
mb.ms.r-407 1h nmr 400mhz

7.10  
7.10  
7.09  
7.08  
7.06  
6.67  
6.65  
6.64  
6.64  
6.63  
6.54  
6.52  
6.52  
3.92  
3.91  
3.90  
3.90  
3.89  
3.89  
3.89  
3.88  
3.87  
3.86  
3.51  
3.49  
3.48  
3.47  
3.46  
3.44  
3.43  
3.41  
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3.19  
3.18  
3.06  
3.04  
3.03  
3.01

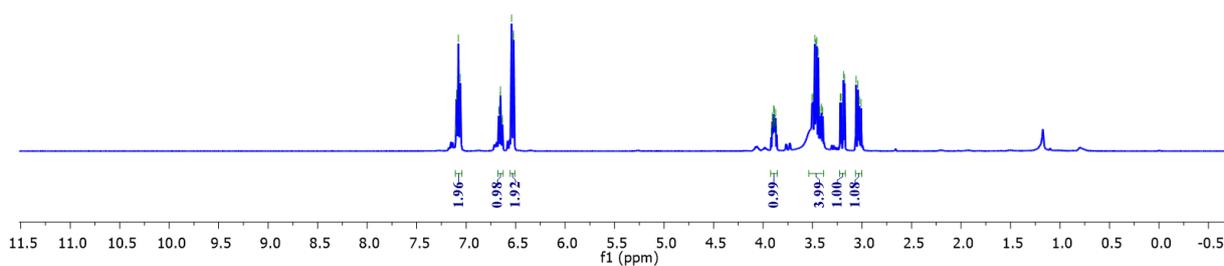
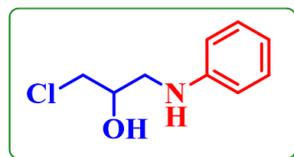


Figure S76 <sup>1</sup>H NMR spectrum of 3ab

mb.ms.r-407  
mb.ms.r-407 13c nmr 400mhz

147.70  
129.50  
118.48  
113.59  
69.83  
47.59  
47.29

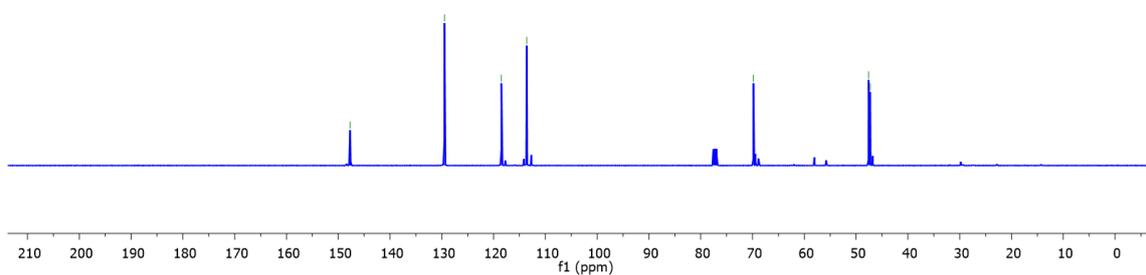
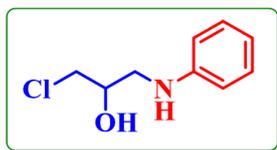
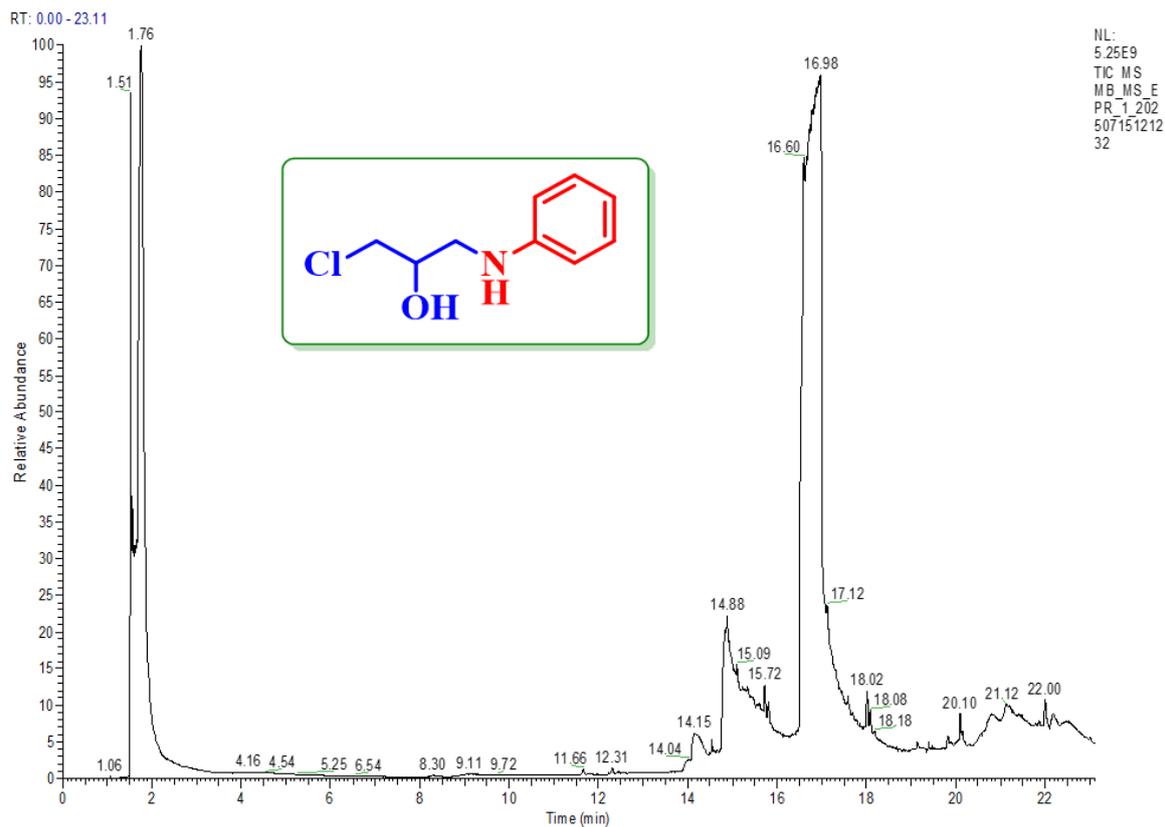


Figure S77  $^{13}\text{C}$  NMR spectrum of 3ab



MB MS EPR 1 20250715121232 #4699 RT: 16.98 AV: 1 NL: 9.83E8  
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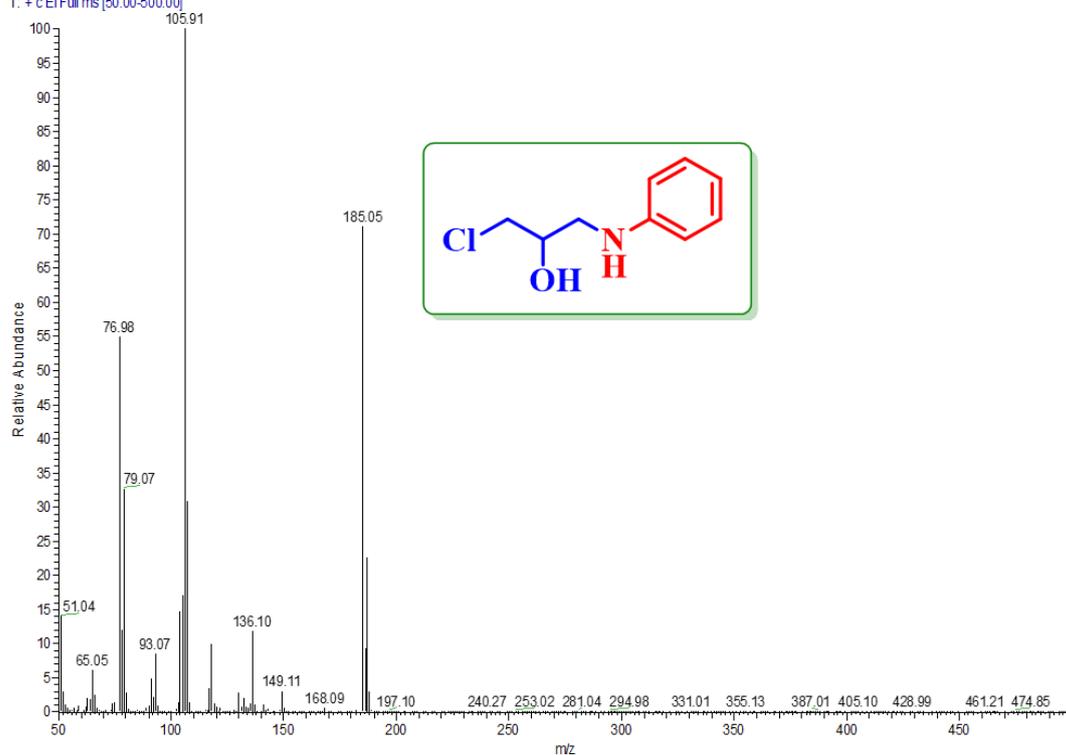


Figure S78 GC-MS spectra of **3ab**

mb.msr445  
mb / ms / r - 445 a

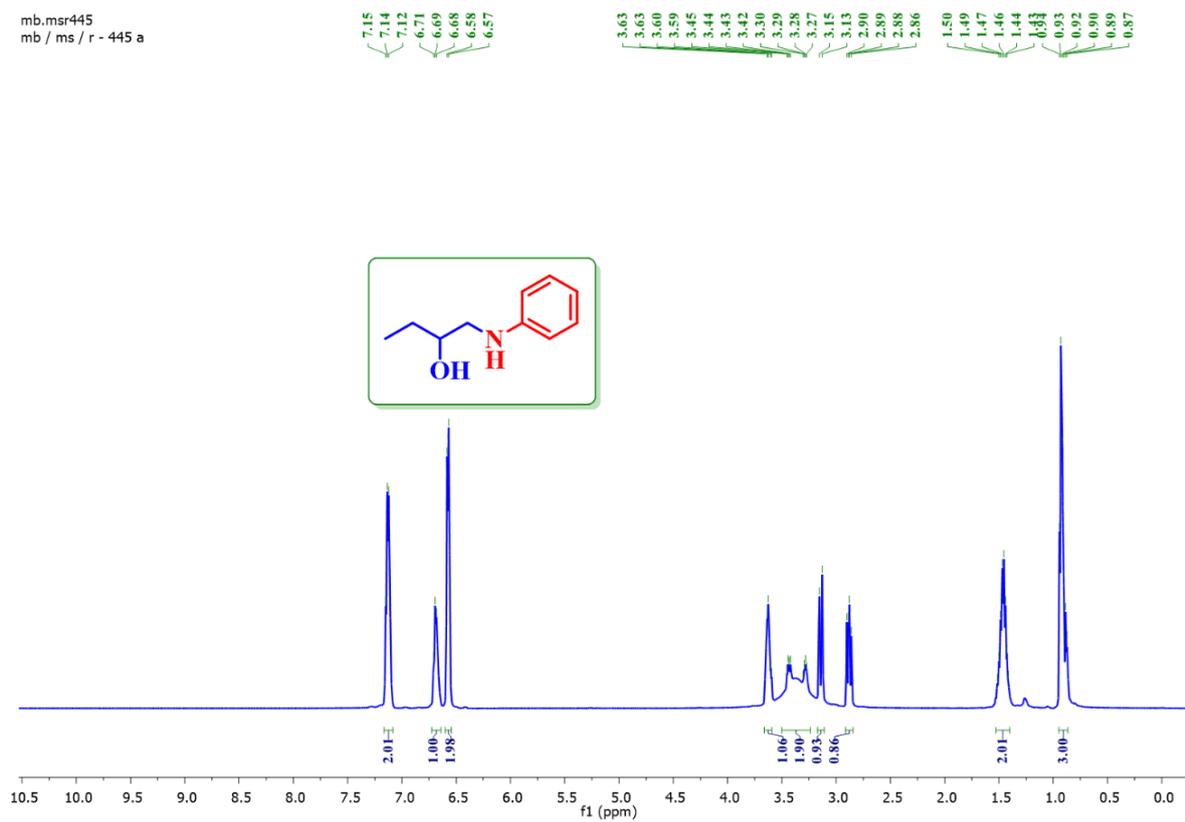


Figure S79 <sup>1</sup>H NMR spectrum of **3ac**

mb.msr445  
mb / ms / r - 445 a

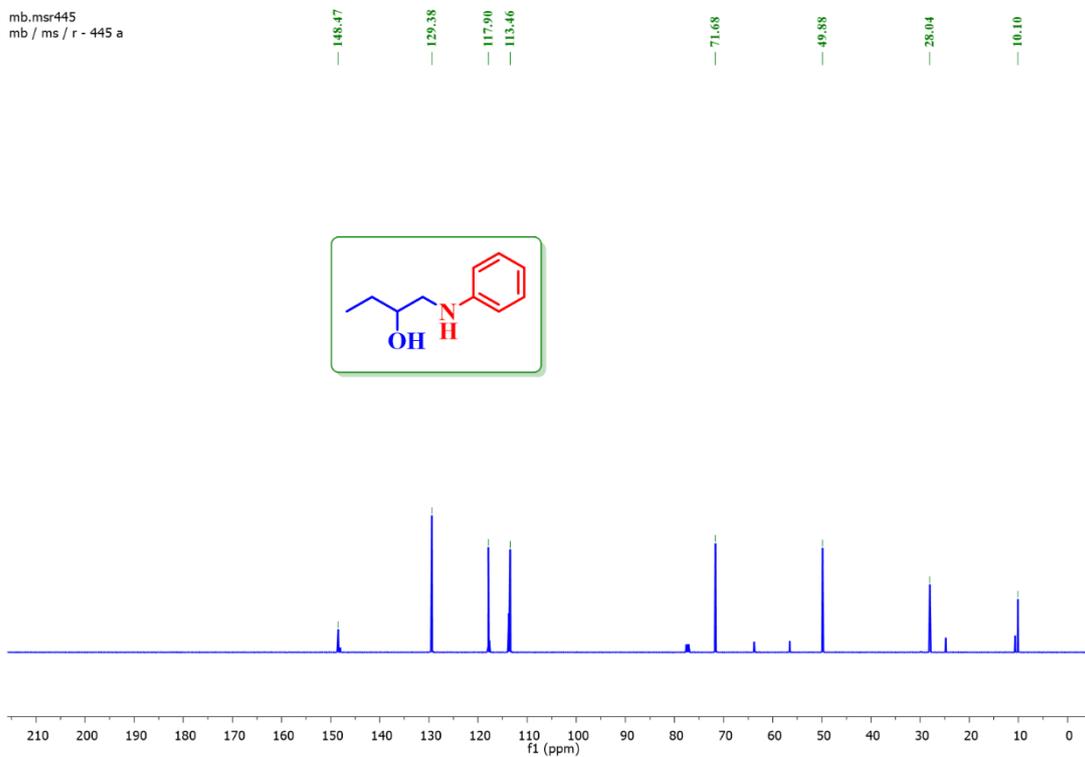


Figure S80 <sup>13</sup>C NMR spectrum of **3ac**

mb.msr447a  
mb / ms / r - 447 a

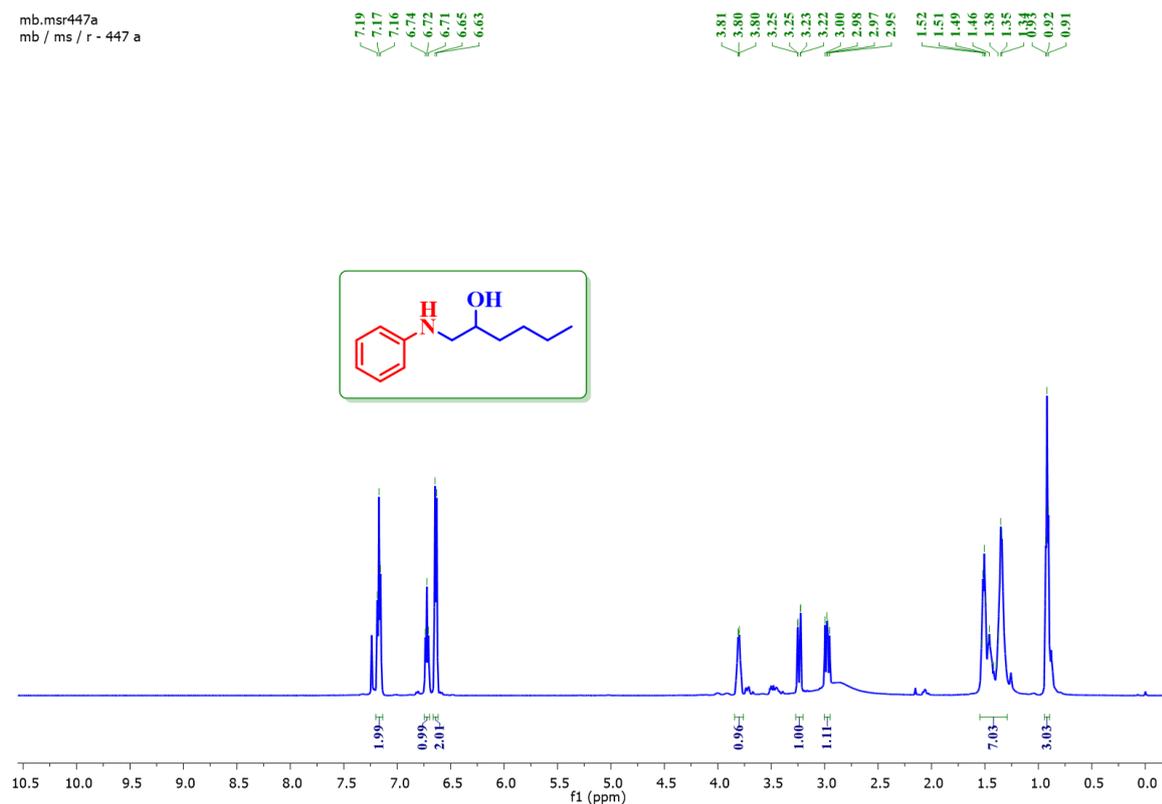


Figure S81 <sup>1</sup>H NMR spectrum of 3ad

mb.msr447a  
mb / ms / r - 447 a

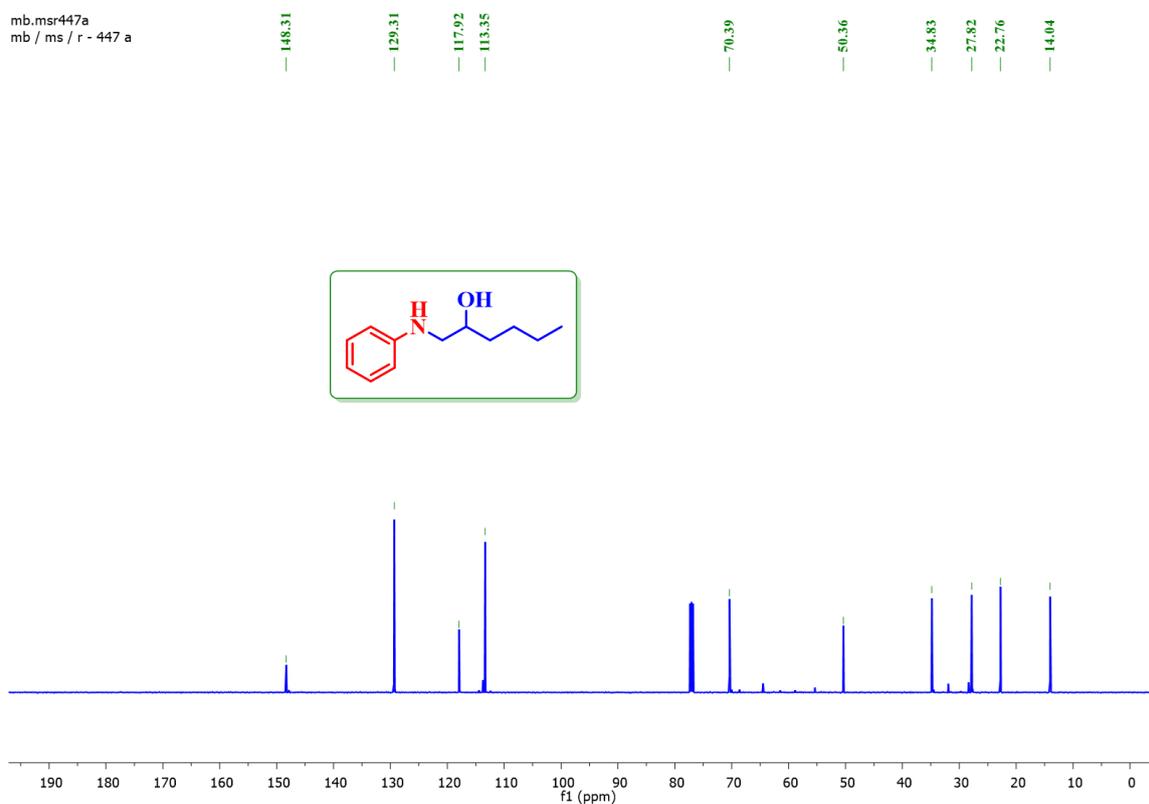


Figure S82 <sup>13</sup>C NMR spectrum of 3ad

mb.msr459i  
mb / ms / r - 459 i

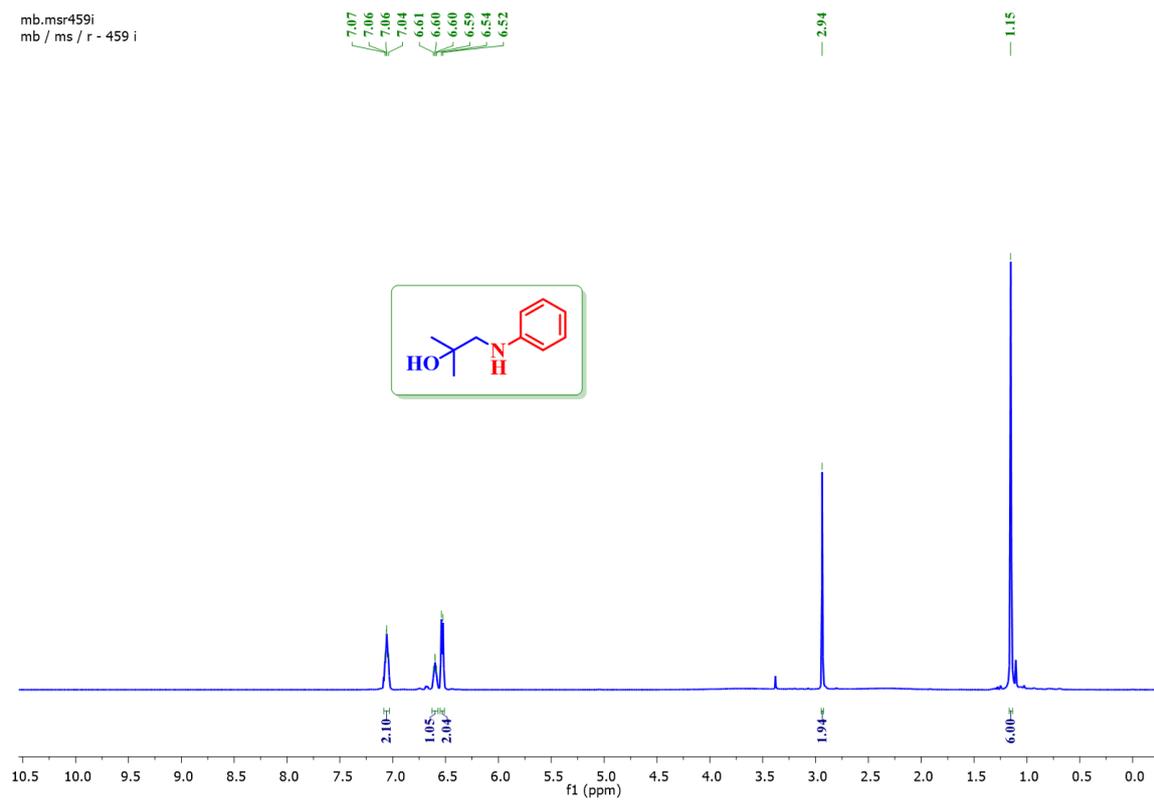


Figure S83 <sup>1</sup>H NMR spectrum of 3ae

mb.msr459i  
mb / ms / r - 459 i

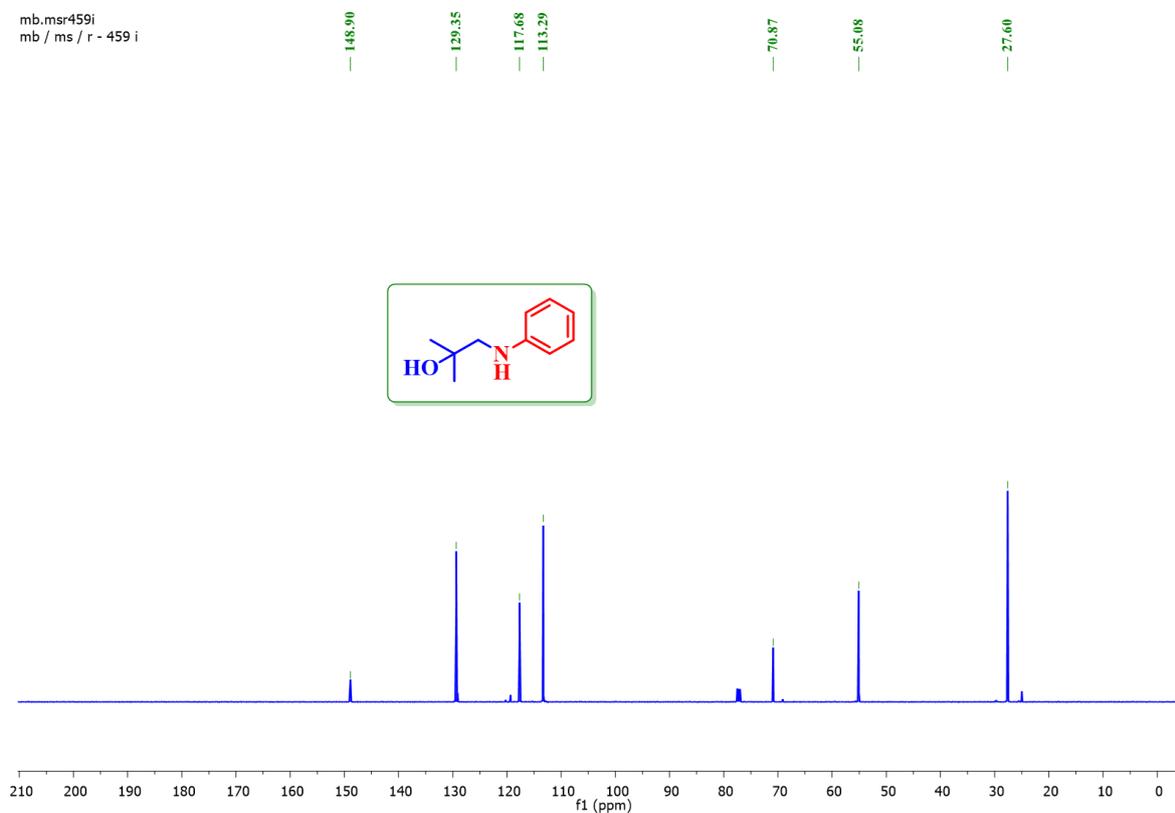
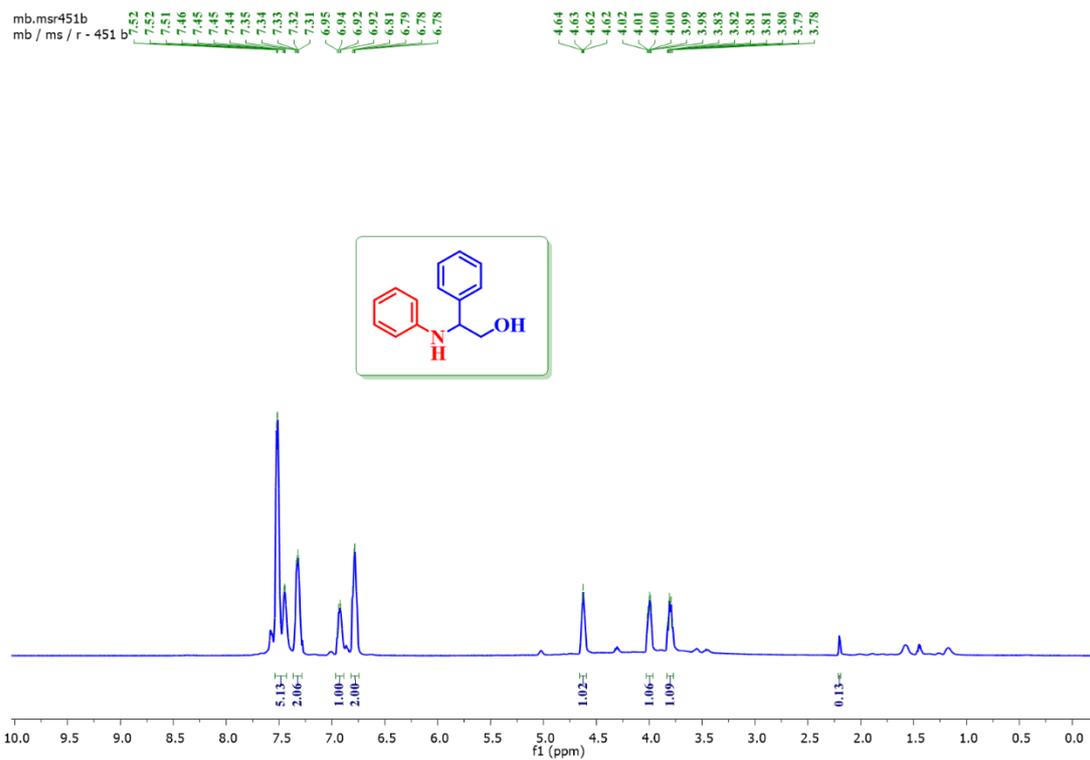
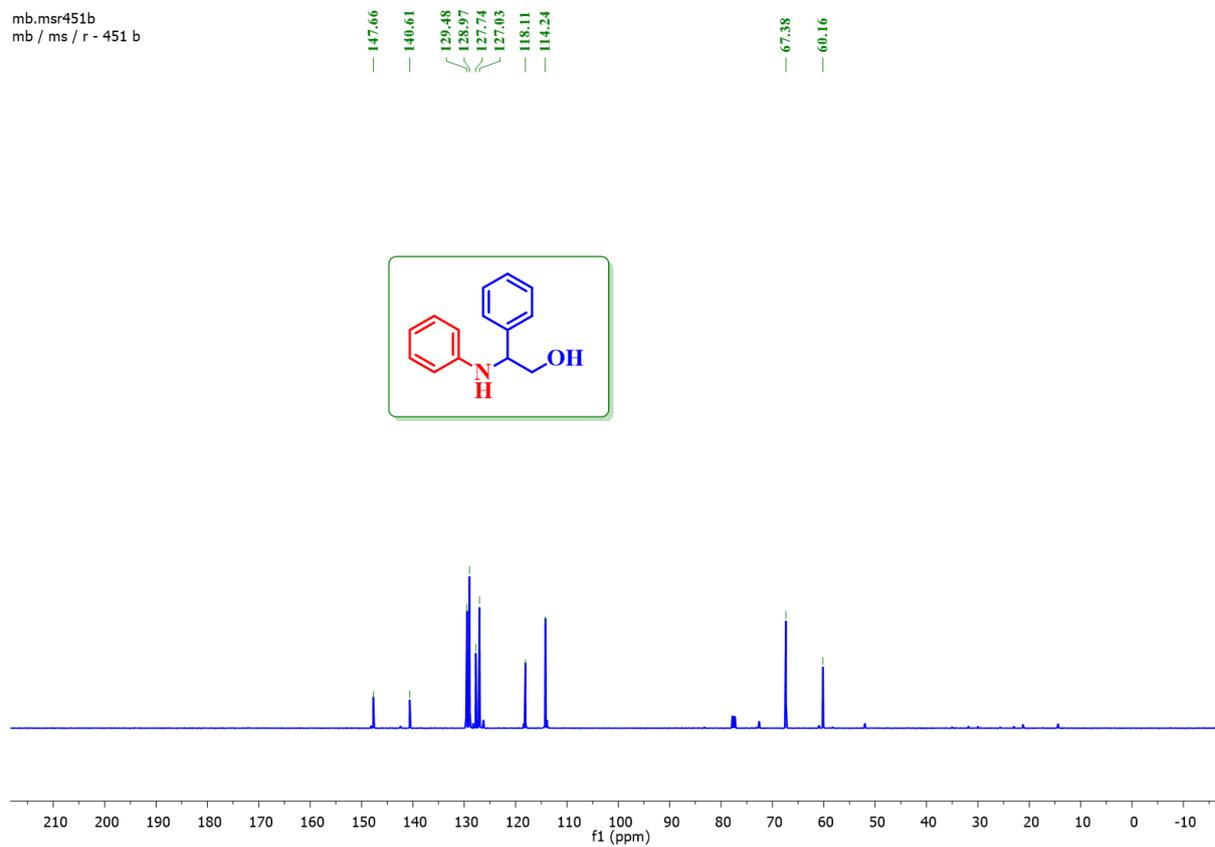


Figure S84 <sup>13</sup>C NMR spectrum of 3ae



**Figure S85**  $^1\text{H}$  NMR spectrum of **3af**



**Figure S86**  $^{13}\text{C}$  NMR spectrum of **3af**

mb.msr450b  
mb / ms / r - 450b

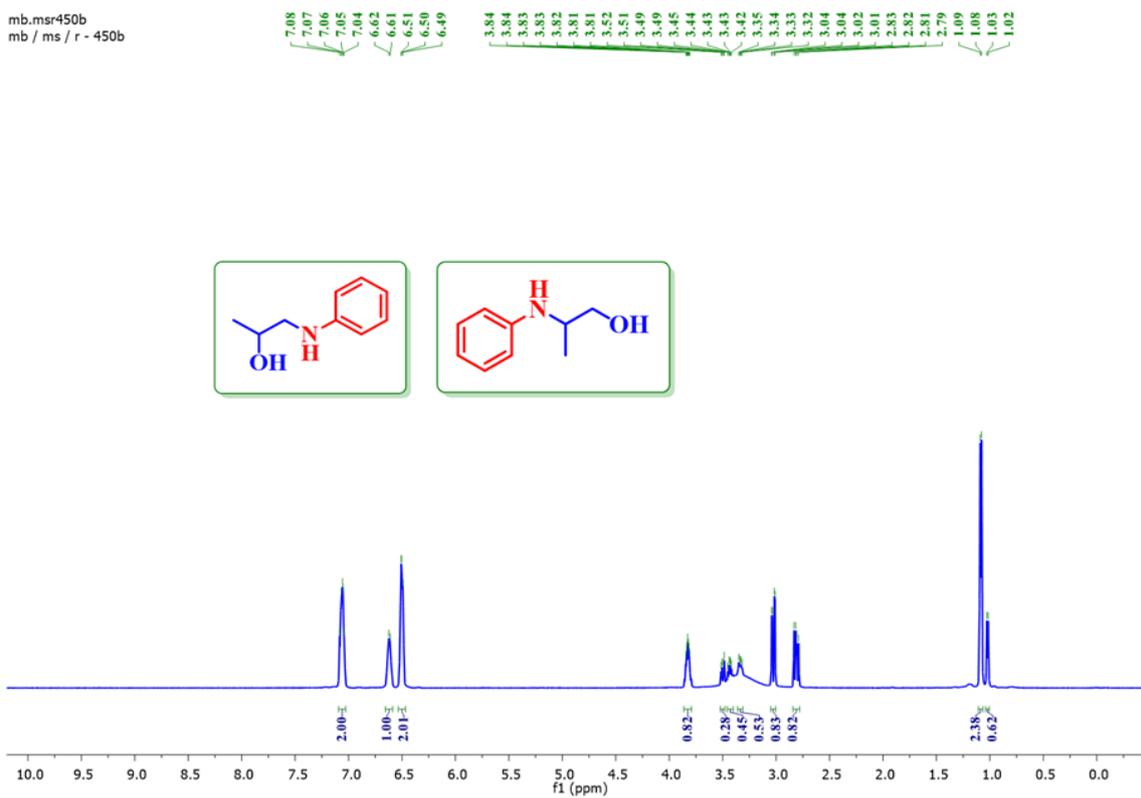


Figure S87 <sup>1</sup>H NMR spectrum of 3ag

mb.msr450b  
mb / ms / r - 450b

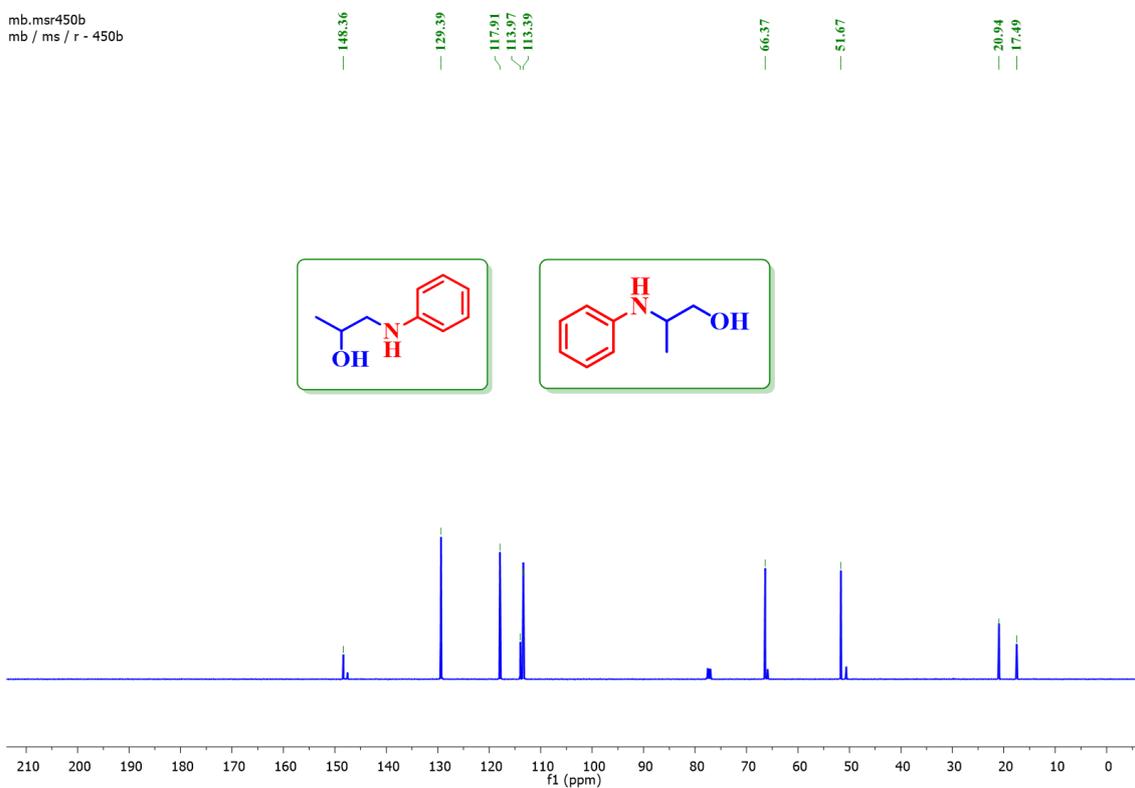


Figure S88 <sup>13</sup>C NMR spectrum of 3ag

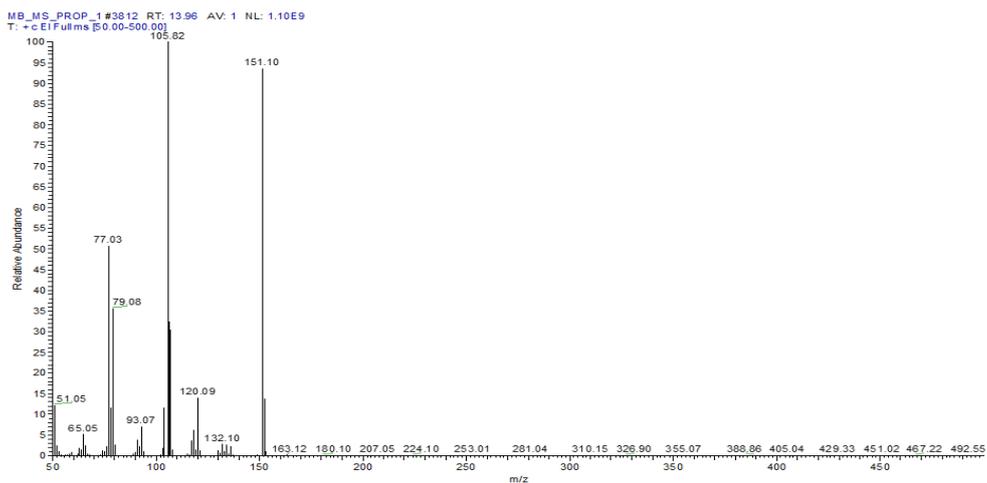
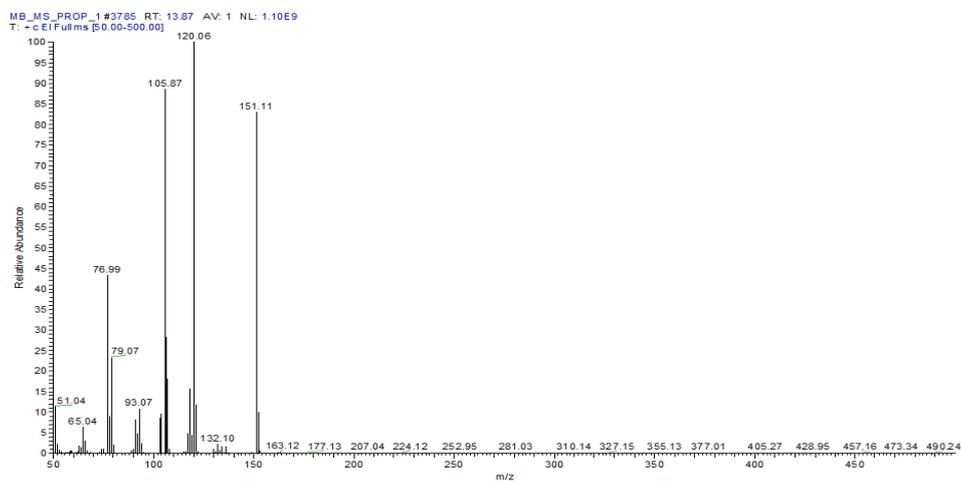
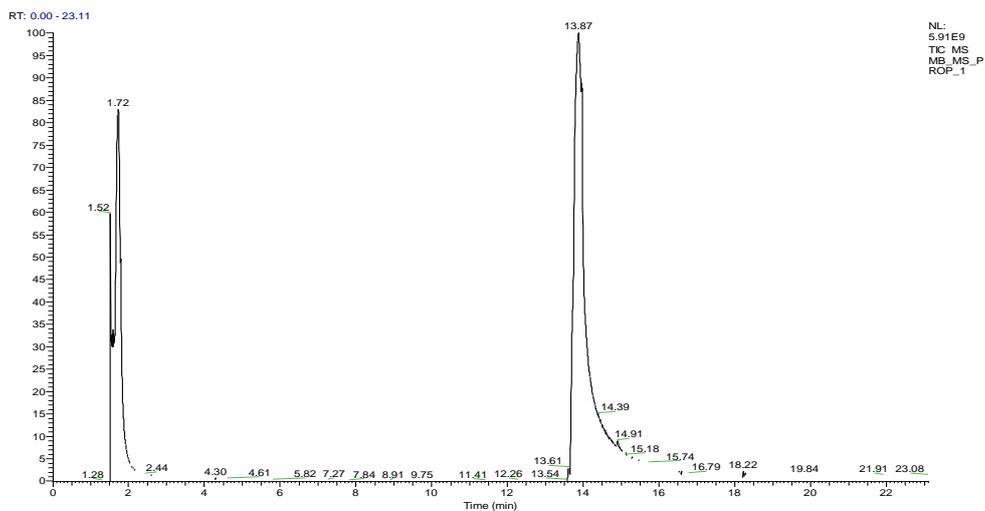


Figure S93 GC-MS spectra of **3ag**

**Table S1** Comparison of the catalytic activity of the Cu-MOG, with the reported systems

Sl. No	Catalyst	Catalyst loading	Reaction conditions	Yield	Selectivity	Ref.
1	Acetic Acid	1.0 equiv	RT, solvent-free, 1 h	99	Excellent	S1
2	Montmorillonite K-10	10 wt %	Solvent-free, RT, N <sub>2</sub> , 5 h	70-88	Good	S2
3	Pd/C (Metal Catalyst)	5 mol%	Methanol, 80 °C, 6 h	85-90	High	S3
4	SZ-2-600	20 mg	80 °C, solvent-free, RT	82-97	Excellent	S4
5	Sulfated zirconia	60 mg	solvent-free, RT		Excellent	S5
6	Zn(BF <sub>4</sub> ) <sub>2</sub> ·xH <sub>2</sub> O	(2 mol%)	solvent-free, RT		Excellent	S6
7	[O(CH <sub>2</sub> C <sub>6</sub> H <sub>4</sub> ) <sub>2</sub> Bi(OH <sub>2</sub> )] <sup>+</sup> [OSO <sub>2</sub> CF <sub>3</sub> ] <sup>-2</sup>	5 mol%	RT, solvent(2 ml), 2.5 h	75-95	Good	S7
8	Biogenic Iron Oxide Nanoparticles	20 mg	Solvent-free, 80 °C, 4.5-5 h	90	Good	S8
9	NaY Zeolite (Heterogeneous Catalyst)	100 mg	Solvent-free, 80 °C, 5 h	85-92	Excellent	S9
10	Copper(0)		Ambient Temp, solvent-free	95	High	S10
10	Sc(OTf) <sub>3</sub>	5 mol%	solvent-free/CHCl <sub>3</sub> , RT, 12 h	85-96	High	S11
11	Bi(NO <sub>3</sub> ) <sub>3</sub> ·5H <sub>2</sub> O	1 mol%	Solvent-free, microwave irradiation, 100 °C, 10 min	90%	More substituted carbon	S12
12	2,4,6-trichloro-1,3,5-triazine	5 mol%	Neat, RT	82%		S13
13	Zn(ClO <sub>4</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	2 mol %	Neat, RT or 80 °C, 1 h	88-100%	Excellent	S14
14	<b>Cu-MOG</b>	<b>2 mg</b>	<b>RT, solvent-free, 1 h</b>	<b>90-100</b>	<b>Excellent</b>	<b>This work</b>

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