

## Supporting Information

# H- $\beta$ -Zeolite Catalyzed Transamidation of Carboxamides, Phthalimide, Formamides and Thioamides with Amines under neat Conditions

Sadu Nageswara Rao, Darapaneni Chandra Mohan and Subbarayappa Adimurthy \*

Central Salt & Marine Chemicals Research Institute, Council of Scientific & Industrial Research, G.B. Marg, Bhavnagar-364 002. Gujarat (INDIA); Fax: +91-278-2567562

[adimurthy@csmcri.org](mailto:adimurthy@csmcri.org)

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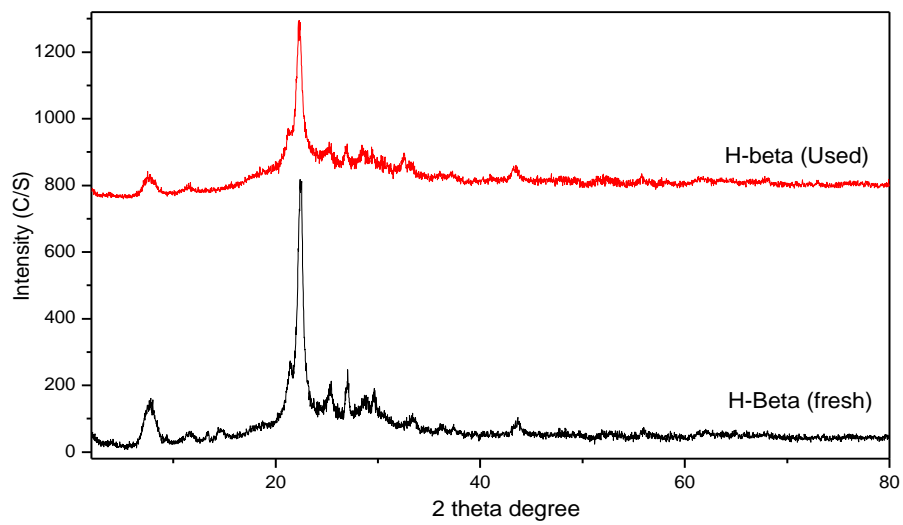
## I. General Experimental Section:

**General:** All commercially available chemicals and reagents were used without any further purification unless otherwise indicated.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded at 500/200, and 125/50 MHz, respectively. The spectra were recorded in  $\text{CDCl}_3$  as solvent. Multiplicity was indicated as follows: s (singlet); d (doublet); t (triplet); m (multiplet); dd (doublet of doublets), etc. and coupling constants (J) were given in Hz. Chemical shifts are reported in ppm relative to TMS as an internal standard. The peaks around delta values of  $^1\text{H}$  NMR (7.2), and  $^{13}\text{C}$  NMR (77.0) are correspond to deuterated solvents chloroform. Mass spectra were obtained using electron impact (EI) ionization method. Progress of the reactions was monitored by thin layer chromatography (TLC). All products were purified through column chromatography using silica gel 200-400 mesh size using dichloromethane (DCM)/ethyl acetate as (80:20) eluent unless otherwise indicated.

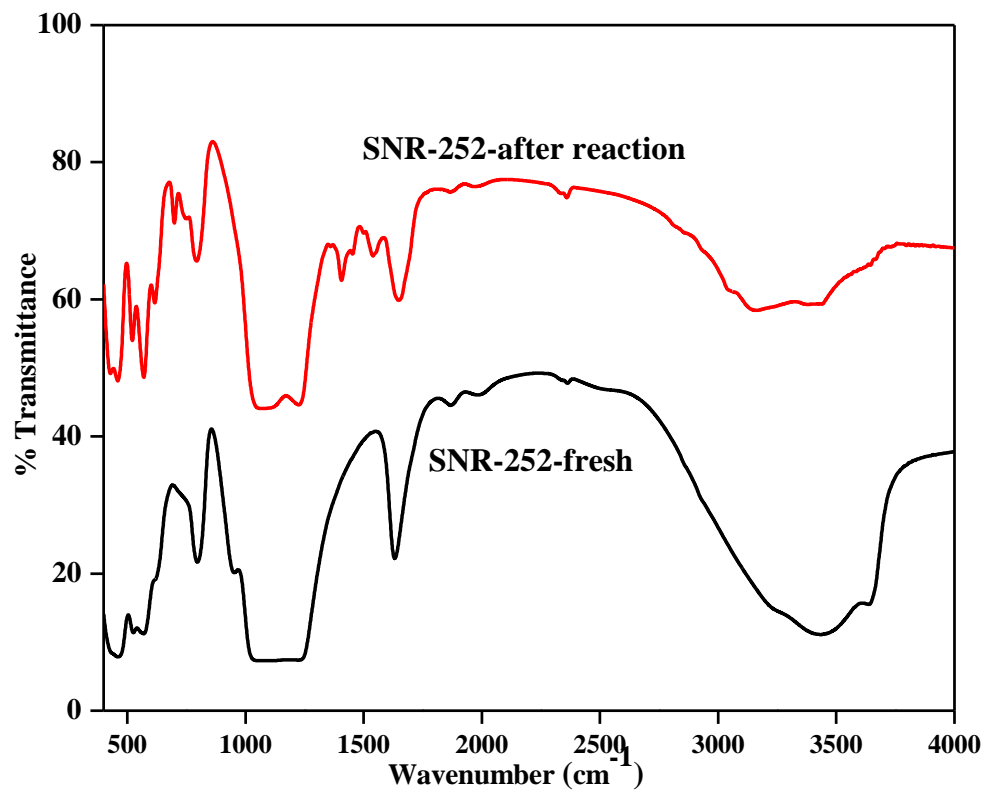
**General experimental procedure:** A mixture of amide (4 mmol), amine (4 mmol) and H- $\beta$  Zeolite (20 wt. %) was stirred in sealed tube at indicated temperature for indicated reaction time (See Table 1 and Schemes 2-4). After being cooled to room temperature, the reaction mixture was dissolved with DCM (20 mL). After removal of solvent, the crude reaction mixture left out was purified by silica gel (200-400mesh) column chromatography (dissolved in dichloromethane, eluted with dichloromethane and ethyl acetate). The yields are mentioned in tables 2-4. The catalyst H- $\beta$  Zeolite ( $\text{SiO}_2/\text{Al}_2\text{O}_3 = 24$ ) was procured from zeochem, Switzerland.

### The catalyst recycling experiment

The transamidation reaction was repeated up to four consecutive cycles with the H- $\beta$  Zeolite catalyst, which was recovered after each reaction. Reactions were carried out for 24-36 h. After completion of the reaction, the catalyst was filtered off, washed with dichloromethane and then dried in oven for 20-30 min and catalyst was reused accordingly.



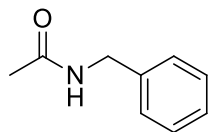
**Figure S1:** The XRD pattern of fresh and used H- $\beta$  Zeolite



**Figure S2:** The FT-IR Spectrum of before and after used H- $\beta$  Zeolite

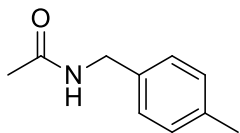
### Characterization data of the compounds

#### N-benzylacetamide (3a)



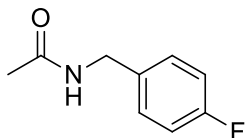
(Eluent: 20% EtOAc in DCM); white solid; 97% yield (578mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57-7.27 (m, 5H), 6.19(br s, 1H, NH), 4.46(d,  $J = 4.5$  Hz, 2H), 2.02 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 142.4, 127.8, 125.5, 43.1, 23.1.

**N-(4-methylbenzyl)acetamide(3b)**



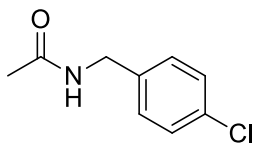
(Eluent: 20% EtOAc in DCM); white solid; 83% yield (541mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.18-7.15 (m, 4H), 5.70(br s, 1H, NH), 4.39 (d,  $J = 5.5$  Hz, 2H), 2.33 (s, 3H), 2.00 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.7, 131.2, 129.1, 123.3, 121.8, 37.5, 17.2, 15.0.

**N-(4-fluorobenzyl)acetamide(3c)**



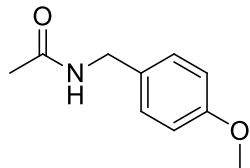
(Eluent: 20% EtOAc in DCM); white solid; 81% yield (538mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26-7.23 (m, 2H), 7.02(t,  $J = 8.5$  Hz, 2H), 5.86 (br s, 1H, NH), 4.39 (d,  $J = 6.0$  Hz, 2H), 2.01 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.8, 157.1, 155.1, 128.0, 123.4, 109.5, 109.3, 36.9, 17.1.

**N-(4-chlorobenzyl)acetamide (3d)**



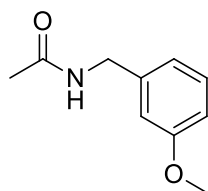
(Eluent: 20% EtOAc in DCM); white solid; 92% yield (674mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.26-7.17 (m, 4H), 5.62 (br s, 1H, NH), 4.42 (d,  $J = 5.5$  Hz, 2H), 2.32 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  169.3, 136.0, 135.4, 130.1, 128.2, 127.4, 125.8, 41.4, 22.7, 18.5.

**N-(4-methoxybenzyl)acetamide (3e)**



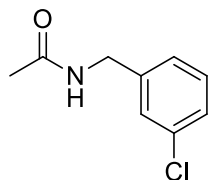
(Eluent: 20% EtOAc in DCM); white solid; 78% yield (559mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.21(d,  $J = 8.0$  Hz, 4H), 6.86(d,  $J = 8.5$  Hz, 2H), 5.81 (br s, 1H, NH), 4.35 (d,  $J = 5.5$  Hz, 2H), 3.79 (s, 3H), 1.99 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  169.8, 159.0, 130.3, 129.2, 114.0, 55.3, 43.2, 23.2.

**N-(3-methoxybenzyl)acetamide(3f)**



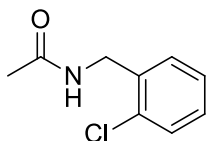
(Eluent: 20% EtOAc in DCM); white solid; 84% yield (601mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.24 (t,  $J = 8.0$  Hz, 1H), 6.84 (m, 3H), 6.23 (br s, 1H, NH), 4.36 (d,  $J = 5.5$  Hz, 2H), 3.77 (s, 3H), 1.98 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  169.7, 159.4, 139.4, 129.2, 119.5, 113.0, 112.4, 54.8, 43.2, 22.7.

**N-(3-chlorobenzyl)acetamide(3g)**



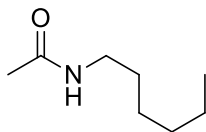
(Eluent: 20% EtOAc in DCM); white solid; 86% yield (631mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.19-7.09 (m, 4H), 7.08 (br, s, 1H, NH), 4.27 (s, 2H), 1.92 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 140.0, 133.7, 129.3, 127.0, 126.8, 125.1, 42.3, 22.3. HRMS calcd for  $\text{C}_{16}\text{H}_{18}\text{NO}_2$ : 256.1338, found: 256.1336.

**N-(2-chlorobenzyl)acetamide(3h)**



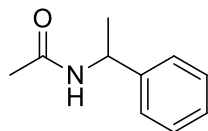
(Eluent: 20% EtOAc in DCM); white solid; 89% yield (655mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.39-7.36(m, 2H), 7.26-7.23 (m, 2H), 5.89 (s, br, 1H, NH), 4.53 (d,  $J = 6.0$  Hz, 2H), 2.02 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 135.6, 133.5, 130.0, 129.4, 128.8, 127.0, 41.4, 23.1.

**N-hexylacetamide (3i)**



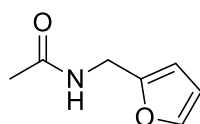
(Eluent: 20% EtOAc in DCM); colorless liquid; 81% yield (461mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.12 (s, 1H, NH), 3.23-3.19 (q,  $J = 6.5$  Hz, 3H), 2.45 (s, 1H), 1.97 (s, 3H), 1.50-1.47 (m, 2H), 1.29 (s, 4H), 0.89 (m, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 39.6, 31.4, 29.4, 26.5, 22.5, 13.9.

**N-(1-phenylethyl)acetamide(3j)**



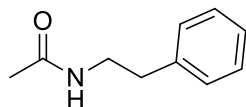
(Eluent: 20% EtOAc in DCM); white solid; 85% yield (557mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35-7.26 (m, 5H), 5.85 (br, s, 1H, NH), 5.15-5.09 (q,  $J=7.0\text{Hz}$ , 1H), 1.97 (s, 3H), 1.48 (d,  $J = 7.0$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  168.6, 142.8, 128.2, 126.9, 125.7, 48.3, 47.5, 22.9, 21.2.

**N-(furan-2-ylmethyl)acetamide (3k)**



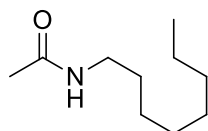
(Eluent: 20% EtOAc in DCM); colorless liquid; 78% yield (432mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.33 (br, 1H), 6.46 (br, s, 1H, NH), 6.31-6.30 (m, 1H), 6.21 (br, 1H), 4.39 (d,  $J = 5.5$  Hz, 2H), 1.98 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 151.3, 142.1, 110.4, 107.3, 36.5, 22.9.

**N-phenethylacetamide(3l)**



(Eluent: 20% EtOAc in DCM); white solid; 75% yield (492mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.32-7.18 (m, 5H), 5.68 (br, s, 1H, NH), 3.52 (q,  $J = 7.0$  Hz, 2H), 2.82 (t,  $J = 7.0$  Hz, 2H), 1.92 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.1, 138.9, 128.7, 128.6, 126.5, 40.6, 35.6, 23.2.

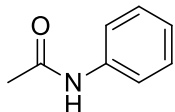
**N-octylacetamide (3m)**





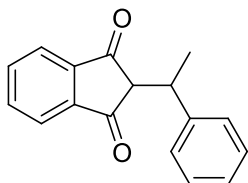
(Eluent: 20% EtOAc in DCM); white solid; 78% yield (536mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.82 (br, s, 1H, NH), 3.24 (q,  $J = 7.0$  Hz, 2H), 1.97 (s, 3H), 1.29-1.26 (br, 12H), 0.89 (t,  $J = 7.0$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.2, 39.6, 31.7, 29.5, 29.2, 29.1, 26.9, 23.1, 22.5, 14.0.

**N-phenylacetamide (3n)**



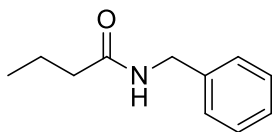
(Eluent: 20% EtOAc in DCM); white solid; 60% yield (326mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 (d,  $J = 8.0$  Hz, 2H), 7.41 (br, s, 1H, NH), 7.33 (t,  $J = 7.5$  Hz, 2H), 7.12 (t,  $J = 7.5$  Hz, 1H), 2.17 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  168.4, 137.8, 128.9, 124.3, 119.9, 24.5.

**N-(m-tolyl)acetamide (3o)**



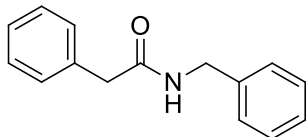
(Eluent: 20% EtOAc in DCM); white solid; 63% yield (374mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74 (br, s, 1H, NH), 7.34 (s, 1H), 7.28 (d,  $J = 8.0$  Hz, 1H), 7.18 (t,  $J = 7.5$  Hz, 1H), 6.91 (d,  $J = 7.5$  Hz, 1H), 2.30 (s, 3H), 2.13 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  168.2, 138.4, 137.5, 128.3, 124.6, 120.3, 116.7, 24.0, 21.0.

**N-benzylbutyramide (3p)**



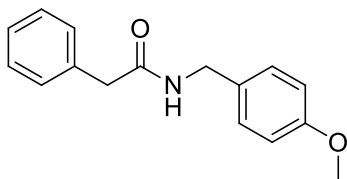
(Eluent: 20% EtOAc in DCM); white solid; 50% yield (355mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.31-7.25 (m, 5H), 5.95 (br, s, 1H, NH), 4.42 (d,  $J = 5.5$  Hz, 2H), 2.19 (t,  $J = 7.5$  Hz, 2H), 1.69 (q,  $J = 7.5$  Hz, 2H), 0.95 (t,  $J = 7.5$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  172.8, 138.4, 128.6, 127.7, 43.5, 38.6, 19.1, 13.7.

### **N-benzyl-2-phenylacetamide (3q)**



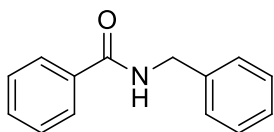
(Eluent: 20% EtOAc in DCM); white solid; 80% yield (724mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36-7.16 (m, 10H), 5.70 (br, s, 1H, NH), 4.41 (d,  $J = 5.5$  Hz, 2H), 3.62 (s, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.8, 138.1, 134.7, 129.4, 129.0, 1128.6, 127.48, 127.42, 43.8, 43.5.

### **N-(4-methoxybenzyl)-2-phenylacetamide(3r)**



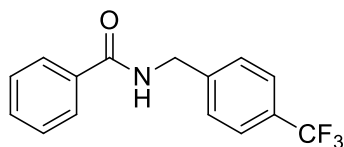
(Eluent: 20% EtOAc in DCM); white solid; 86% yield (882mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35-7.25 (m, 7H), 7.11 (d,  $J = 8.5$  Hz, 2H), 6.83 (d,  $J = 8.5$  Hz, 2H), 5.61 (br, s, 1H, NH), 4.34 (d,  $J = 5.5$  Hz, 2H), 3.78 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  170.7, 134.7, 129.5, 129.4, 129.1, 129.0, 128.8, 127.3, 114.0, 99.9, 55.2, 43.8, 43.1.

### **N-benzylbenzamide (4a)**



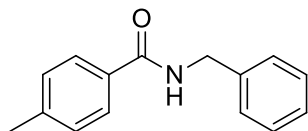
(Eluent: 20% EtOAc in DCM); white solid; 77% yield (647mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.79 (d,  $J = 7.5$  Hz, 2H), 7.50-7.30 (m, 8H), 6.51 (br, s, 1H, NH), 4.63 (d,  $J = 5.0$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.3, 138.2, 134.3, 131.5, 128.7, 128.5, 127.9, 126.9, 44.1.

**N-(4-(trifluoromethylbenzyl)benzamide(4b)**



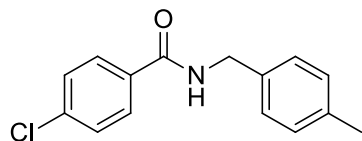
(Eluent: 20% EtOAc in DCM); white solid; 68% yield (681mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81 (d,  $J = 8.5$  Hz, 2H), 7.61 (d,  $J = 7.5$  Hz, 2H), 7.54-7.43 (m, 5H), 6.53 (br, s, 1H, NH), 4.71 (d,  $J = 4.0$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.5, 142.3, 134.0, 131.8, 128.7, 127.9, 126.9, 125.7, 43.5.

**N-benzyl-4-methylbenzamide (4c)**



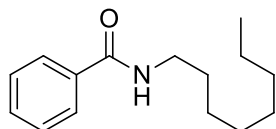
(Eluent: 20% EtOAc in DCM); white solid; 76% yield (682mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (d,  $J = 8.0$  Hz, 2H), 7.35-7.21 (m, 7H), 6.38 (br, s, 1H, NH), 4.64 (d,  $J = 5.5$  Hz, 2H), 2.39 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.2, 141.9, 138.3, 131.5, 129.2, 128.7, 127.9, 127.5, 126.9, 44.0, 21.4.

**4-chloro-N-(4-methylbenzyl)benzamide (4d)**



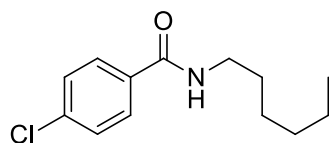
(Eluent: 20% EtOAc in DCM); white solid; 71% yield (741mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 (d,  $J = 8.5$  Hz, 2H), 7.40 (d,  $J = 8.5$  Hz, 2H), 7.26-7.23 (t,  $J = 6.5$  Hz, 2H), 7.17 (d,  $J = 8.0$  Hz, 2H), 6.32 (br, s, 1H, NH), 4.59 (d,  $J = 5.5$  Hz, 2H), 2.34 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  166.1, 137.7, 137.4, 134.8, 132.8, 129.4, 128.8, 128.3, 127.9, 44.0, 30.8, 21.0.

#### **N-octylbenzamide(4e)**



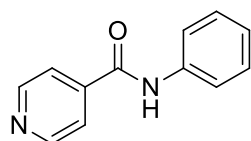
(Eluent: 20% EtOAc in DCM); white solid; 85% yield (796mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (d,  $J = 6.0$  Hz, 2H), 7.47-7.41 (m, 3H), 6.29 (br, s, 1H, NH), 3.43 (br, 2H), 1.60 (br, 2H), 1.28 (br, 10H), 0.87 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.5, 134.8, 131.2, 128.5, 126.8, 40.1, 31.7, 29.6, 29.29, 29.21, 27.0, 22.6, 14.0.

#### **4-chloro-N-hexylbenzamide (4f)**



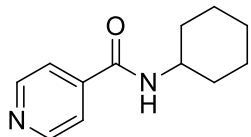
(Eluent: 20% EtOAc in DCM); white solid; 72% yield (689mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J = 8.5$  Hz, 2H), 7.40 (d,  $J = 8.5$  Hz, 2H), 6.17 (br, s, 1H, NH), 3.45 (q,  $J = 7.0$  Hz, 2H), 1.72-1.57 (m, 3H), 1.37-1.31 (m, 5H), 0.89 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  166.5, 137.4, 133.2, 128.7, 128.3, 40.2, 31.4, 29.5, 26.6, 22.5, 14.0.

#### **N-phenylisonicotinamide (4g)**



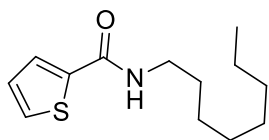
(Eluent: 20% EtOAc in DCM); Grey solid; 55% yield (433mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.78 (s, 2H), 8.02 (br, s, 1H, NH), 7.71 (d,  $J = 5.5$  Hz, 2H), 7.65 (d,  $J = 8.0$  Hz, 2H), 7.41 (t,  $J = 7.5$  Hz, 2H), 7.21 (t,  $J = 7.5$  Hz, 1H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.8, 150.7, 142.1, 137.2, 129.2, 125.2, 120.8, 120.4.

**N-cyclohexylisonicotinamide(4h)**



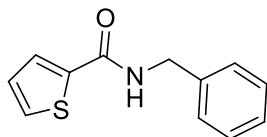
(Eluent: 20% EtOAc in DCM); white solid; 67% yield (552mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.72-8.71 (m, 2H), 7.59 (d,  $J = 4.5$  Hz, 2H), 6.14 (br, s, 1H, NH), 4.00-3.94 (m, 1H), 2.04 (br, 2H), 1.79-1.65 (m, 4H), 1.46-1.39 (m, 2H), 1.28-1.22 (m, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  164.6, 150.5, 142.1, 120.8, 49.0, 33.0, 25.4, 24.8.

**N-octylthiophene-2-carboxamide(4i)**



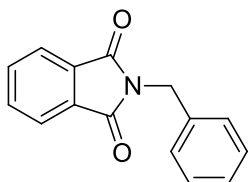
(Eluent: 20% EtOAc in DCM); white solid; 71% yield (682mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (br, 1H), 7.44 (d,  $J = 5.0$  Hz, 1H), 7.06 (t,  $J = 5.0$  Hz, 1H), 6.24 (br, s, 1H, NH), 3.43 (q,  $J = 6.5$  Hz, 2H), 1.62 (q,  $J = 7.0$  Hz, 2H), 1.35-1.26 (m, 10H), 0.88 (t,  $J = 6.0$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.5, 138.9, 129.1, 127.4, 127.1, 39.6, 31.3, 29.2, 28.8, 28.7, 26.5, 22.1, 13.6.

**N-benzylthiophene-2-carboxamide (4j)**



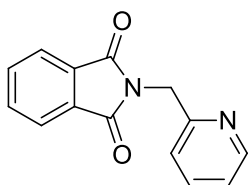
(Eluent: 20% EtOAc in DCM); white solid; 63% yield (550mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50-7.47 (br, 2H), 7.40-7.25 (br, 5H), 7.07-7.06 (m, 1H), 6.26 (br, s, 1H, NH), 4.63 (d,  $J = 6.0$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  161.7, 138.7, 138.0, 130.0, 128.8, 128.1, 127.9, 127.69, 127.64, 44.0.

**(2-benzylisoindoline-1,3-dione(4k))**



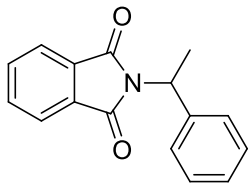
(Eluent: 20% EtOAc in DCM); white solid; 83% yield (786mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.85-7.83 (m, 2H), 7.70-7.69 (m, 2H), 7.44 (d,  $J = 7.0$  Hz, 2H), 7.33-7.26(m, 3H), 4.84(s, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  168.0, 136.3, 133.9, 132.1, 128.6, 128.5, 127.8, 123.3, 41.5.

**2-(pyridin-2-ylmethyl)isoindoline-1,3-dione (4l)**



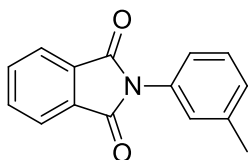
(Eluent: 20% EtOAc in DCM); Yellow solid; 82% yield (777mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.52 (d,  $J = 4.5$  Hz, 1H), 7.89-7.88 (m, 2H), 7.74-7.72 (m, 2H), 7.65-7.63 (t,  $J = 7.5$  Hz, 1H), 7.28 (t,  $J = 8.0$  Hz, 1H), 7.17 (q,  $J = 5.0$  Hz, 1H), 5.02 (s, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 155.3, 149.6, 136.6, 134.0, 132.2, 123.4, 122.4, 121.5, 42.9.

**2-(1-phenylethyl)isoindoline-1,3-dione (4m)**



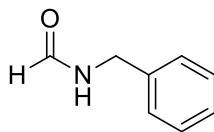
(Eluent: 20% EtOAc in DCM); white solid; 91% yield (910 mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.80-7.78 (m, 2H), 7.68-7.66 (m, 2H), 7.51 (d,  $J = 7.5$  Hz, 2H), 7.34 (t,  $J = 7.5$  Hz, 2H), 7.26 (t,  $J = 7.0$  Hz, 1H), 5.59 (q,  $J = 7.5$  Hz, 1H), 1.93 (d,  $J = 7.5$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  168.1, 140.3, 133.9, 132.0, 128.4, 127.6, 127.4, 123.1, 49.6, 17.5.

**2-(*m*-tolyl)isoindoline-1,3-dione(4n)**



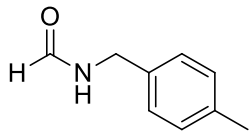
(Eluent: 20% EtOAc in DCM); yellow solid; 50% yield (478mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96-7.94 (m, 2H), 7.79-7.78 (m, 2H), 7.40 (t,  $J = 7.5$  Hz, 1H), 7.25 (t,  $J = 7.5$  Hz, 3H), 2.42 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  167.4, 139.1, 134.3, 131.8, 129.0, 128.9, 127.2, 123.7, 21.4.

**N-benzylformamide (4o)**



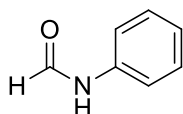
(Eluent: 20% EtOAc in DCM); white solid; 99% yield (532mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.19 (s, 1H), 7.32-7.27 (m, 5H), 6.27 (br, s, 1H, NH), 4.44 (d,  $J = 6.0$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  160.6, 138.9, 137.2, 128.3, 127.3, 41.7.

**N-(4-methylbenzyl)formamide(4p)**



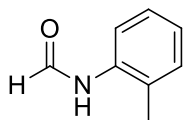
(Eluent: 20% EtOAc in DCM); white solid; 82% yield (491mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.24 (s, 1H), 7.19 (q,  $J = 8.0$  Hz, 4H), 5.81 (br, s, 1H, NH), 4.44 (d,  $J = 6.0$  Hz, 2H), 2.33 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  160.8, 137.4, 134.5, 129.4, 127.8, 45.3, 41.9, 30.9, 21.0.

**N-phenylformamide (4q)**



(Eluent: 20% EtOAc in DCM); white solid; 64% yield (310mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.69 (s, 1H), 7.55 (d,  $J = 7.5$  Hz, 1H), 7.35-7.08 (m, 5H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.1, 159.6, 137.0, 136.8, 129.7, 129.0, 125.2, 124.7, 120.1, 118.8.

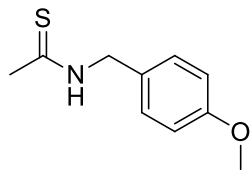
**N-o-tolylformamide(4r)**



(Eluent: 20% EtOAc in DCM); white solid; 65% yield (353mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.54 (s, 1H), 8.16 (br, s, 1H, NH), 7.22-7.14 (m, 4H), 2.31 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  163.3, 159.1, 135.0, 131.2, 130.5, 129.7, 127.1, 126.8, 126.0, 125.5, 123.0, 120.7, 17.6.

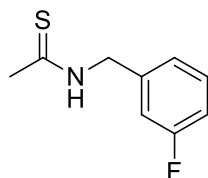
**N-(4-methoxybenzyl)ethanethioamide (5a)**





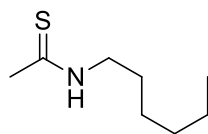
(Eluent: 5% EtOAc in DCM); yellow solid; 74% yield (577mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (br, s, 1H, NH), 7.26 (d,  $J = 8.5$  Hz, 2H) 6.89 (d,  $J = 8.5$  Hz, 2H), 4.72 (d,  $J = 5.0$  Hz, 2H), 3.80 (s, 3H), 2.55 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  200.4, 159.4, 129.8, 128.0, 114.2, 60.4, 55.3, 50.1, 34.0.

**N-(3-fluorobenzyl)ethanethioamide (5b)**



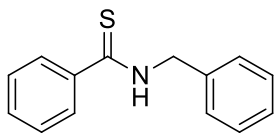
(Eluent: 5% EtOAc in DCM); yellow liquid; 70% yield (514mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63 (br, s, 1H, NH), 7.34-7.27 (m, 1H) 7.11 (d,  $J = 8.0$  Hz, 1H), 7.04-6.99 (m, 2H), 4.83 (d,  $J = 5.5$  Hz, 2H), 2.58 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  201.3, 163.9, 161.9, 138.56, 138.51, 130.5, 130.4, 123.8, 115.1, 115.0, 114.9, 60.4, 49.7, 34.0.

**N-hexylethanethioamide (5c), (new compound).**



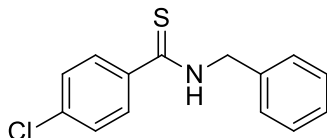
(Eluent: 5% EtOAc in DCM); yellow liquid; 63% yield (401mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  3.65 (q,  $J = 7.0$  Hz, 2H), 2.55 (s, 3H), 1.68-1.58 (m, 2H) 1.38-1.31 (m, 7H), 0.89 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  199.5, 45.5, 33.1, 30.3, 26.9, 25.6, 21.4, 12.9. HRMS calcd for  $\text{C}_8\text{H}_{18}\text{NS}$ : 160.1160, found: 160.1160.

### N-benzylbenzothioamide(5d)



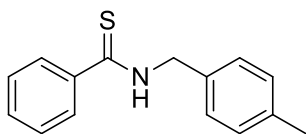
(Eluent: 10% EtOAc in DCM); yellow solid; 89% yield (805mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75 (d,  $J = 8.0$  Hz, 2H), 7.46-7.34 (m, 8H), 4.99 (d,  $J = 5.0$  Hz, 2H) 1.57 (s, 1H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  193.1, 135.6, 130.1, 125.1, 123.0, 122.4, 122.3, 122.2, 120.6, 45.0.

### N-benzyl-4-chlorobenzothioamide (5e)



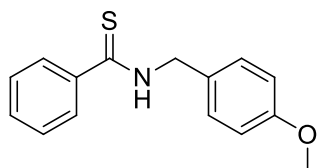
(Eluent: 10% EtOAc in DCM); yellow solid; 80% yield (839mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.73 (d,  $J = 7.5$  Hz, 2H), 7.44 (d,  $J = 7.5$  Hz, 1H), 7.36-7.30 (m, 3H), 6.90 (d,  $J = 6.5$  Hz, 2H) 4.89 (d,  $J = 5.0$  Hz, 2H), 3.80 (s, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.7, 159.5, 141.6, 131.1, 129.8, 128.5, 126.6, 114.3, 55.3, 50.6.

### N-(4-methylbenzyl)benzothioamide (5f)



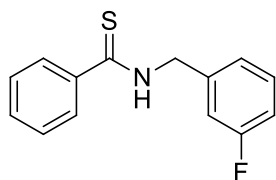
(Eluent: 10% EtOAc in DCM); yellow solid; 86% yield (831mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.75-7.58 (m, 3H), 7.54-7.43 (m, 1H), 7.40-7.35 (m, 2H), 7.29-7.25 (m, 2H) 7.20-7.15 (m, 2H), 4.94 (d,  $J = 5.0$  Hz, 2H), 2.36 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.9, 141.6, 138.1, 133.1, 131.1, 129.7, 128.5, 128.4, 126.6, 50.9, 21.1.

**N-(4-methoxybenzyl)benzothioamide (5g)**



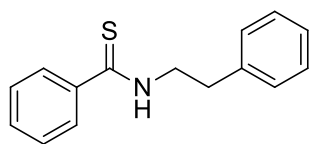
(Eluent: 10% EtOAc in DCM); yellow solid; 76% yield (779mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.74 (d,  $J = 7.0\text{Hz}$ , 2H), 7.66 (br, s, 1H, NH), 7.45 (t,  $J = 7.5\text{ Hz}$ , 1H), 7.37 (t,  $J = 8.0\text{ Hz}$ , 2H) 7.33 (t,  $J = 9.0\text{ Hz}$ , 2H), 6.92 (d,  $J = 8.5\text{ Hz}$ , 2H), 4.91 (d,  $J = 5.0\text{ Hz}$ , 2H), 3.81 (s, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  198.8, 159.5, 141.6, 131.1, 129.8, 128.5, 126.6, 114.3, 55.3, 50.6.

**N-(3-fluorobenzyl)benzothioamide (5h)**



(Eluent: 10% EtOAc in DCM); yellow solid; 82% yield (806mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81 (br, s, 1H, NH), 7.75 (d,  $J = 7.5\text{Hz}$ , 2H), 7.47 (t,  $J = 7.0\text{ Hz}$ , 1H), 7.39-7.34 (m, 3H), 7.16-7.01 (m, 3H) 5.00 (d,  $J = 5.5\text{ Hz}$ , 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  199.5, 163.9, 162.0, 141.4, 138.68, 138.62, 131.3, 128.5, 126.6, 123.7, 115.2, 115.0, 50.1.

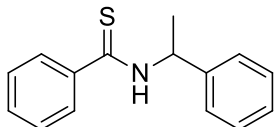
**N-phenethylbenzothioamide (5i), (new compound).**



(Eluent: 10% EtOAc in DCM); yellow solid; 85% yield (817mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.62 (d,  $J = 7.5\text{Hz}$ , 2H), 7.56 (br, s, 1H, NH), 7.43 (t,  $J = 7.5\text{ Hz}$ , 1H), 7.35 (q,  $J = 7.0\text{ Hz}$ , 4H),

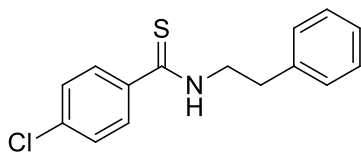
7.26 (d,  $J=8.0$  Hz, 3H) 4.10 (7,  $J = 7.0$  Hz, 2H), 3.08 (t,  $J = 7.0$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  199.2, 141.8, 138.2, 131.0, 128.8, 128.7, 128.4, 126.5, 47.4, 33.8. HRMS calcd for  $\text{C}_{15}\text{H}_{16}\text{NS}$ : 242.1003, found: 242.1005.

**N-(1-phenylethyl) benzothioamide (5j), (new compound).**



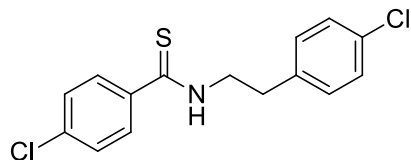
(Eluent: 10% EtOAc in DCM); yellow solid; 72% yield (693mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.72 (d,  $J = 7.5\text{Hz}$ , 3H), 7.44-7.25 (m, 8H), 5.92 (t,  $J = 7.5$  Hz, 1H), 1.71 (d,  $J = 7.0$  Hz, 3H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  192.0, 136.0, 135.3, 125.0, 122.8, 122.4, 121.8, 120.6, 120.5, 49.0, 14.1. HRMS calcd for  $\text{C}_{15}\text{H}_{16}\text{NS}$ : 242.1003, found: 242.0993.

**4-chloro-N-phenethylbenzothioamide (5k), (new compound).**



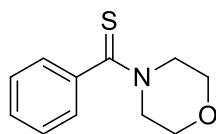
(Eluent: 10% EtOAc in DCM); yellow solid; 79% yield (872mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57 (d,  $J = 8.5\text{Hz}$ , 2H), 7.49 (br, s, 1H, NH), 7.36-7.25 (m, 7H), 4.11 (q,  $J = 7.0$  Hz, 2H), 3.09 (t,  $J = 6.5$  Hz, 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  191.7, 134.0, 132.0, 131.2, 122.8, 122.68, 122.60, 121.8, 120.9, 41.4, 27.7.

**4-chloro-N-(4-chlorophenethyl)benzothioamide, (5l) (new compound).**



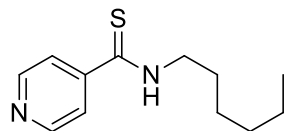
(Eluent: 20% EtOAc in DCM); yellow solid; 70% yield (871mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.58 (d,  $J = 8.5\text{Hz}$ , 2H), 7.52 (br, s, 1H, NH), 7.33 (t,  $J = 8.0\text{ Hz}$ , 4H), 7.19 (d,  $J = 8.5\text{ Hz}$ , 2H), 4.08 (q,  $J = 7.0\text{ Hz}$ , 2H), 3.07 (t,  $J = 7.0\text{ Hz}$ , 2H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  191.9, 133.9, 131.3, 130.5, 126.7, 124.0, 122.9, 122.6, 121.7, 41.2, 27.0. HRMS calcd for  $\text{C}_{15}\text{H}_{14}\text{NSCl}_2$ : 310.0224, found: 310.0238.

**morpholino(phenyl)methanethione(5m)**



(Eluent: 10% EtOAc in DCM); yellow solid; 68% yield (561mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30-7.25 (m, 3H), 7.21-7.18 (m, 2H), 4.37 (t,  $J = 5.0\text{ Hz}$ , 2H), 3.81 (t,  $J = 4.5\text{ Hz}$ , 2H), 3.56-3.51 (m, 4H).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  201.0, 142.4, 128.5, 125.8, 66.7, 66.5, 52.4, 49.5.

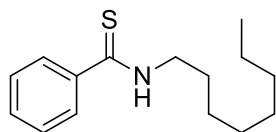
**N-hexylpyridine-4-carbothioamide (5n),(new compound).**



(Eluent: 10% EtOAc in DCM); yellow liquid; 69% yield (610mg);  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.51 (d,  $J = 5.0\text{ Hz}$ , 2H), 8.43 (br, s, 1H, NH), 7.51 (d,  $J = 6.0\text{ Hz}$ , 2H), 3.81 (q,  $J = 7.5\text{ Hz}$ , 2H), 1.78 (q,  $J = 7.5\text{ Hz}$ , 2H), 1.43-1.32 (m, 6H), 0.91 (t,  $J = 7.0\text{ Hz}$ , 3H).  $^{13}\text{C}$  NMR (125 MHz,

CDCl<sub>3</sub>)  $\delta$  195.2, 148.8, 147.7, 119.7, 45.9, 30.3, 26.8, 25.7, 21.4, 12.9. HRMS calcd for C<sub>12</sub>H<sub>19</sub>N<sub>2</sub>S: 223.1269, found: 223.1263.

**N-octylbenzothioamide (5o), (new compound).**



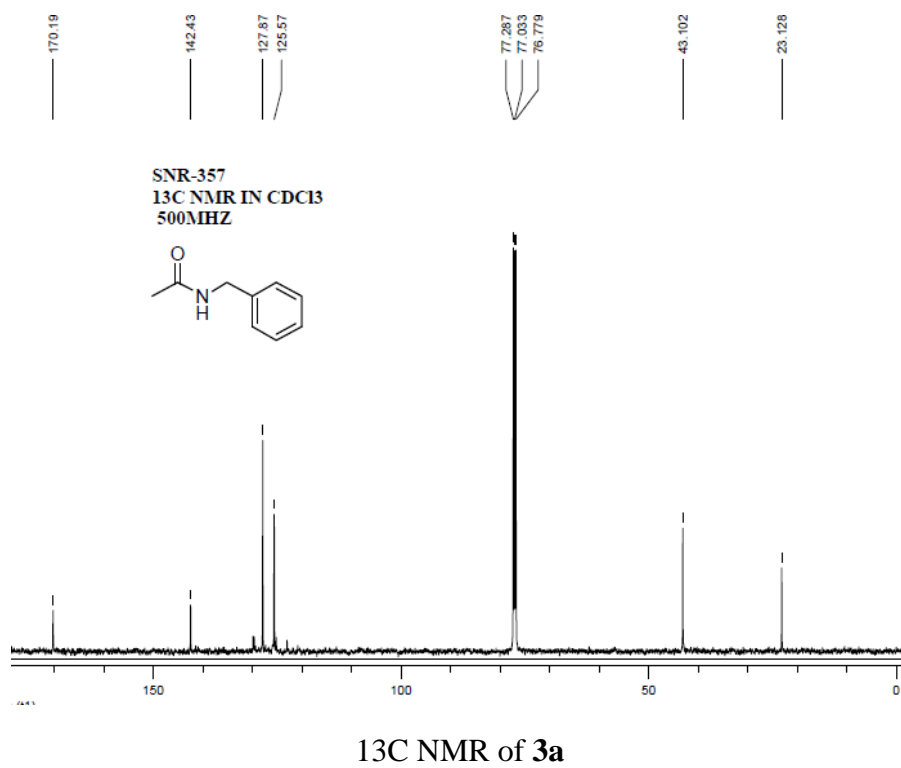
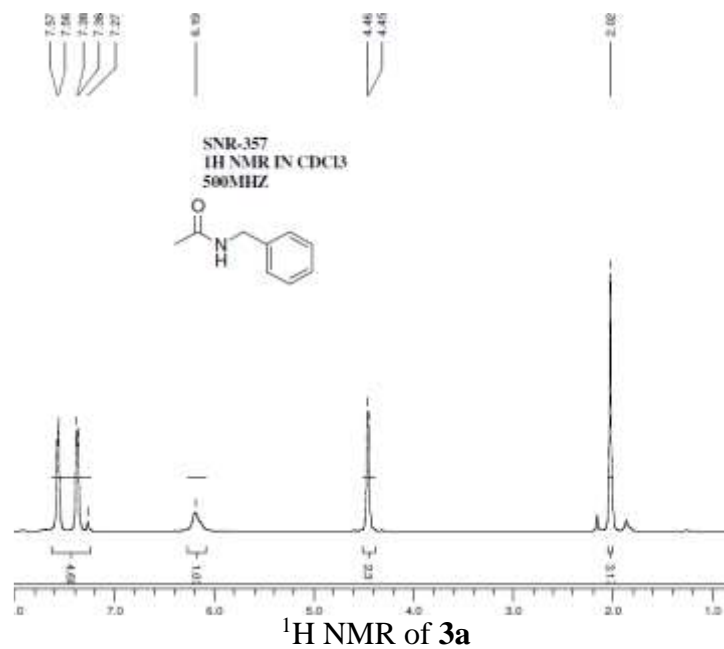
(Eluent: 10% EtOAc in DCM); Brown liquid; 68% yield (675mg); <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.72 (d, *J* = 8.0 Hz, 2H), 7.59 (br, s, 1H, NH), 7.45 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.5 Hz, 2H), 3.81 (q, *J* = 7.5 Hz, 2H), 1.77 (q, *J* = 7.5 Hz, 2H), 1.41-1.28 (m, 10H), 0.89 (t, *J* = 6.0 Hz, 3H). <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)  $\delta$  198.0, 141.0, 127.4, 125.5, 45.9, 30.7, 28.2, 28.1, 27.1, 26.0, 21.6, 13.0. HRMS calcd for C<sub>15</sub>H<sub>24</sub>NS: 250.1629, found: 250.1621.

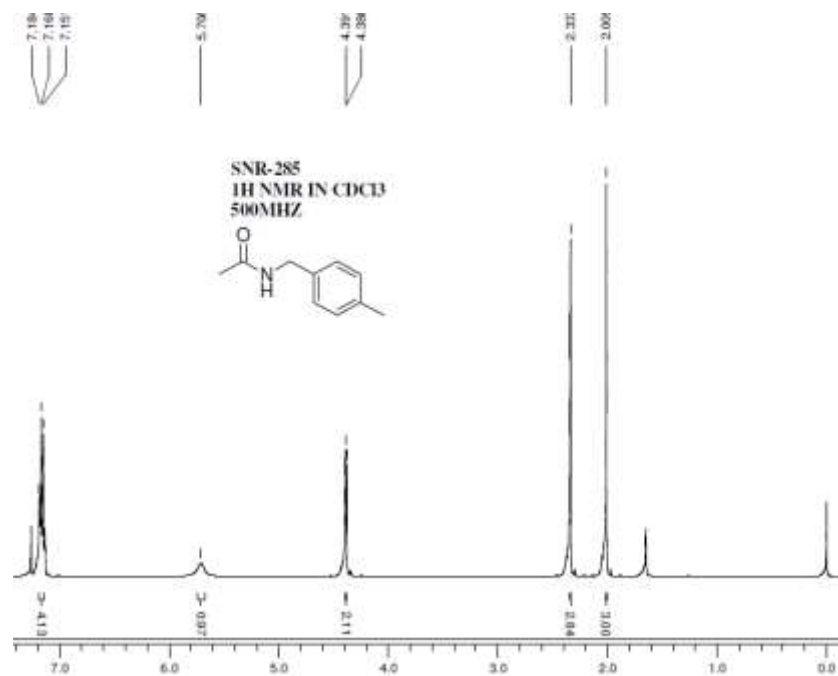
**References:** 1. Lillian, B-F.; Andrea, O-P.; Diego, G.-S, *J. Org. Chem.*, 2014, **79**, 4544.

2. J.W. Wu, Y.D. Wu, J.J. Dai, H.J. Xua, *Adv. Synth. Catal.*, 2014, **356**, 2429.

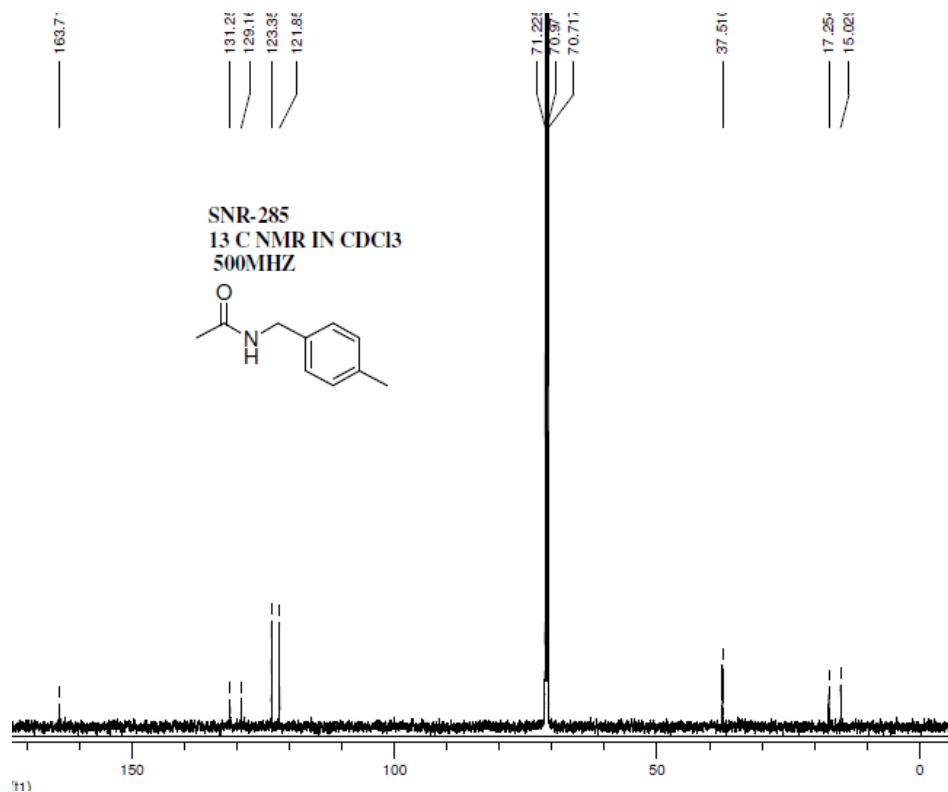
3. O.P. Andrea, G.S. Diego, *Tetrahedron Letters.*, 2015, **56**, 4308.

**Copies of <sup>1</sup>H and <sup>13</sup>C NMR spectra**



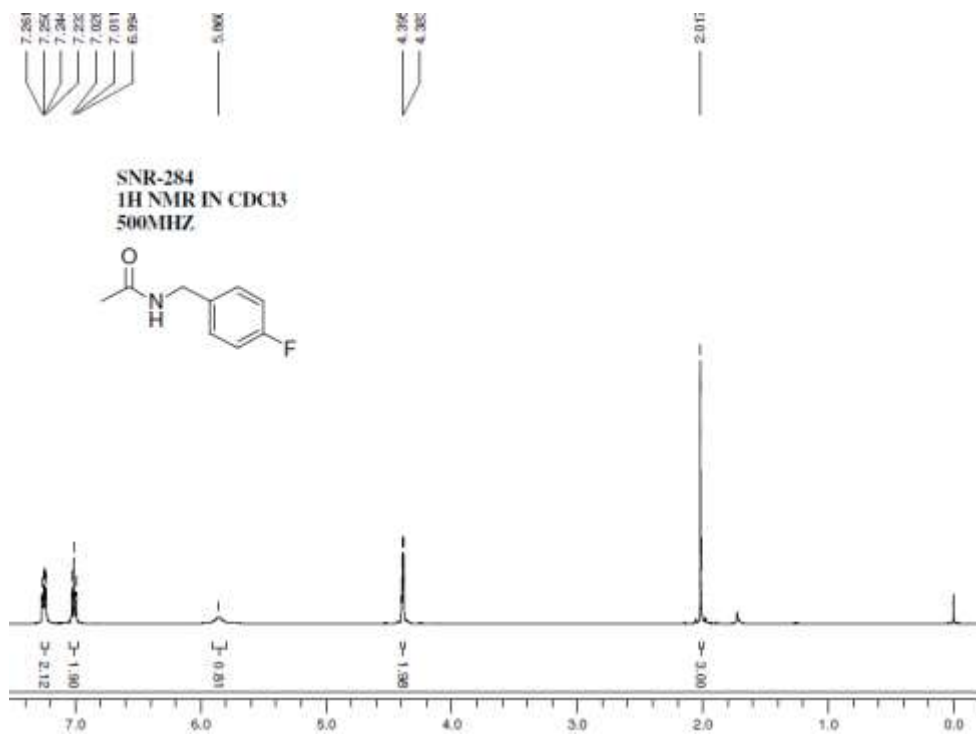


1H NMR of **3b**

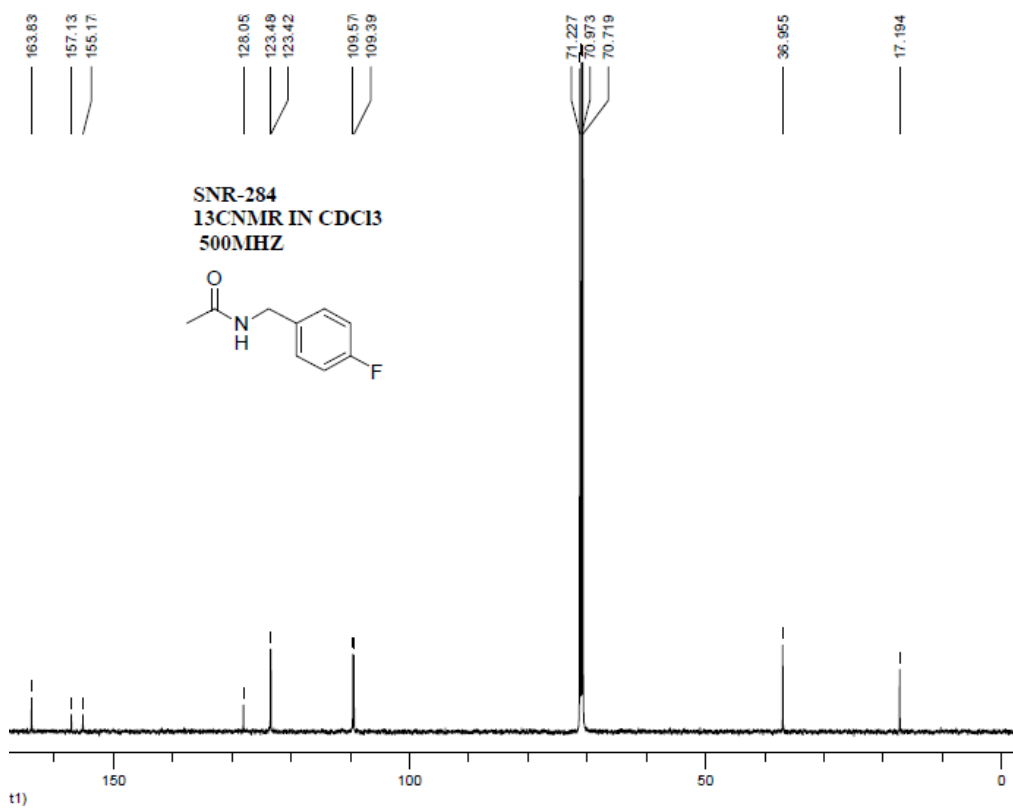


13C NMR of **3b**

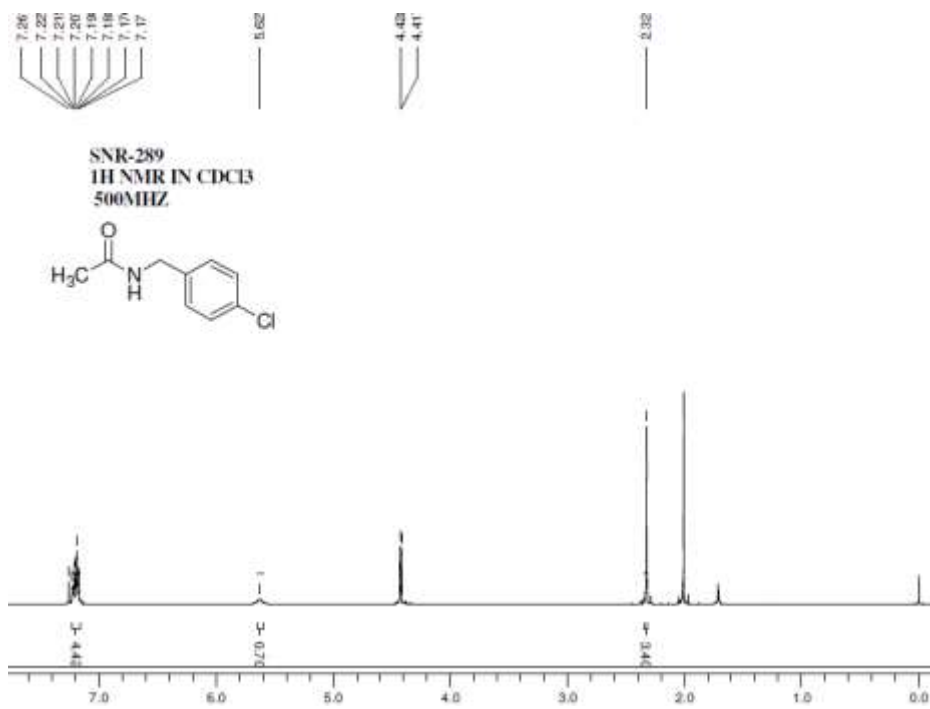




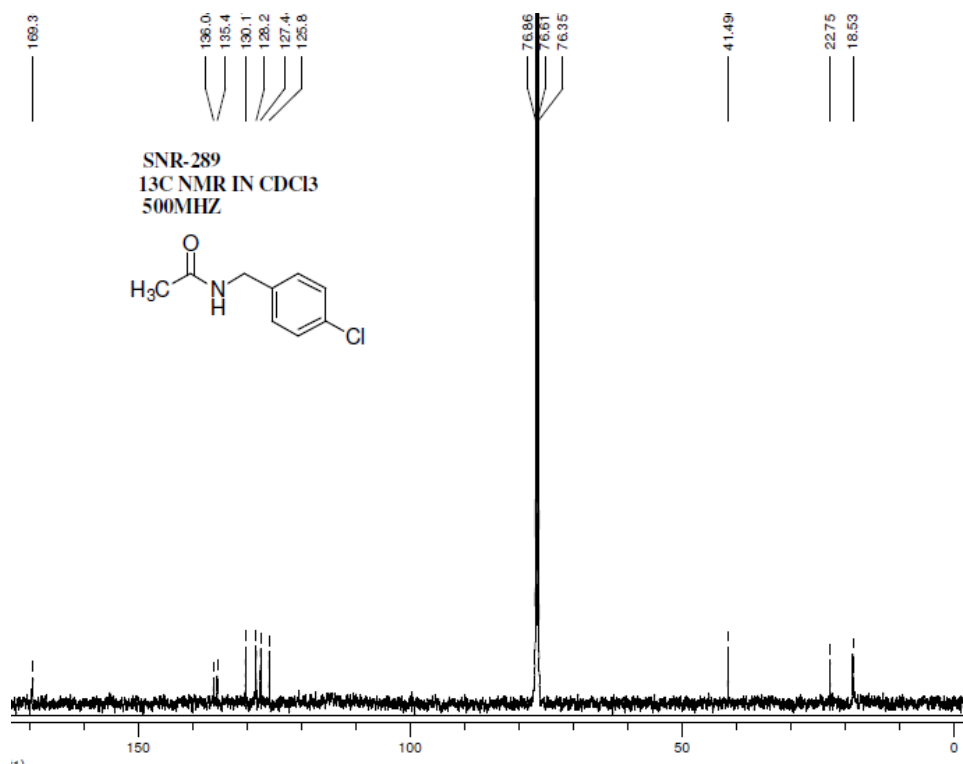
1H NMR of **3c**



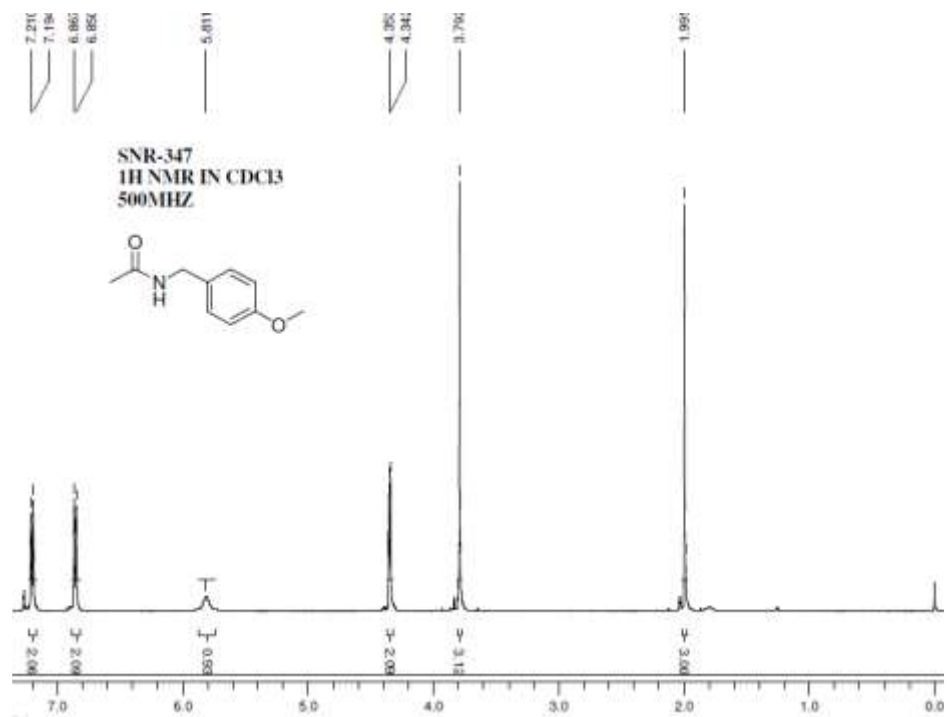
13C NMR of **3c**



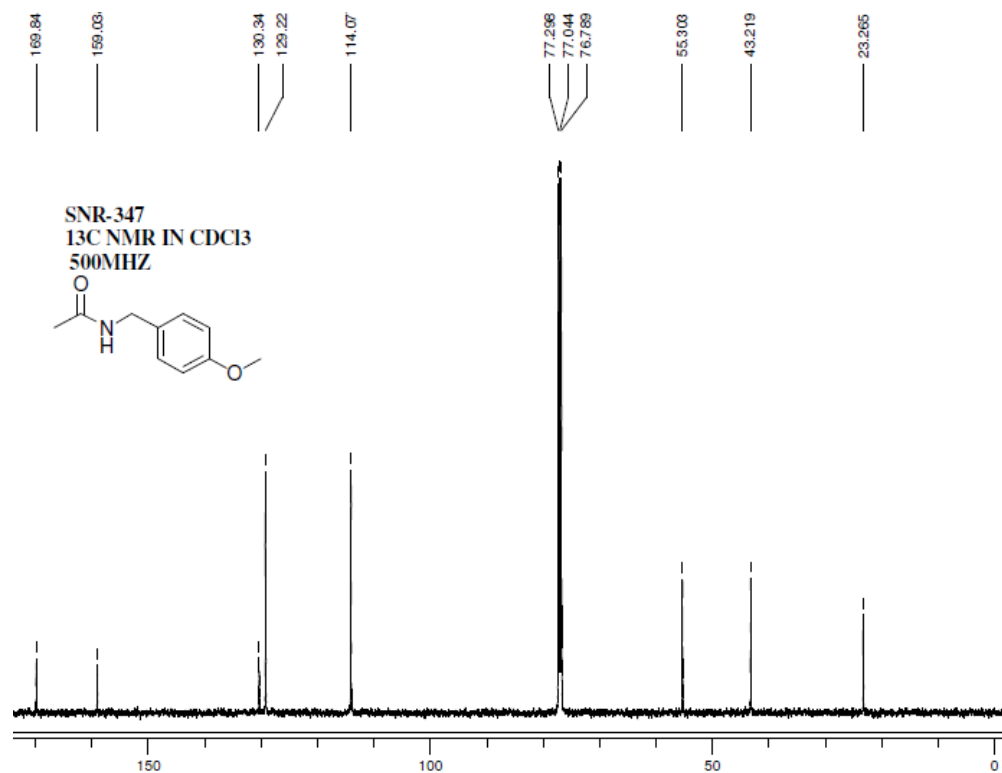
1H NMR of 3d



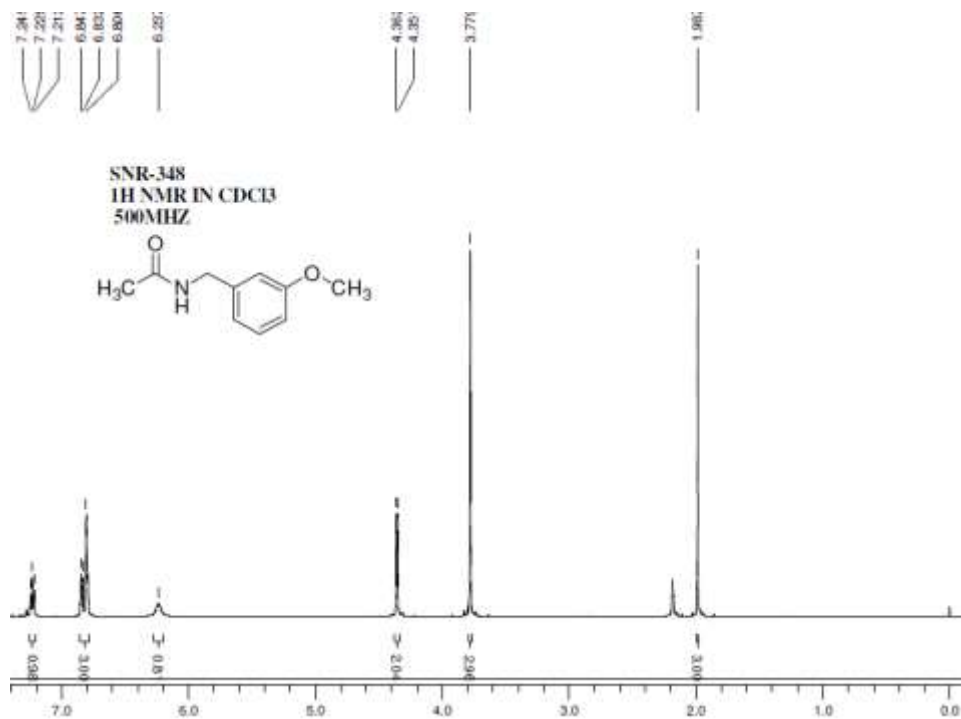
13C NMR of 3d



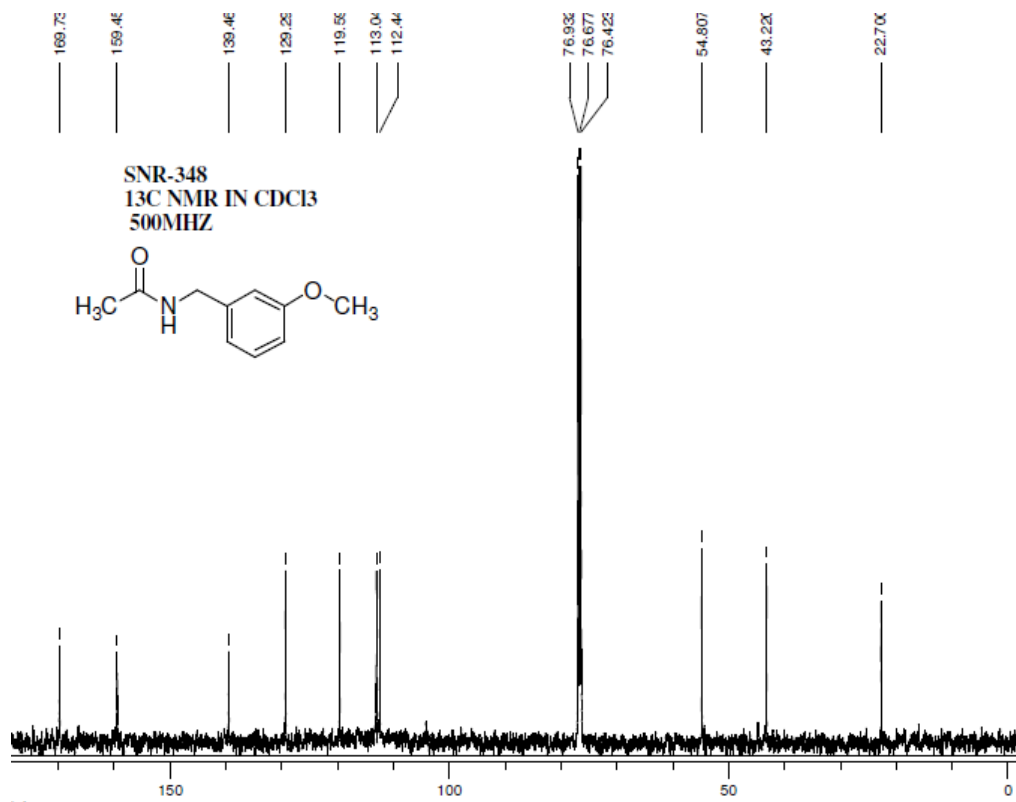
1H NMR of 3e



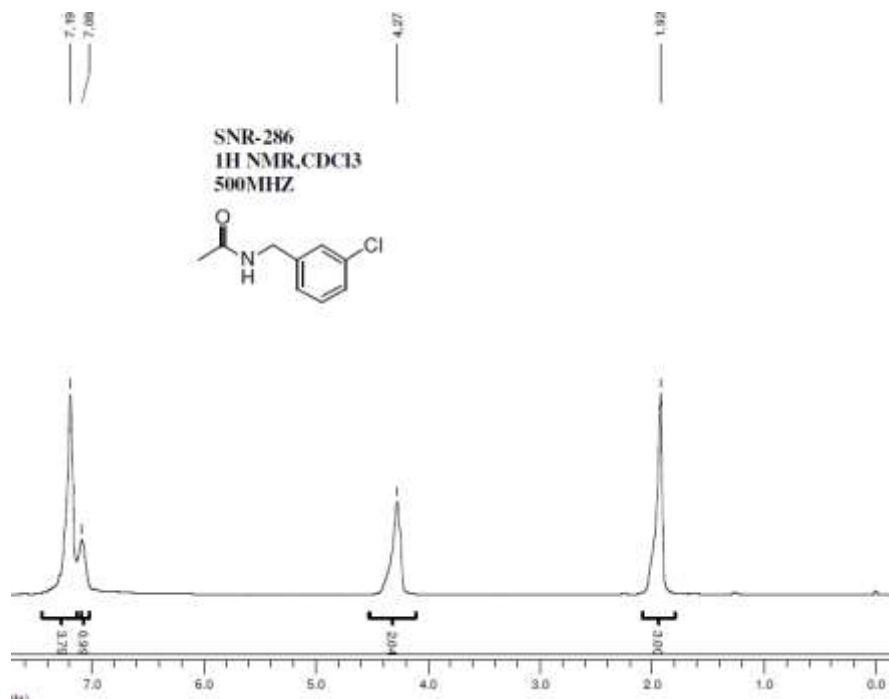
13C NMR of 3e



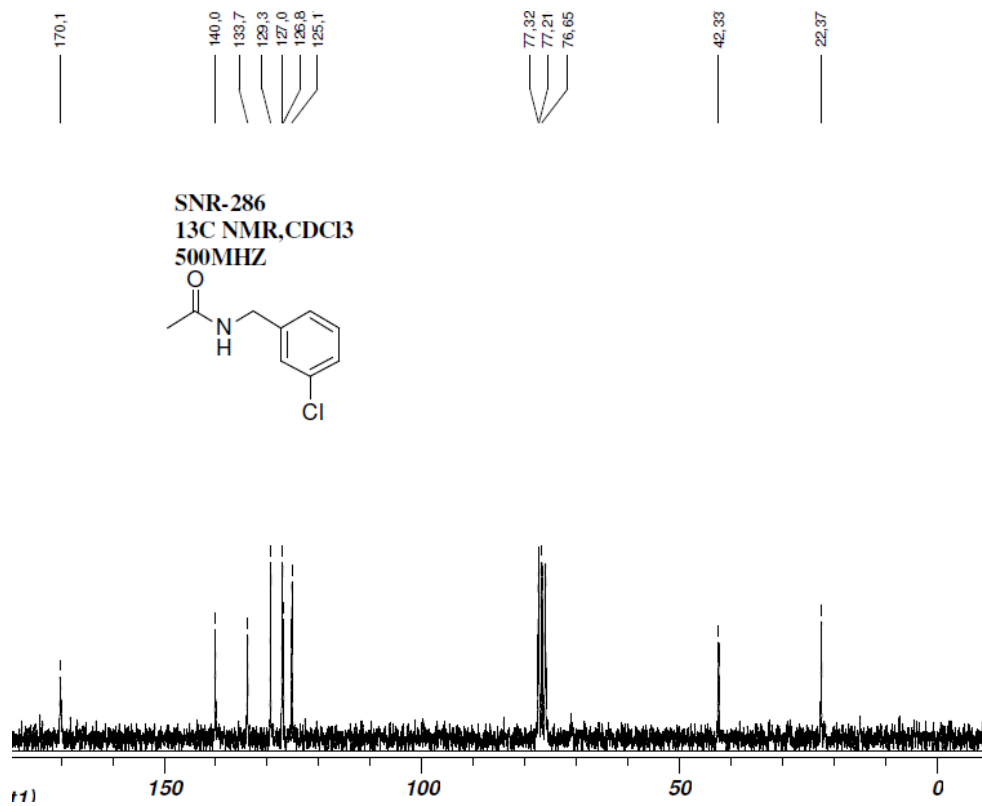
1H NMR of **3f**



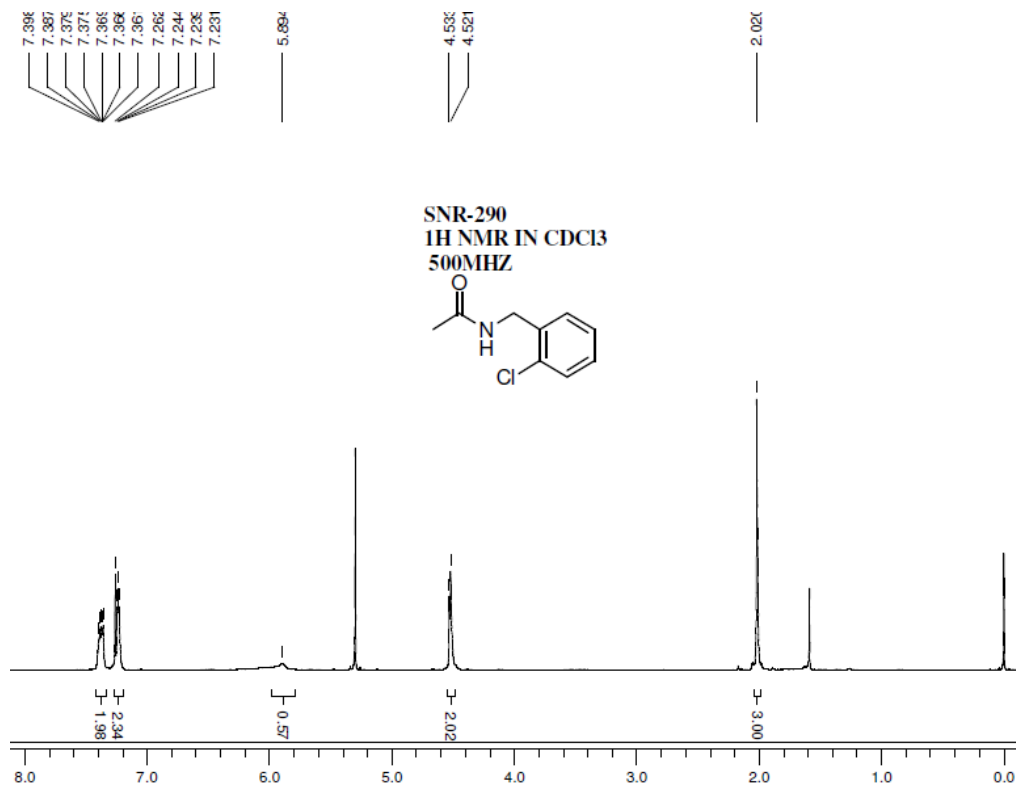
13C NMR of **3f**



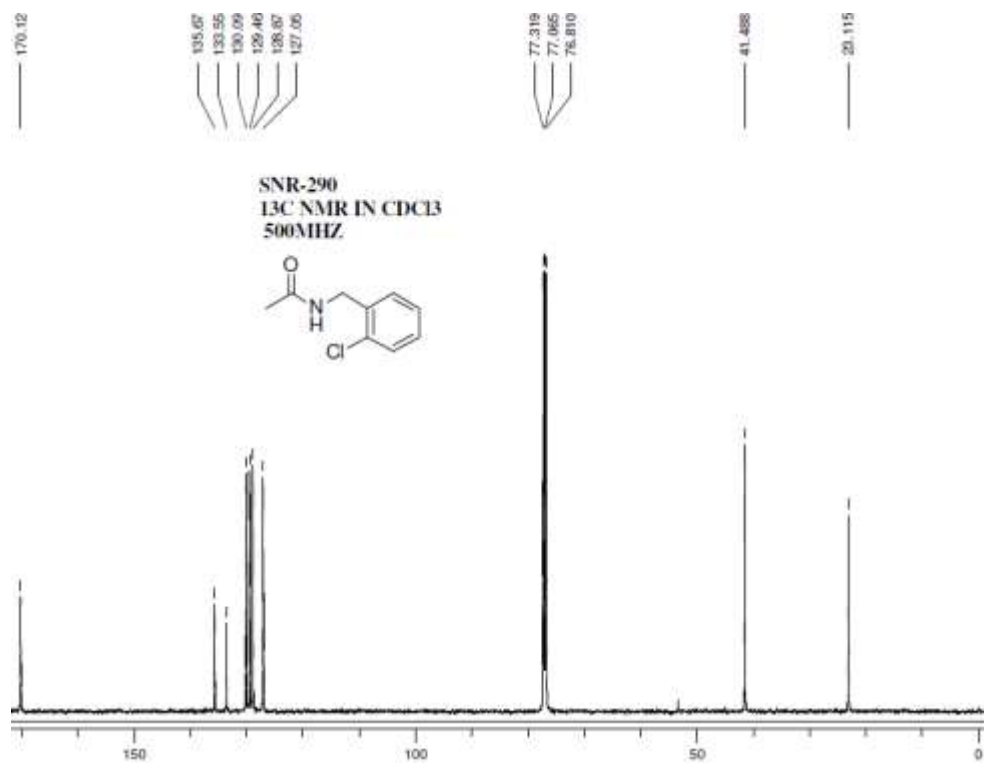
1H NMR of **3g**



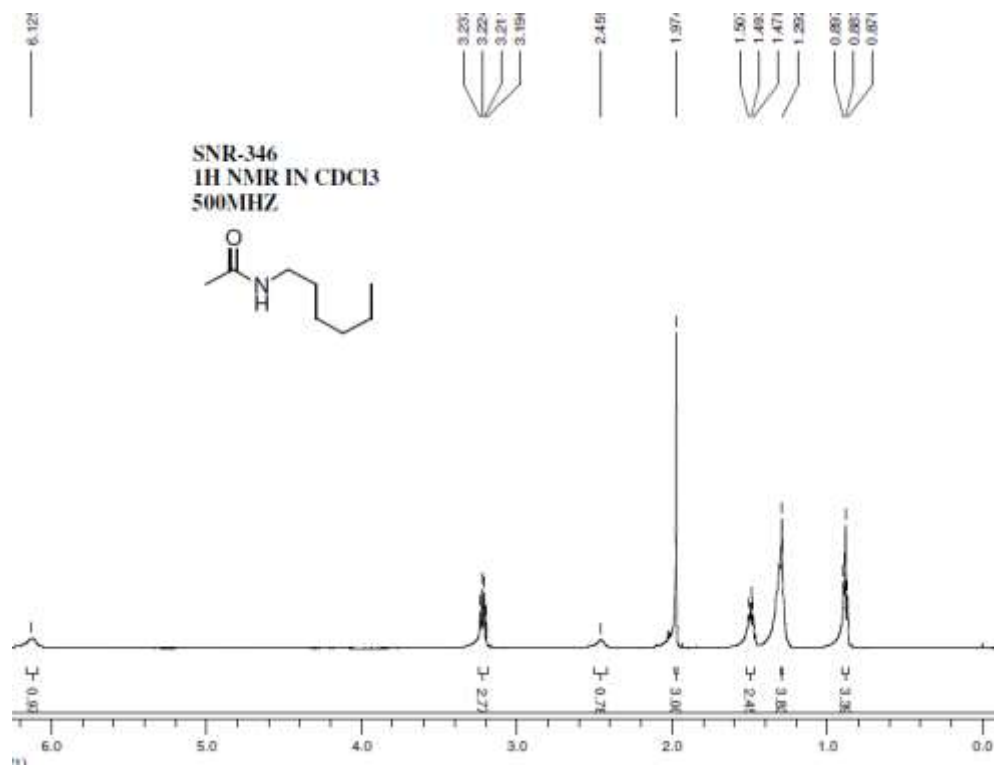
13C NMR of **3g**



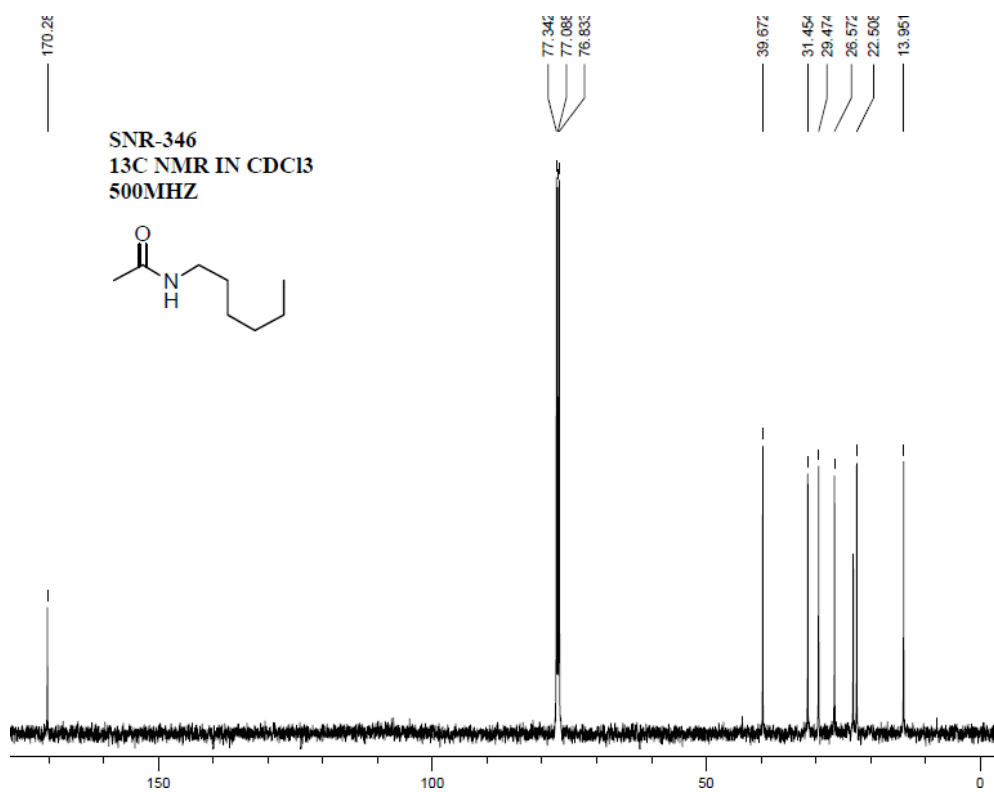
1H NMR of **3h**



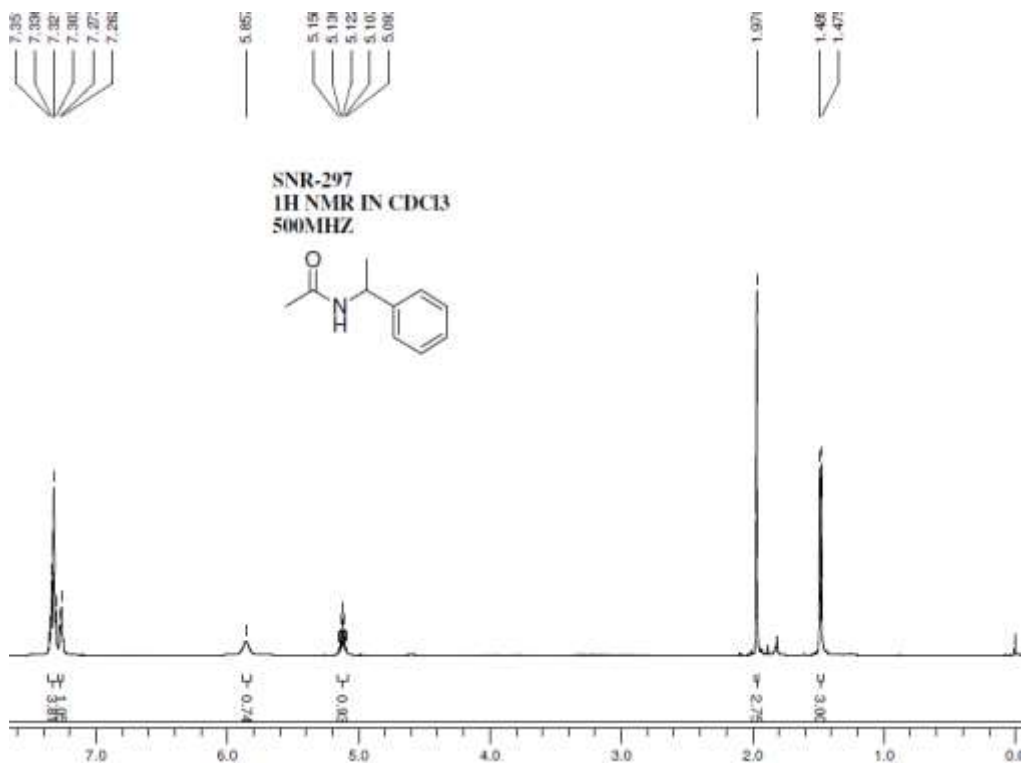
13C NMR of **3h**



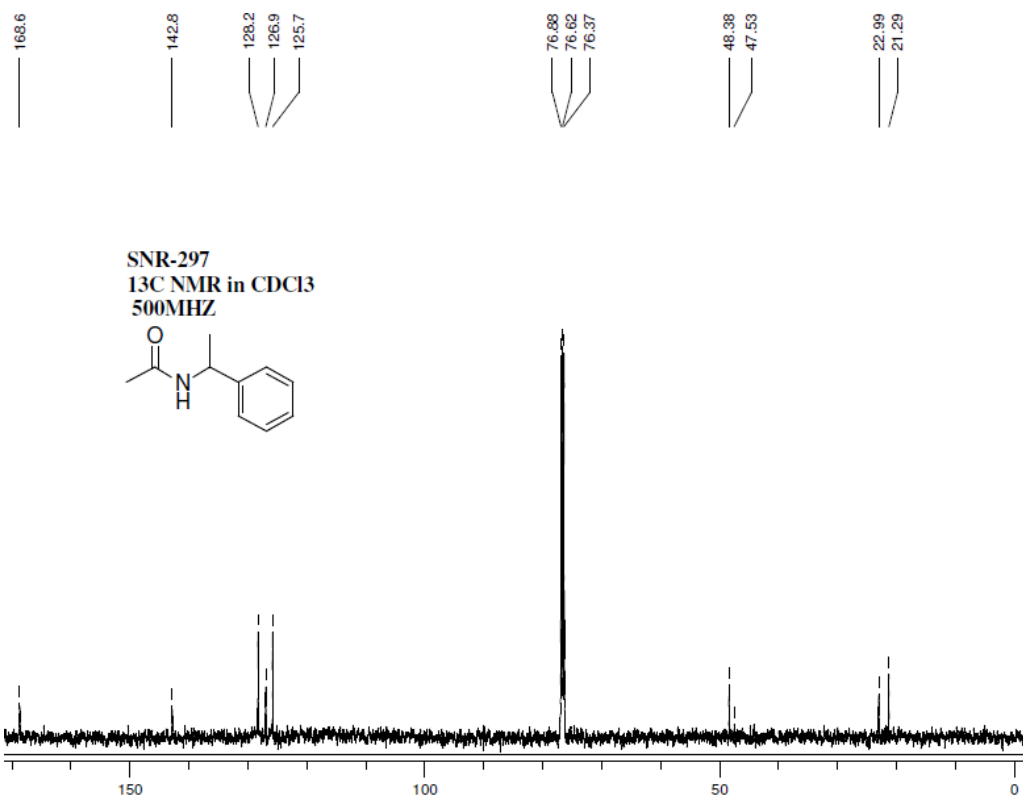
1H NMR of **3i**



13C NMR of **3i**

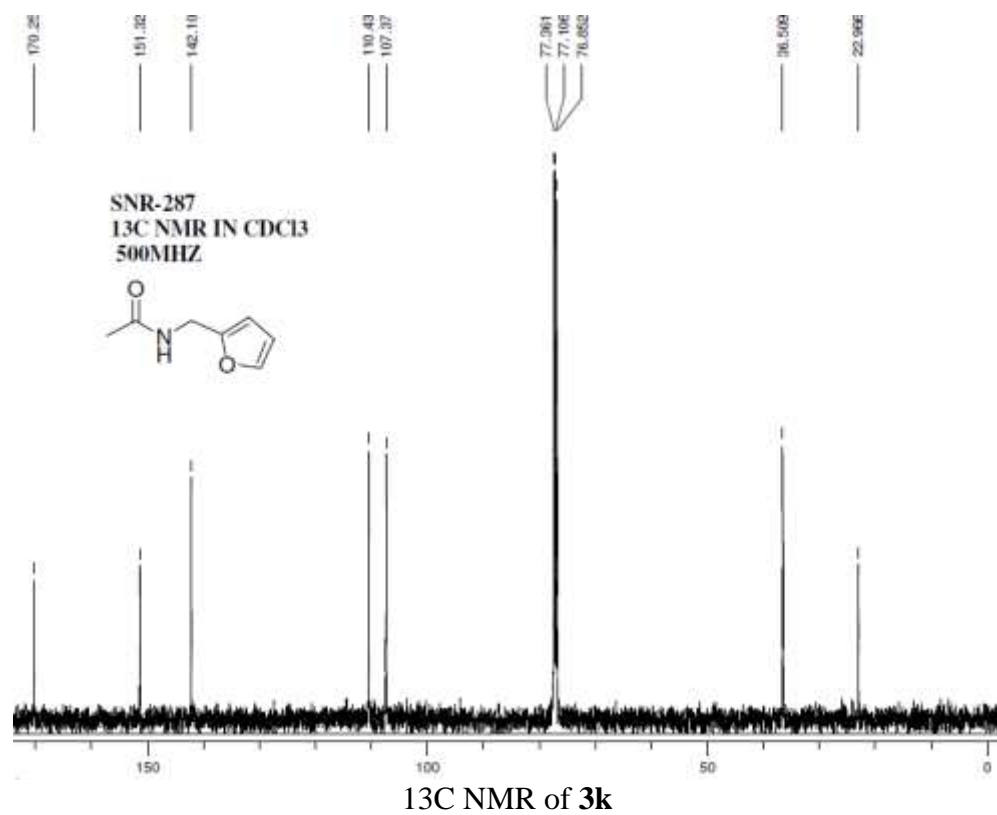
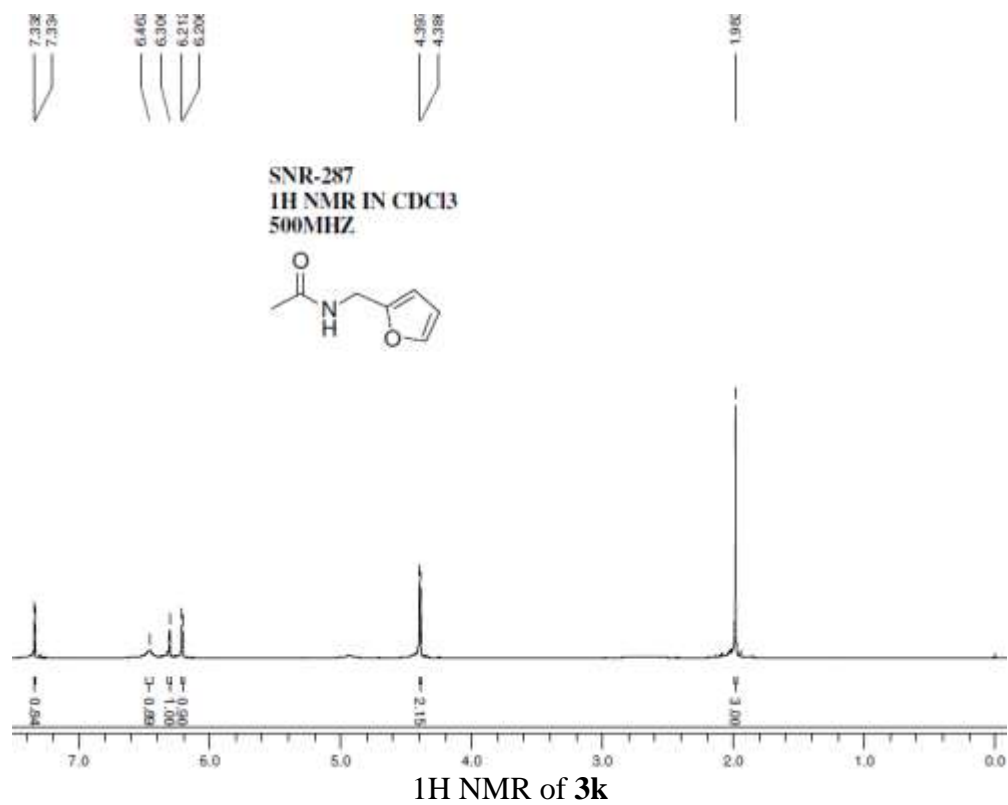


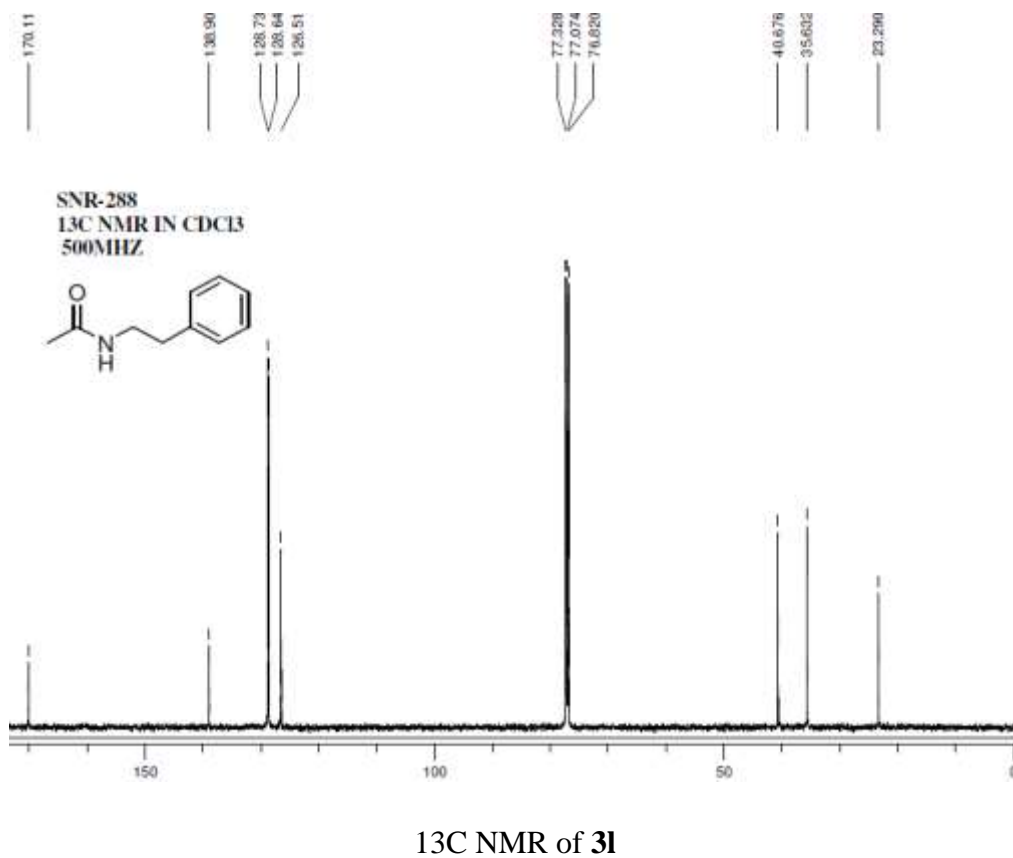
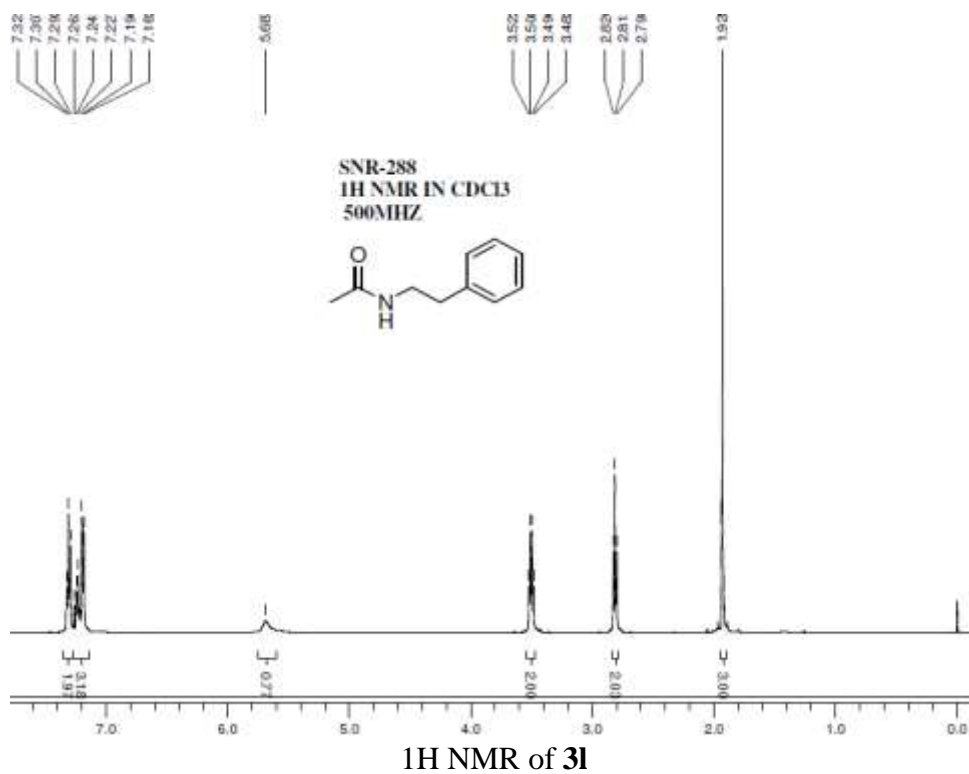
1H NMR of **3j**

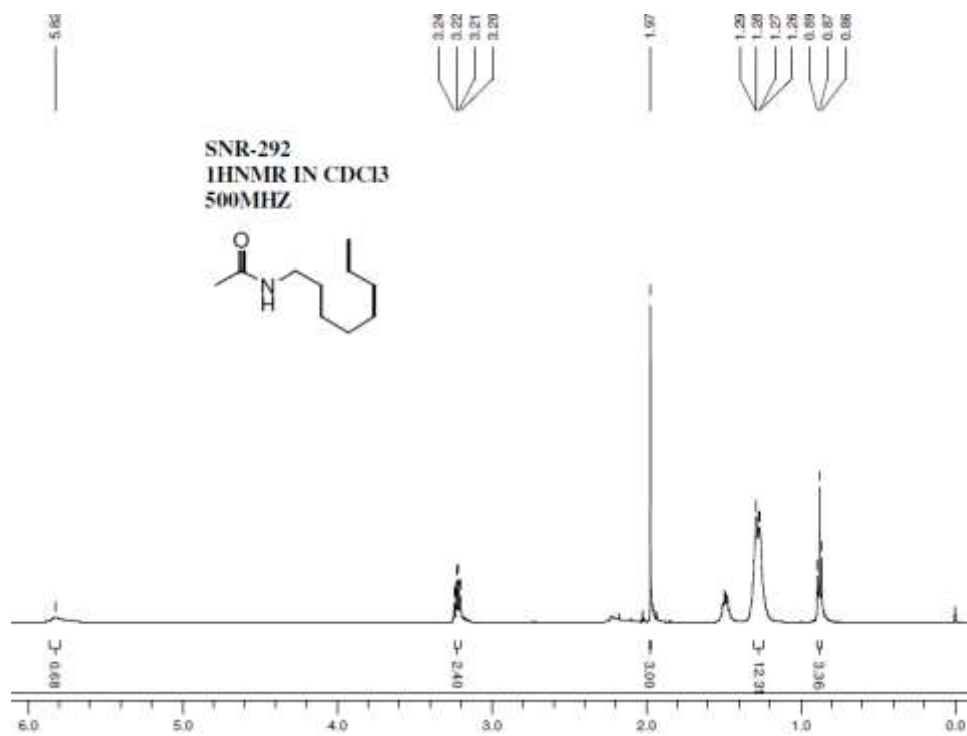


13C NMR of **3j**

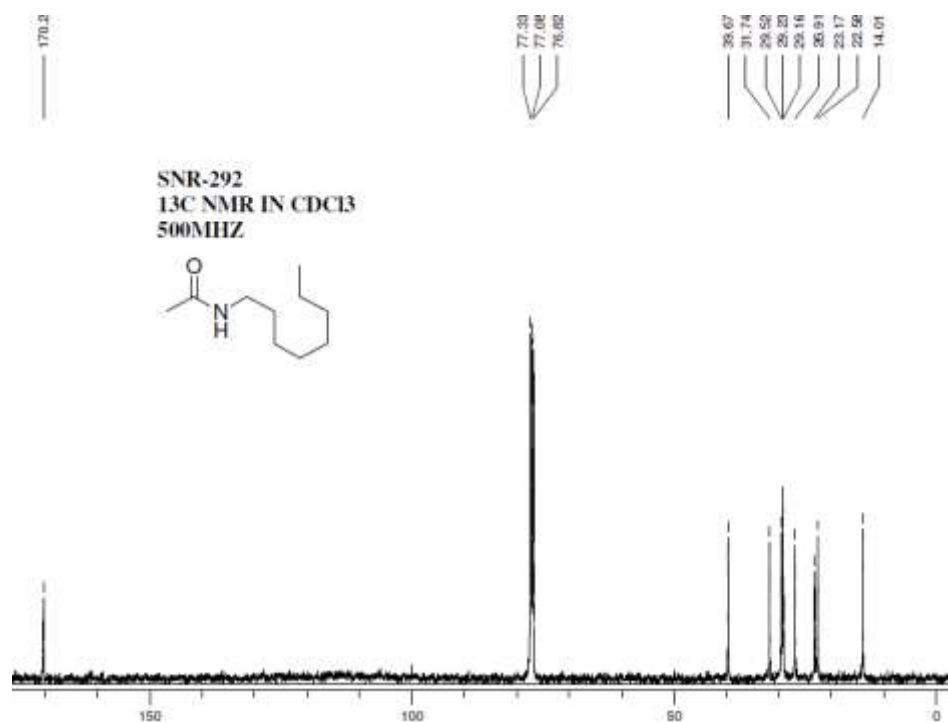




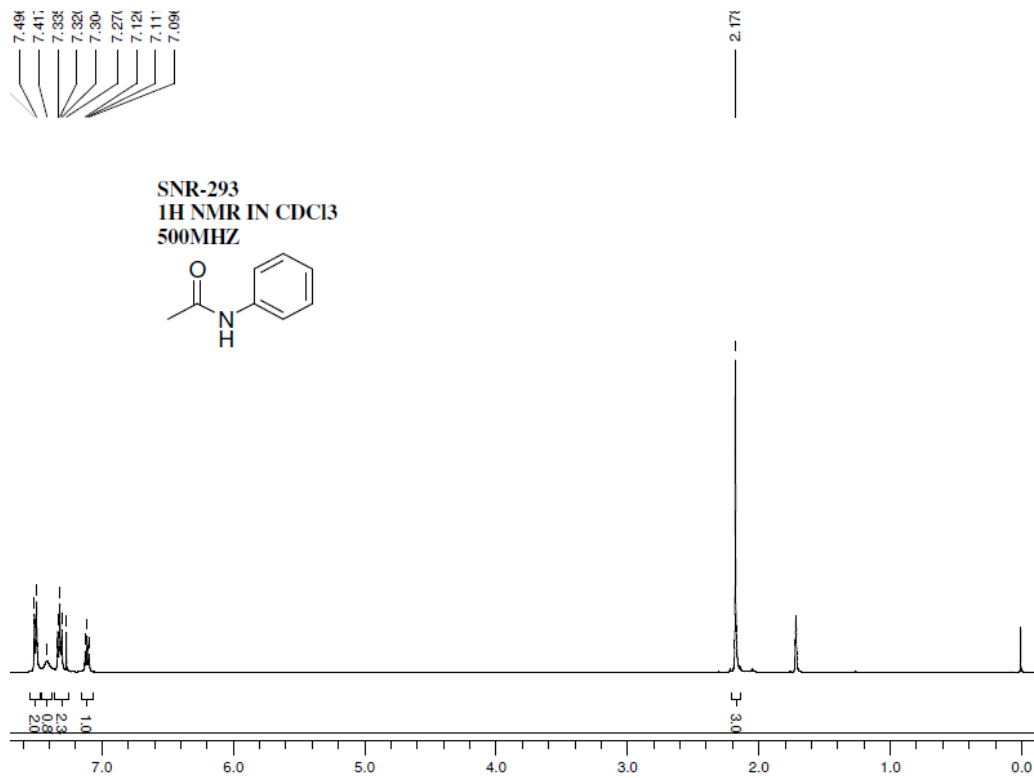




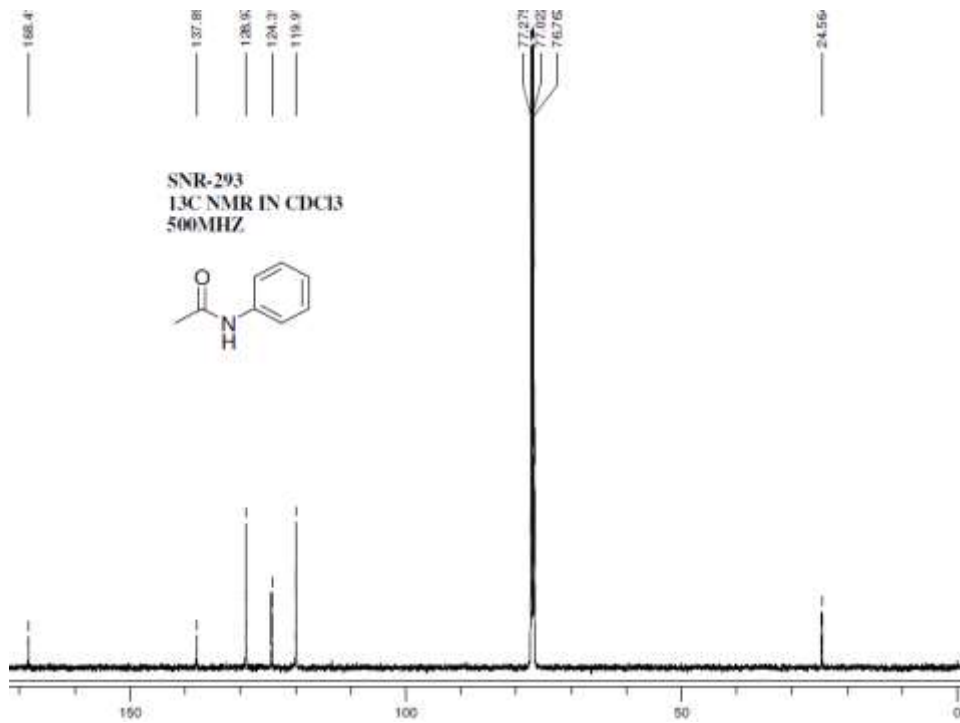
<sup>1</sup>H NMR of **3m**



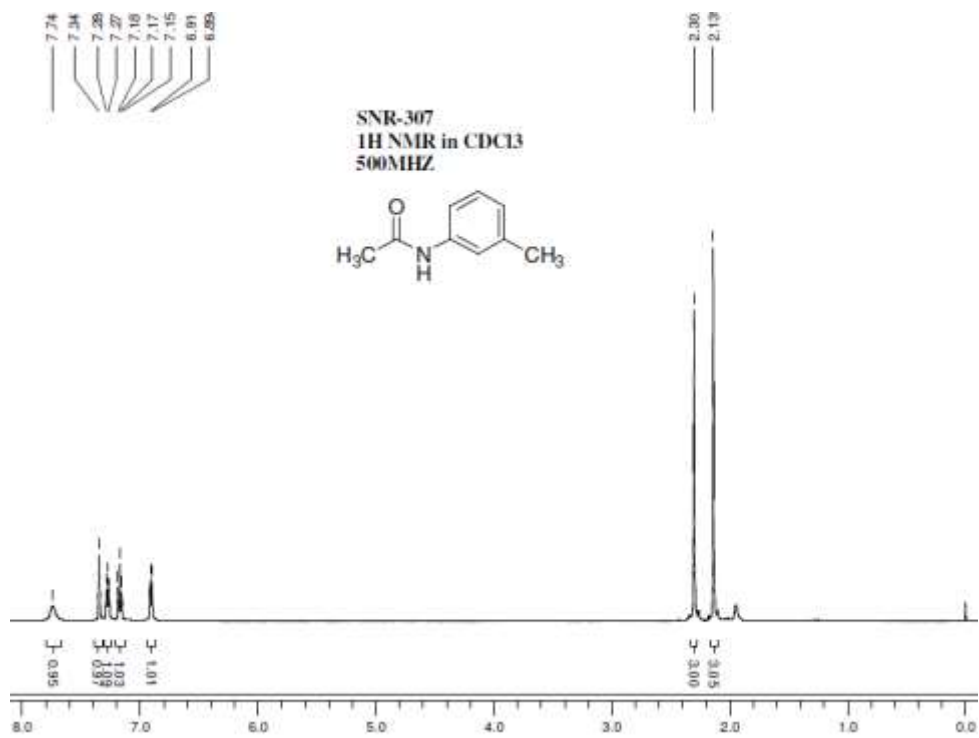
<sup>13</sup>C NMR of **3m**



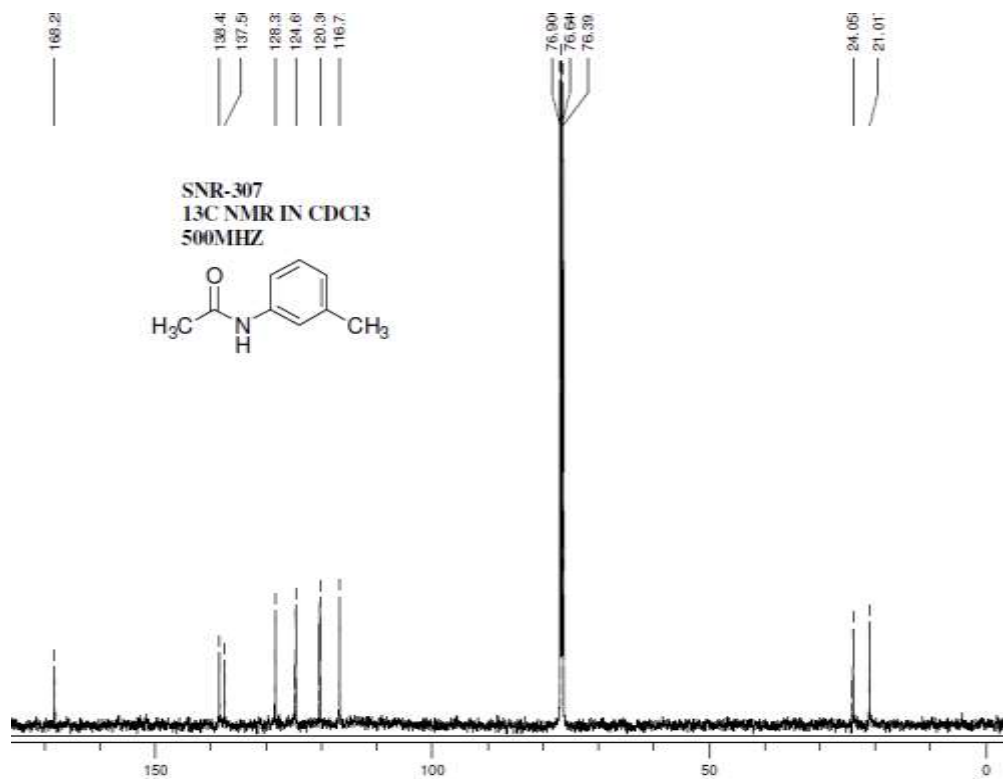
1H NMR of **3n**



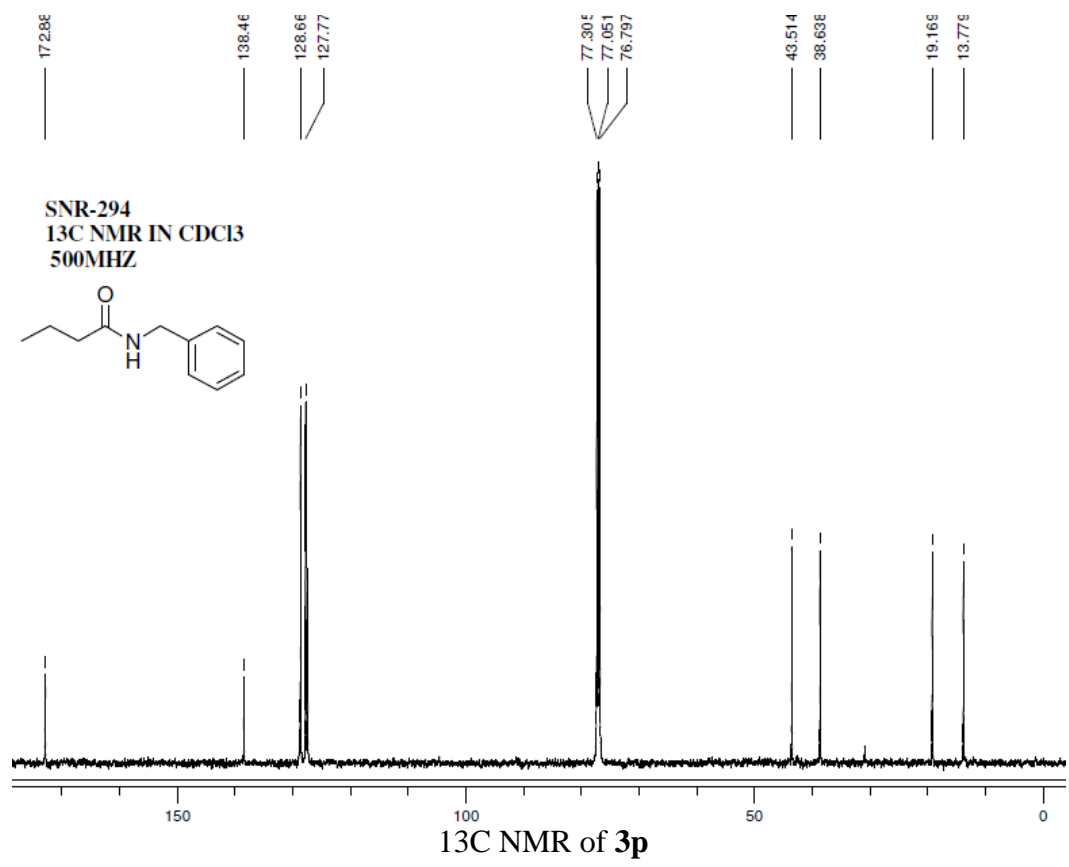
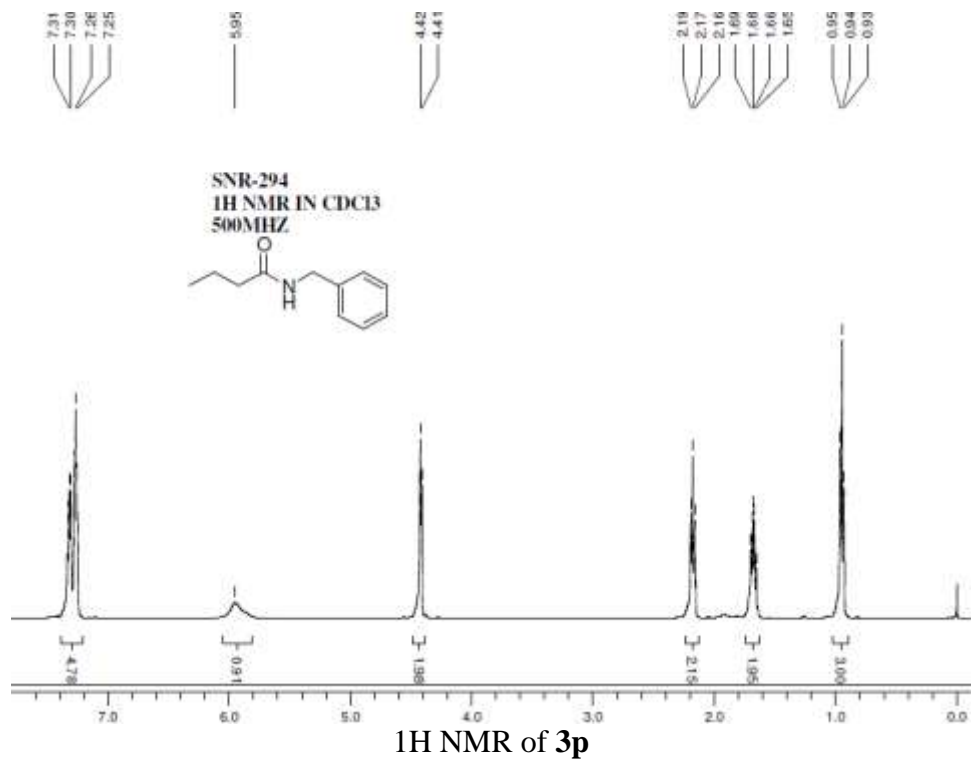
13C NMR of **3n**

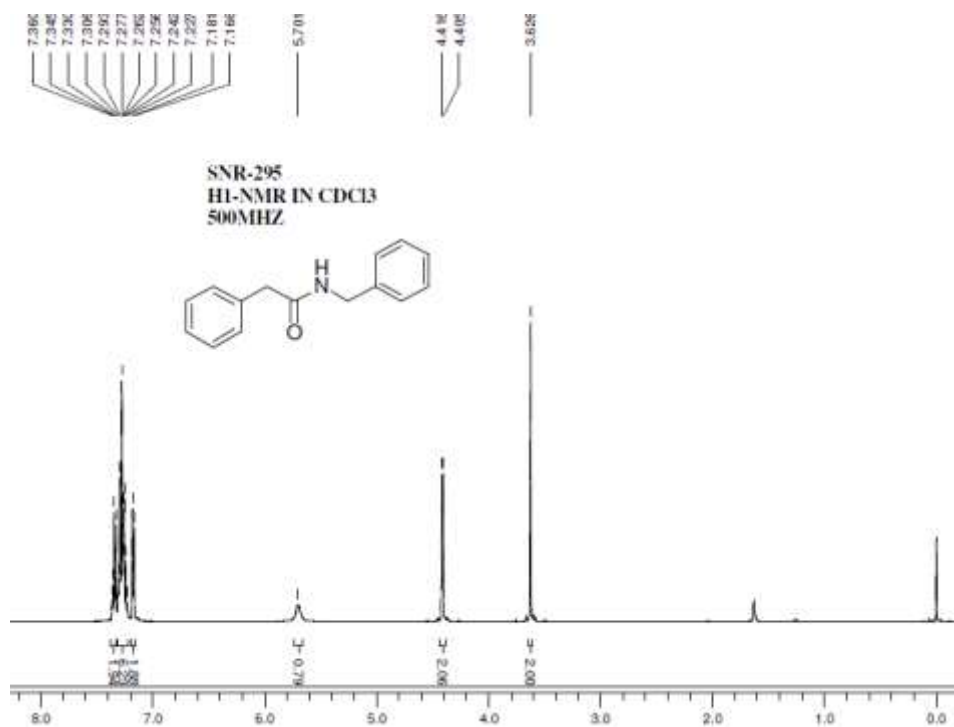


1H NMR of **3o**

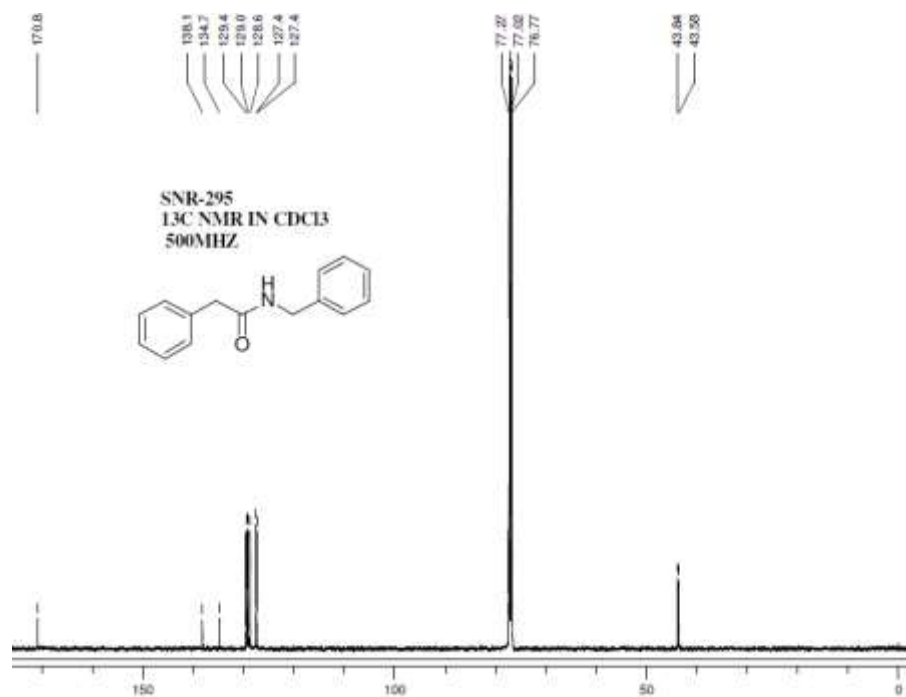


13C NMR of **3o**

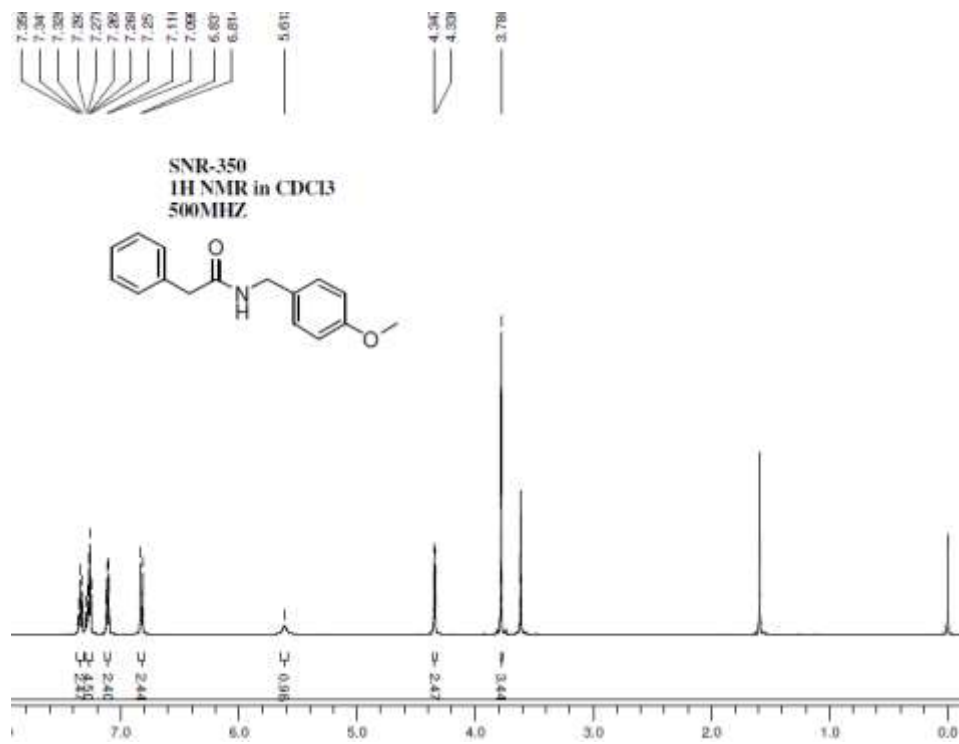




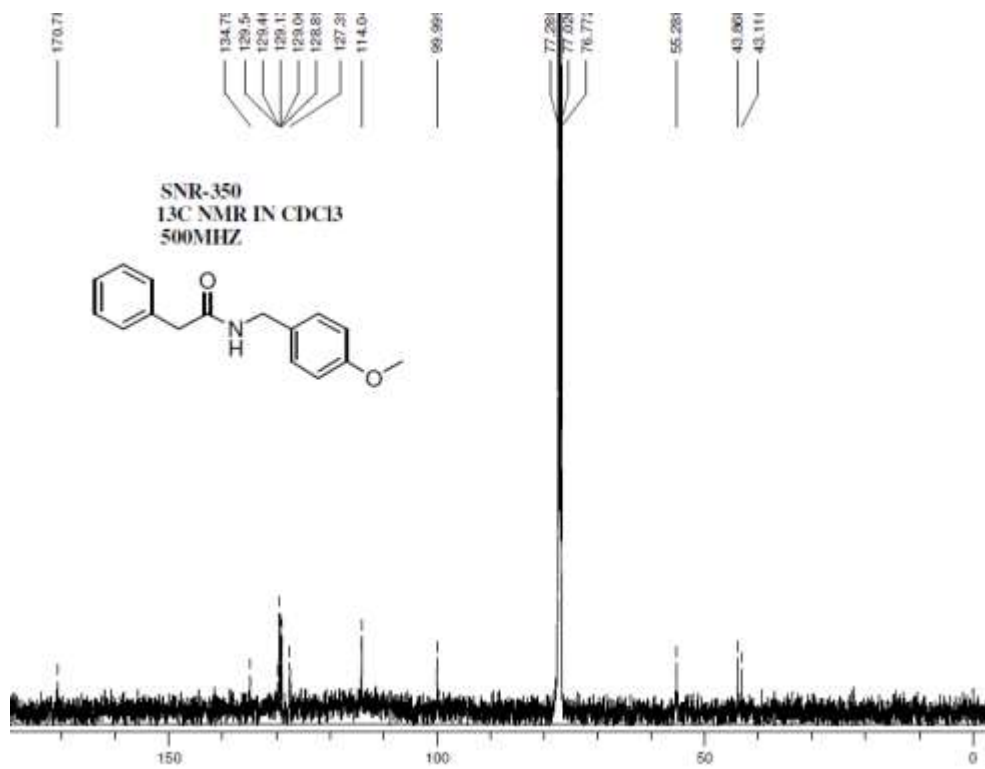
$^1\text{H}$  NMR of **3q**



$^{13}\text{C}$  NMR of **3q**

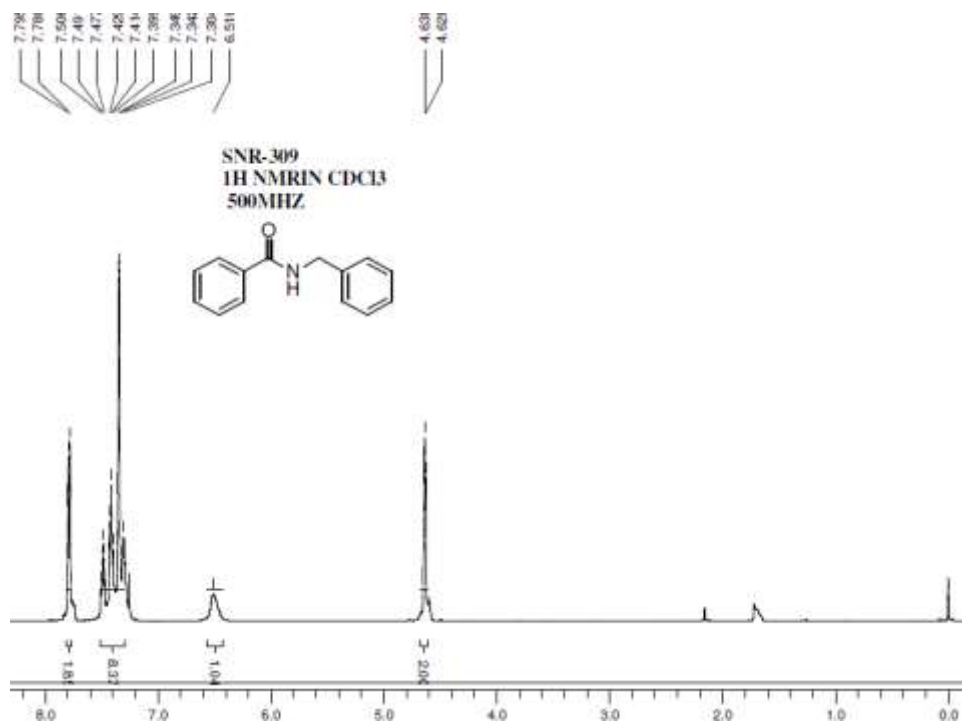


1H NMR of **3r**

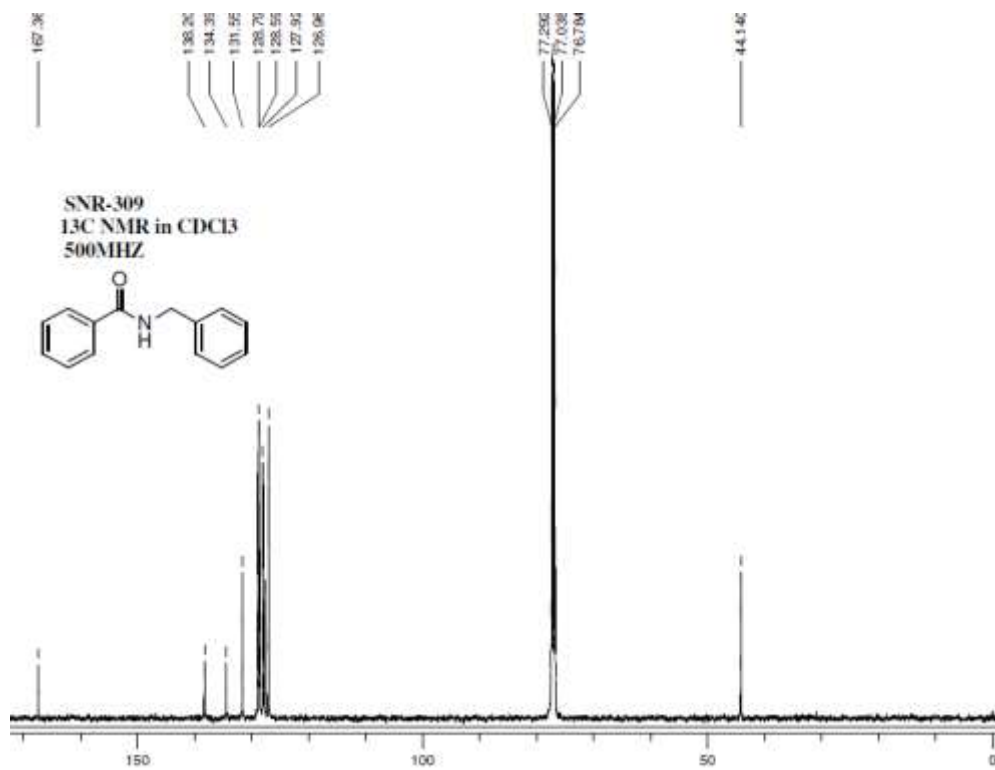


13C NMR of **3r**



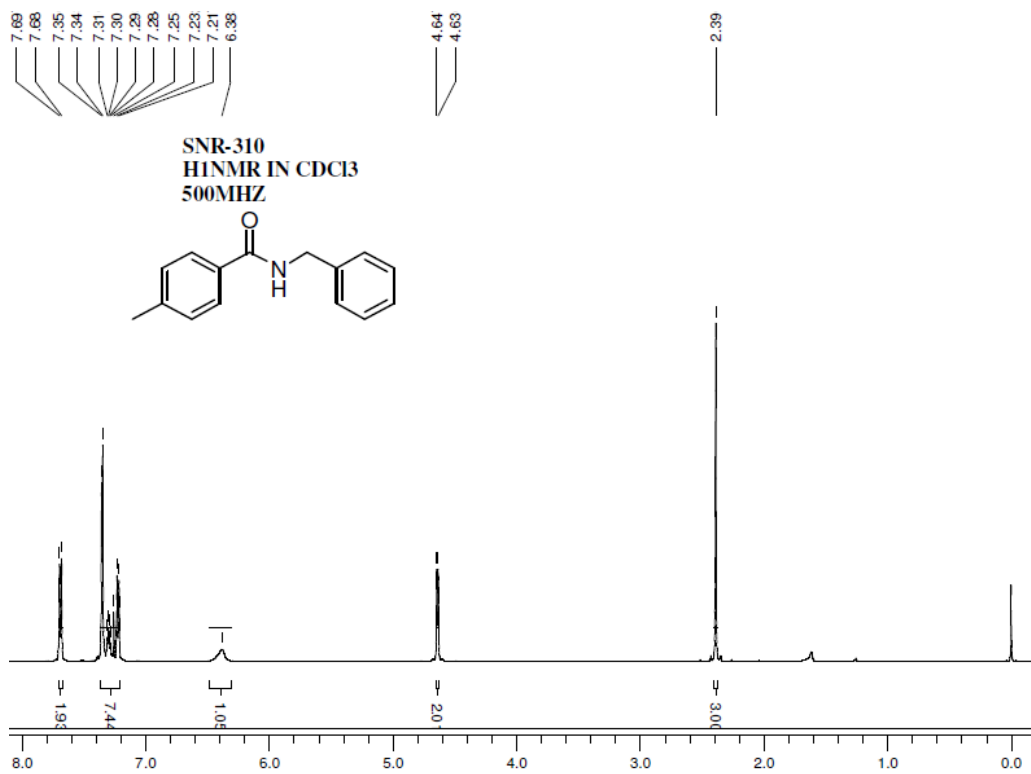


1H NMR of **4a**

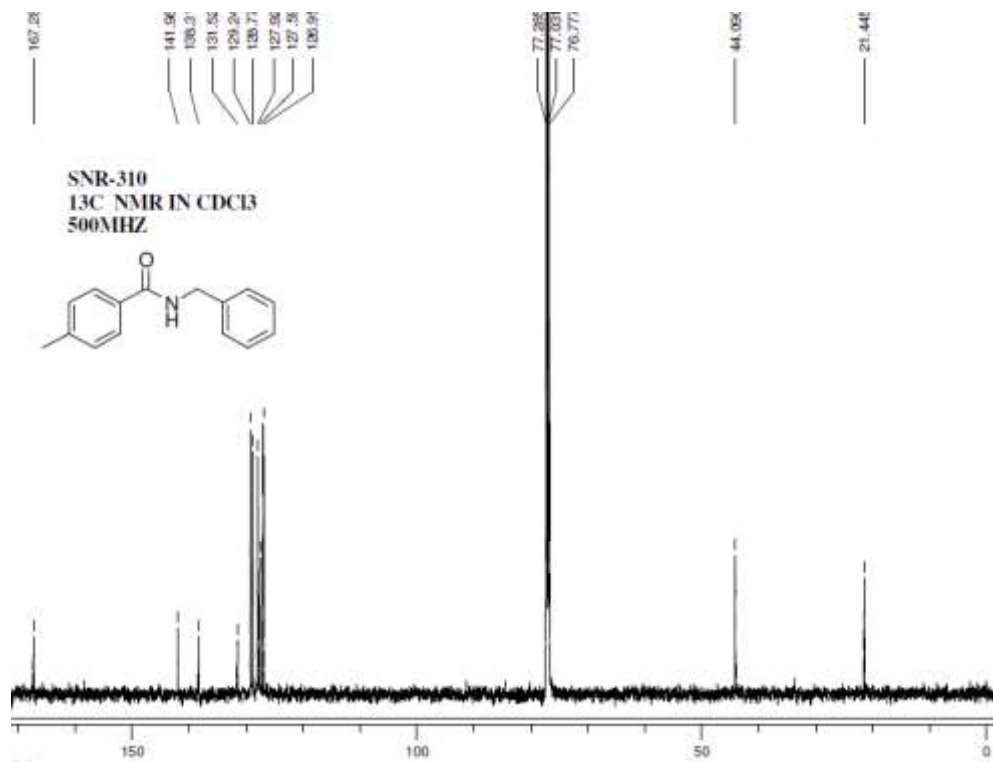


13C NMR of **4a**

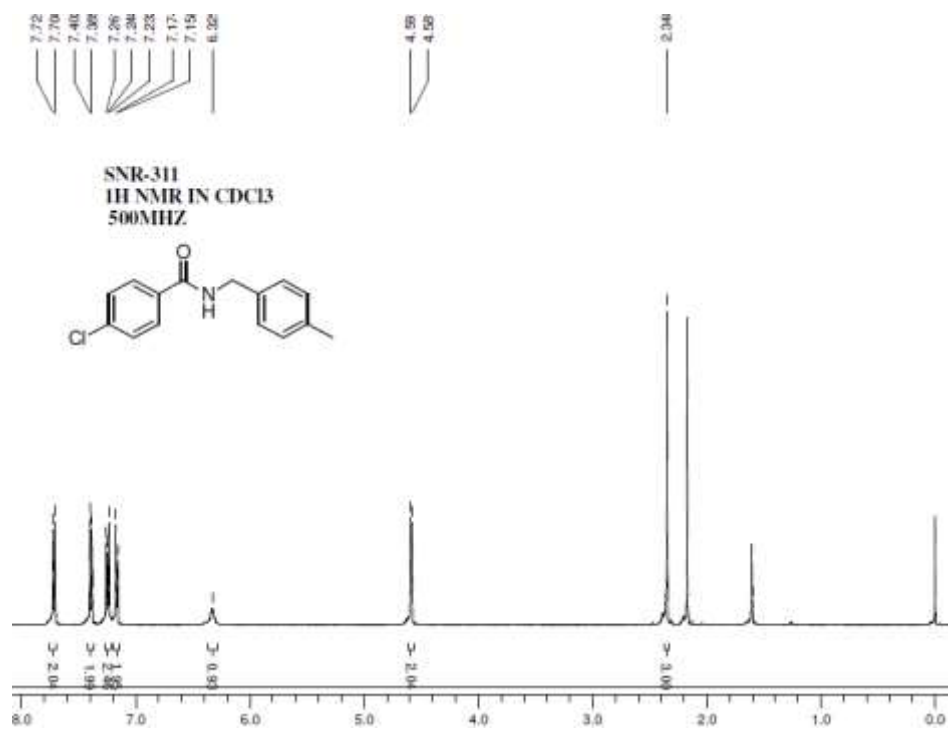




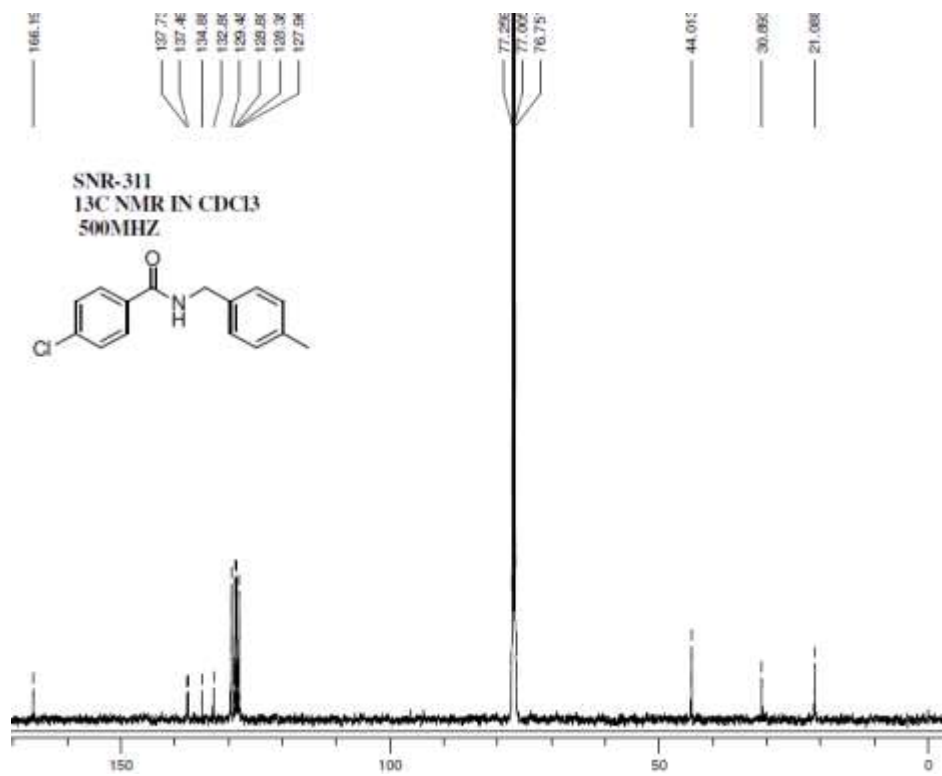
1H NMR of **4c**



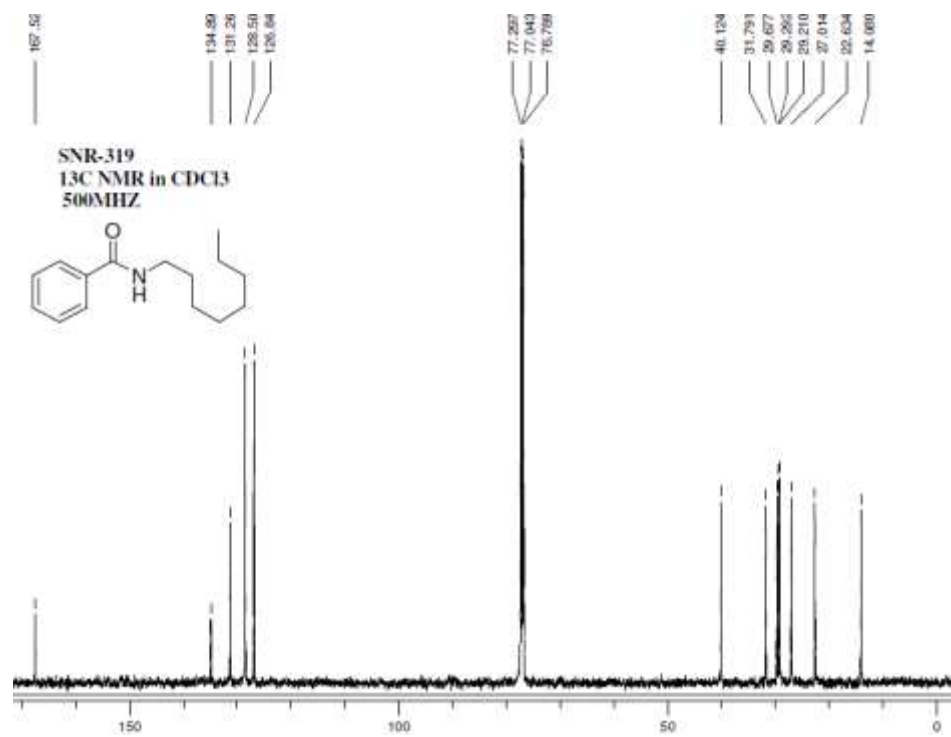
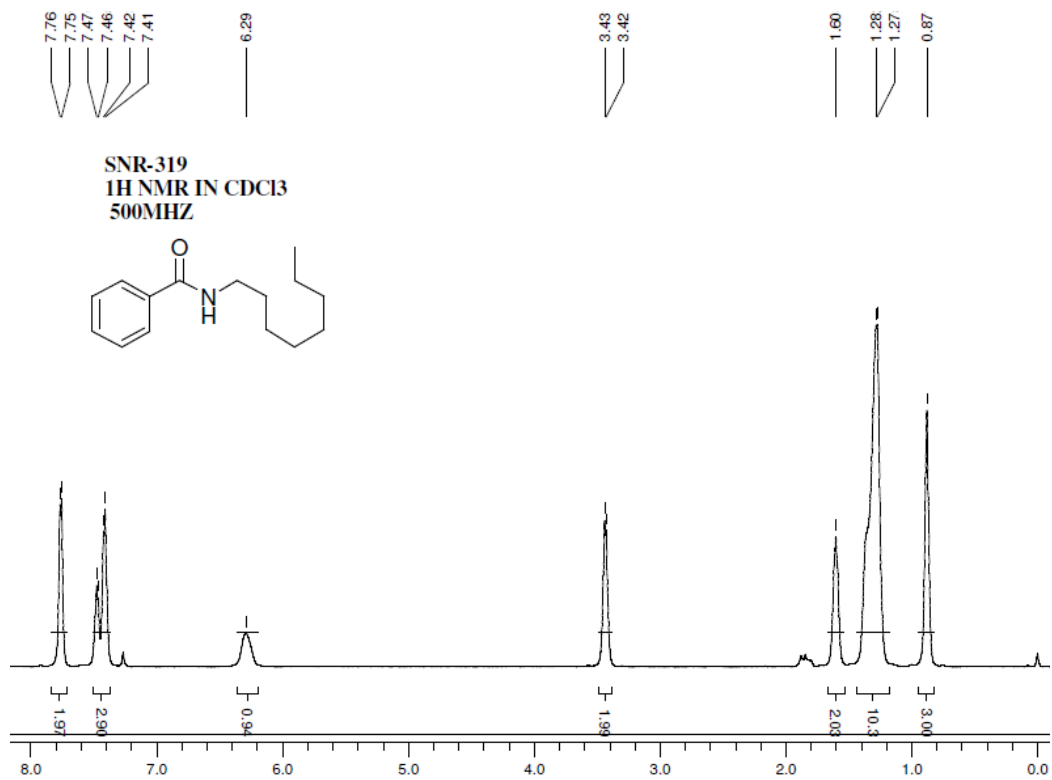
13C NMR of **4c**

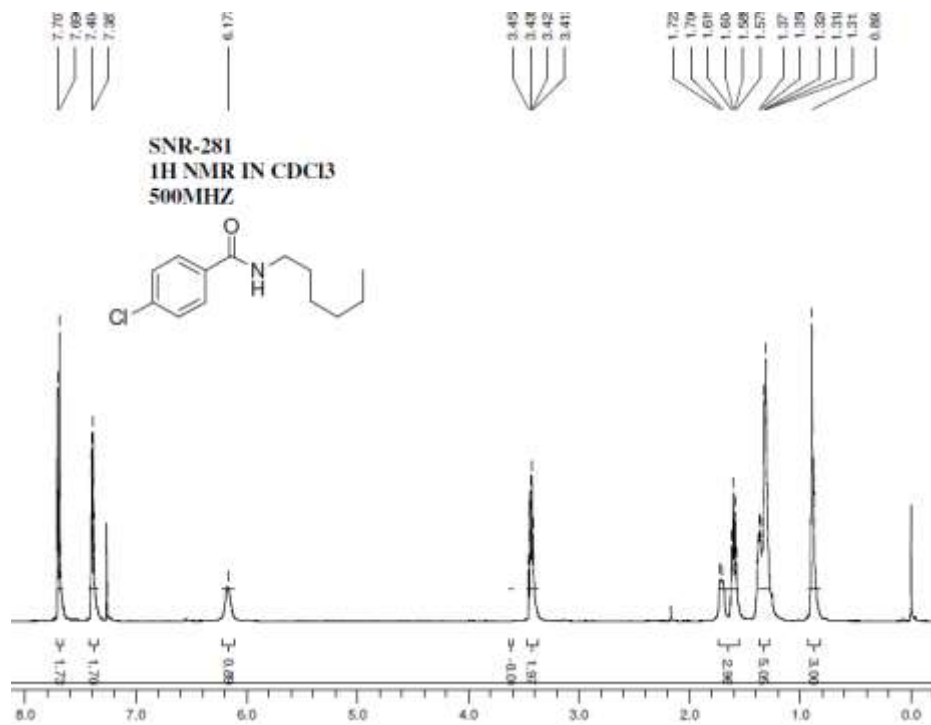


**<sup>1</sup>H NMR of 4d**

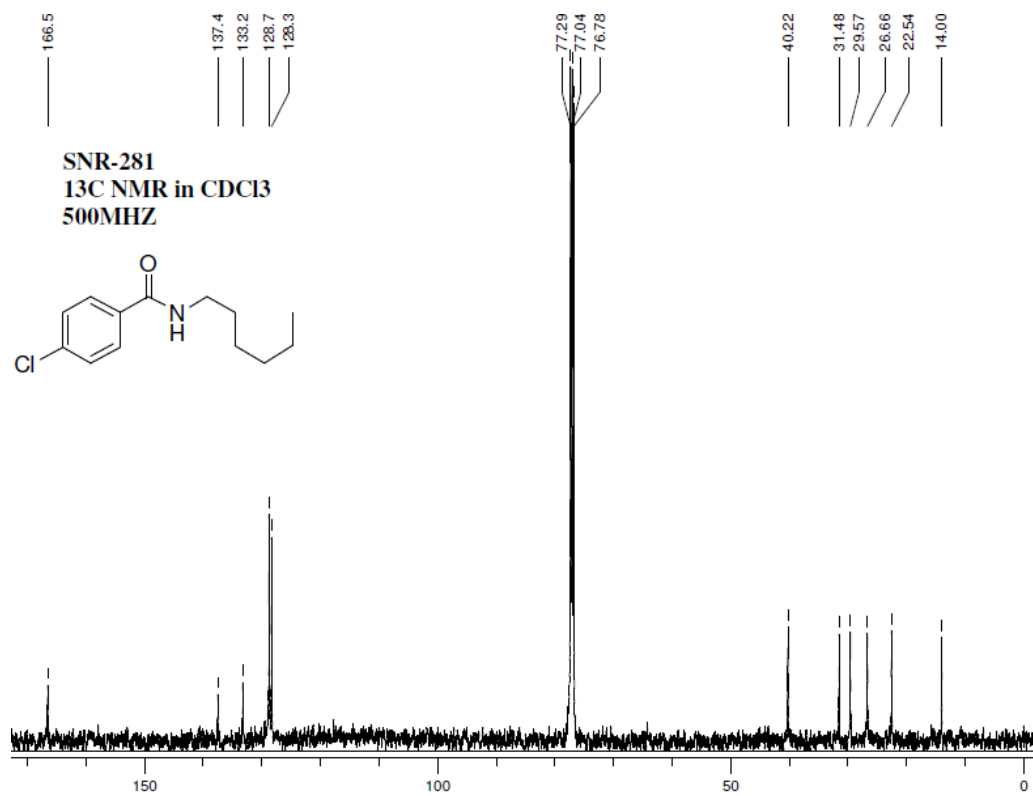


**<sup>13</sup>C NMR of 4d**

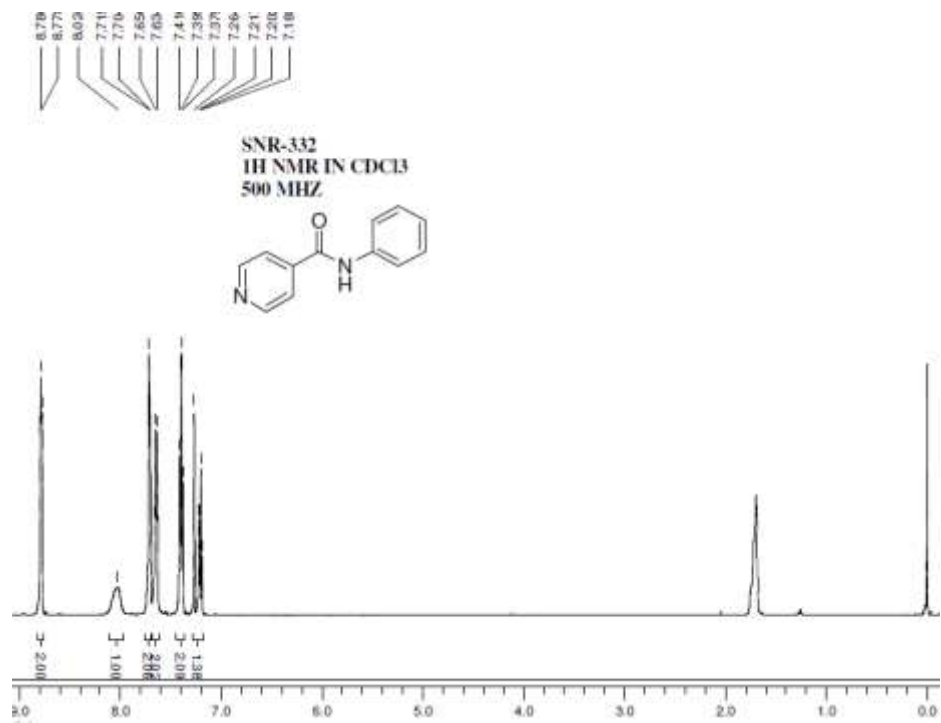




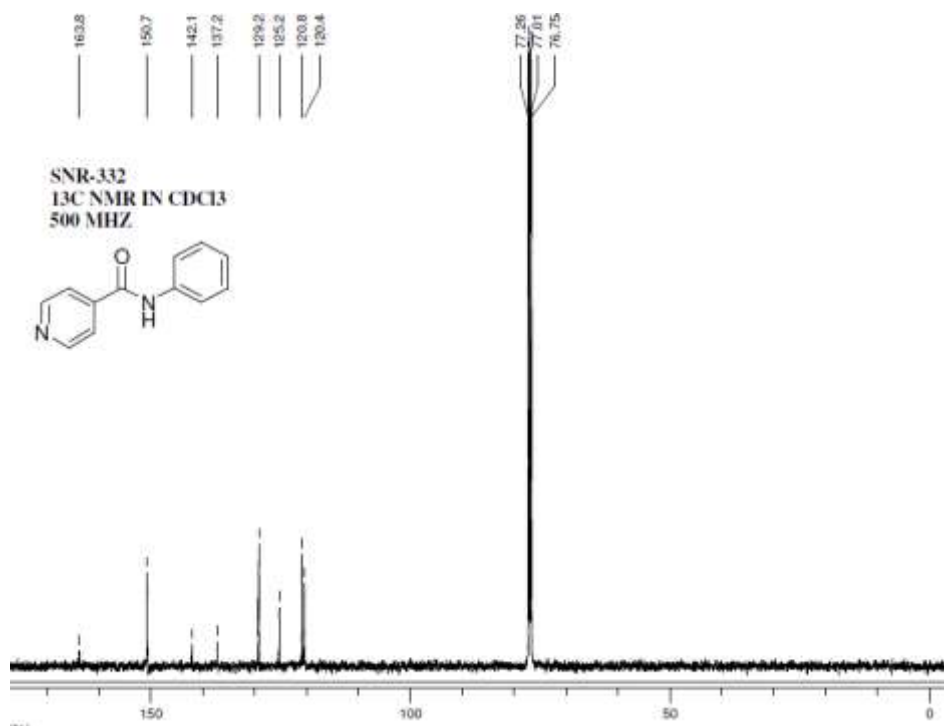
<sup>1</sup>H NMR of **4f**



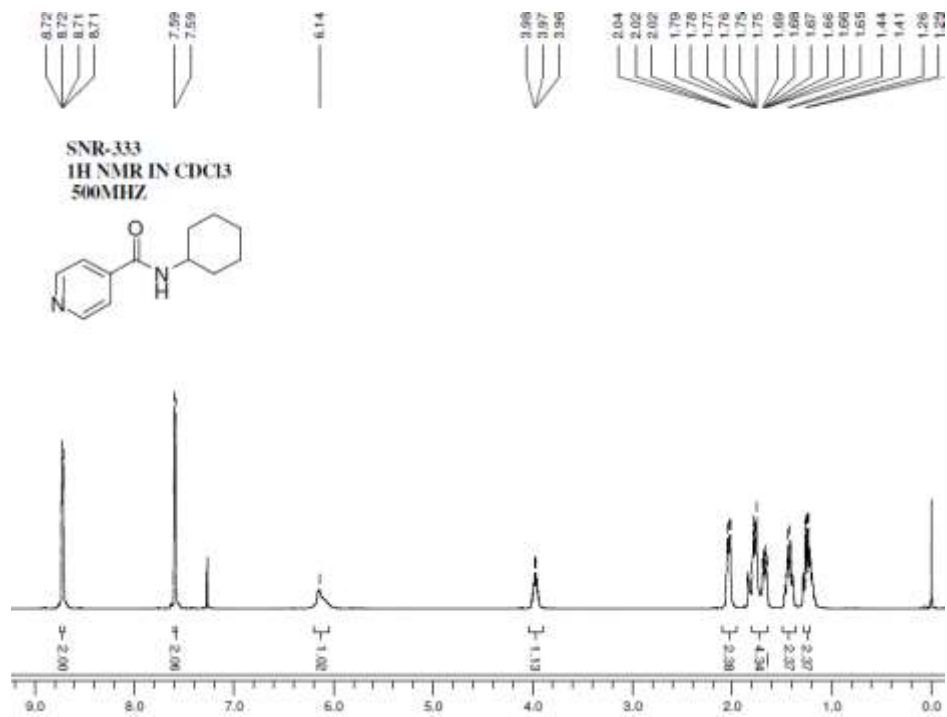
<sup>13</sup>C NMR of **4f**



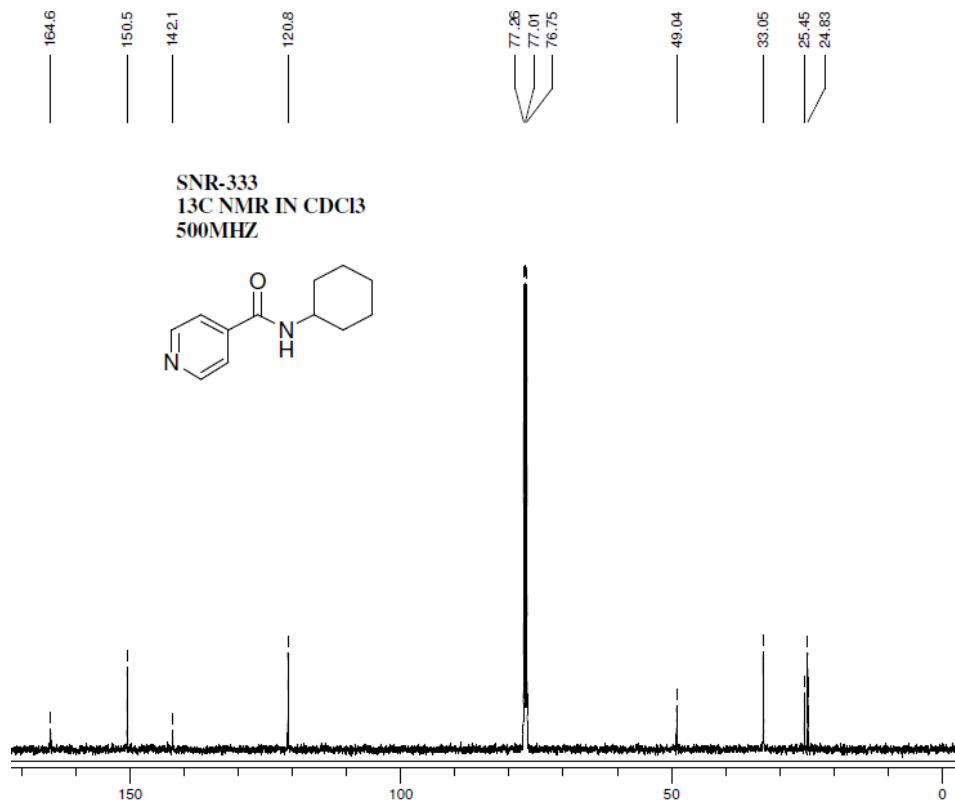
1H NMR of **4g**



13C NMR of **4g**

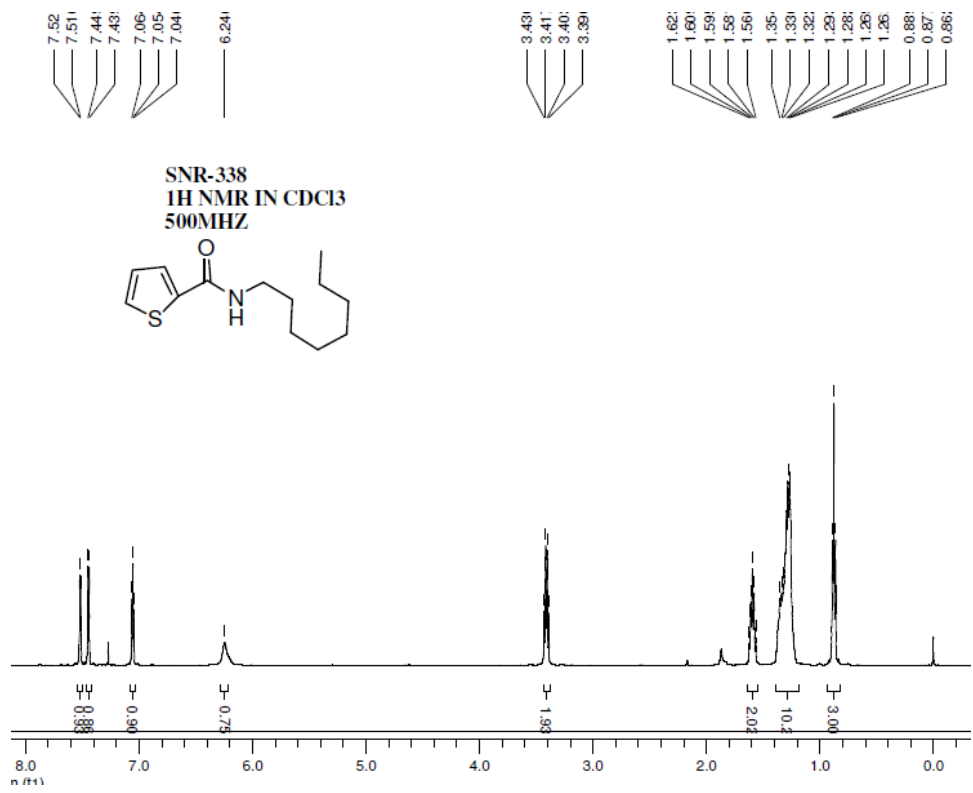


1H NMR of **4h**

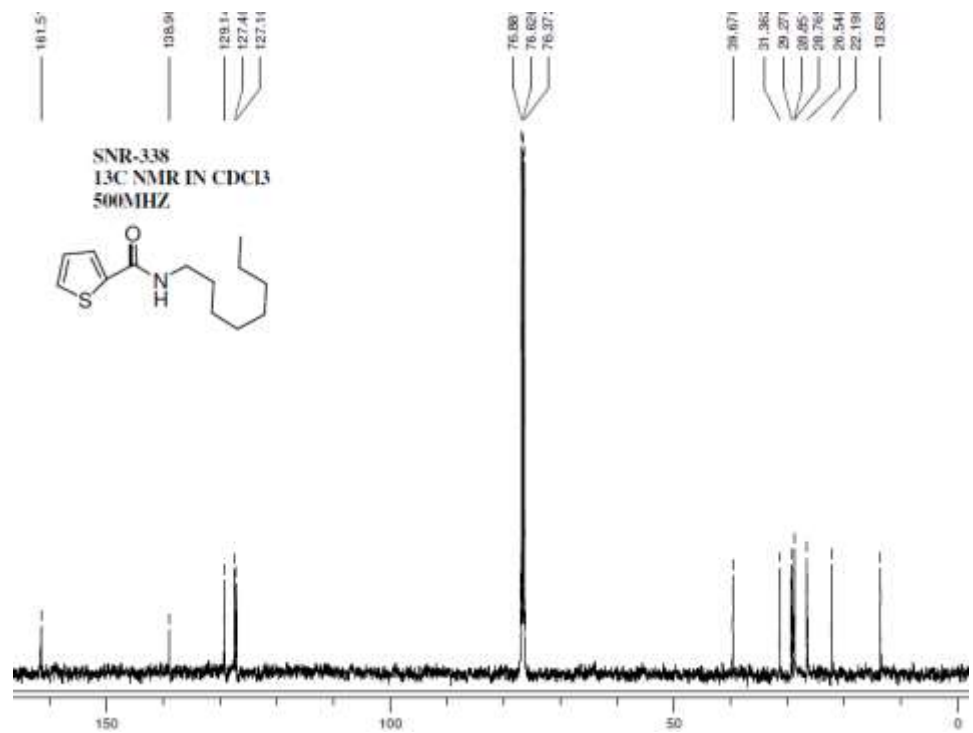


13C NMR of **4h**

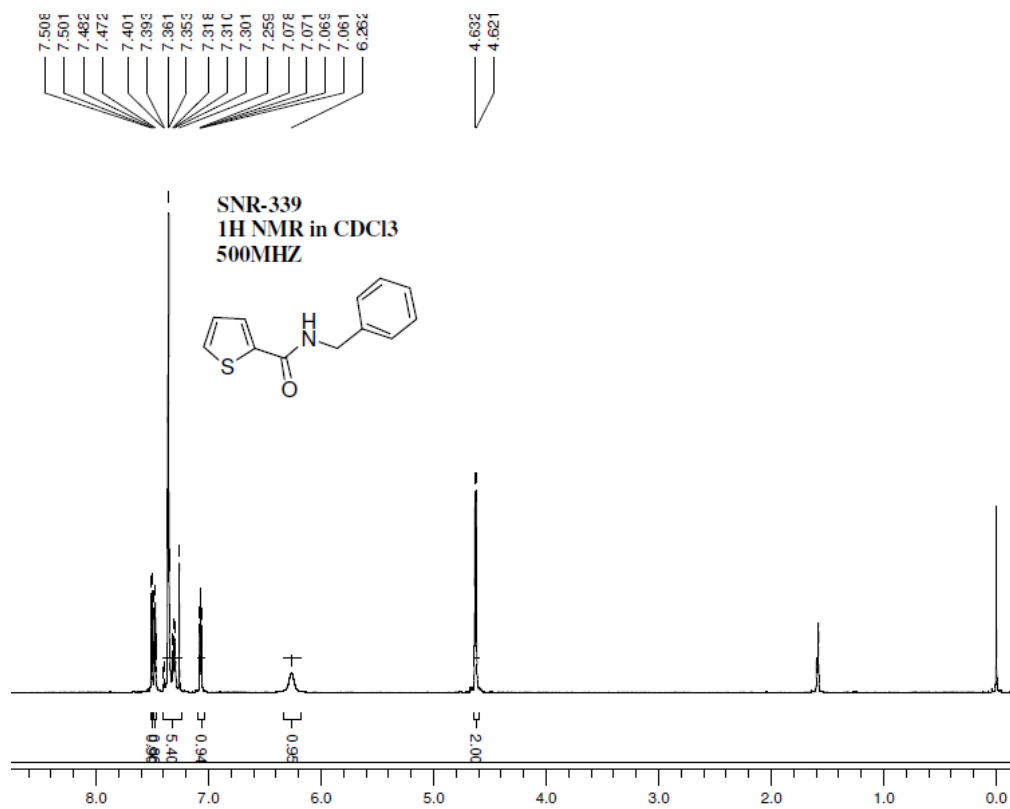




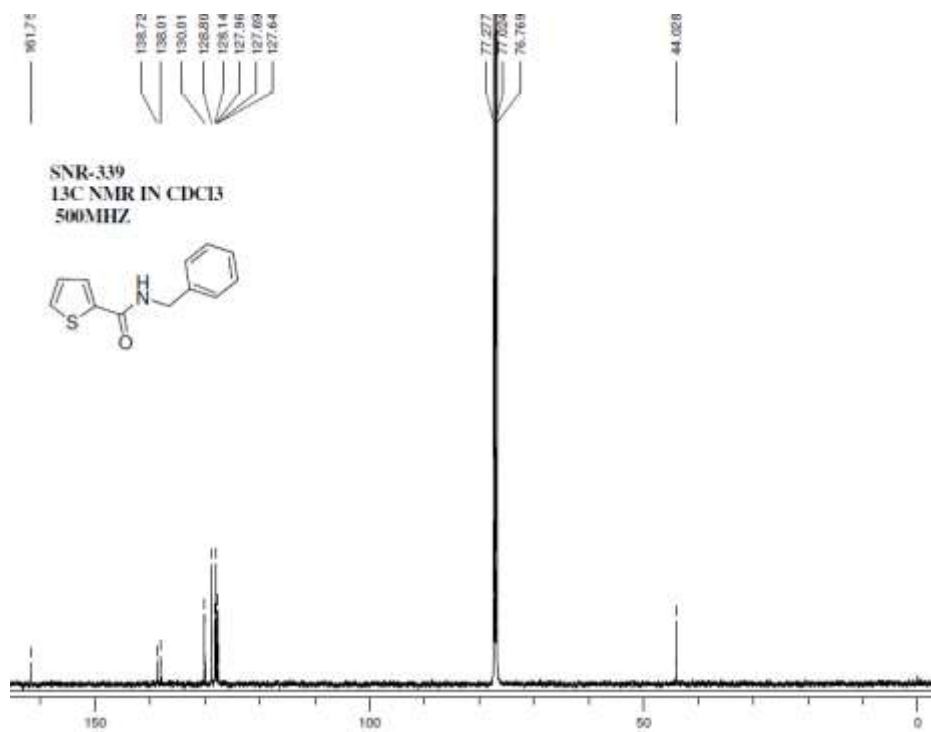
1H NMR of **4i**



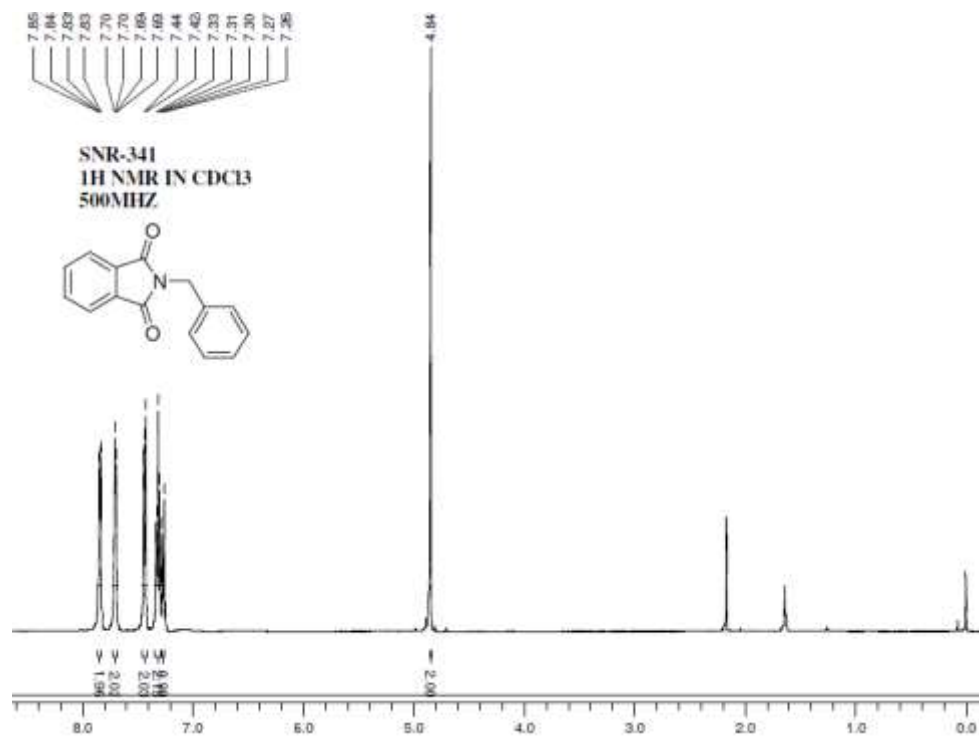
13C NMR of **4i**



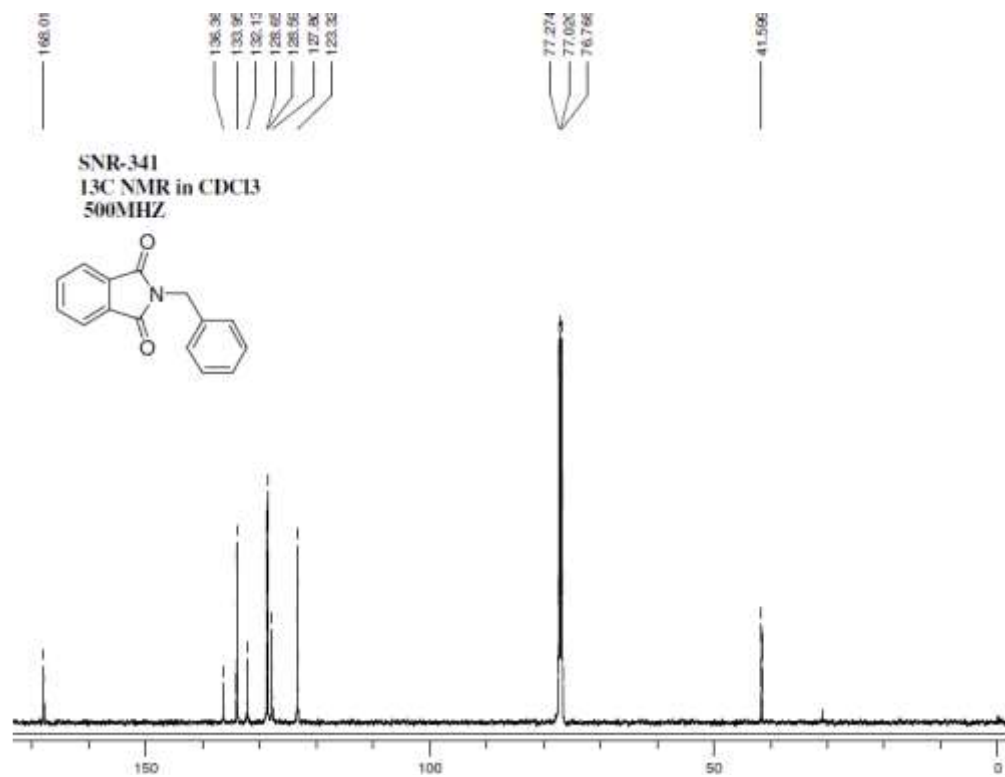
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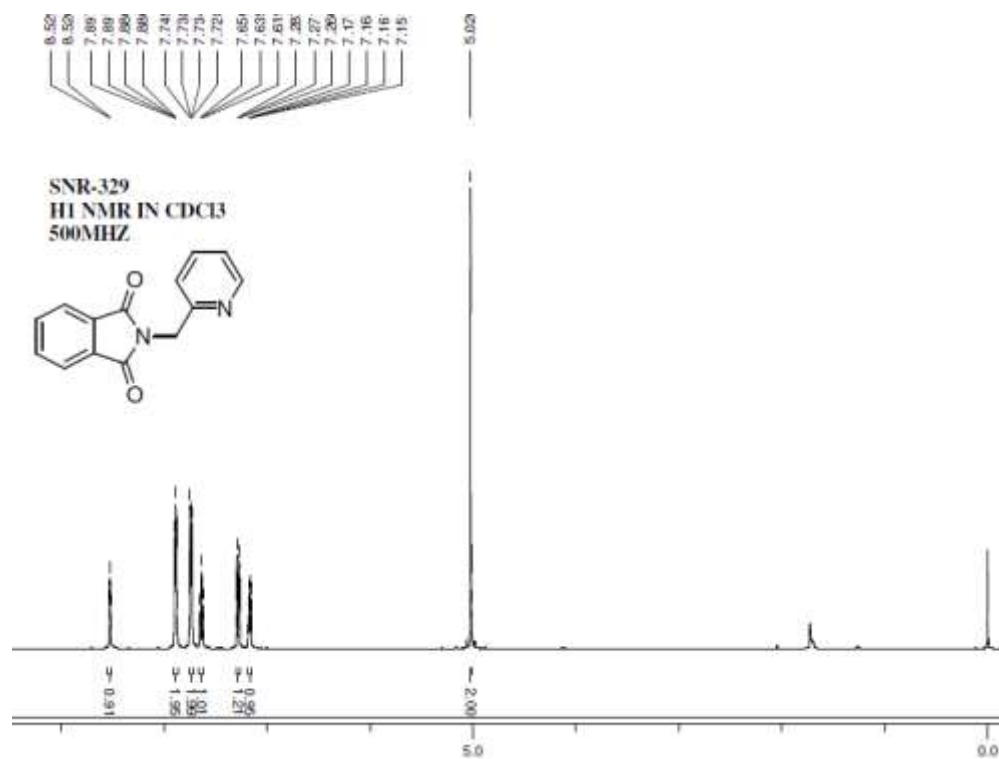
13C NMR of **4j**



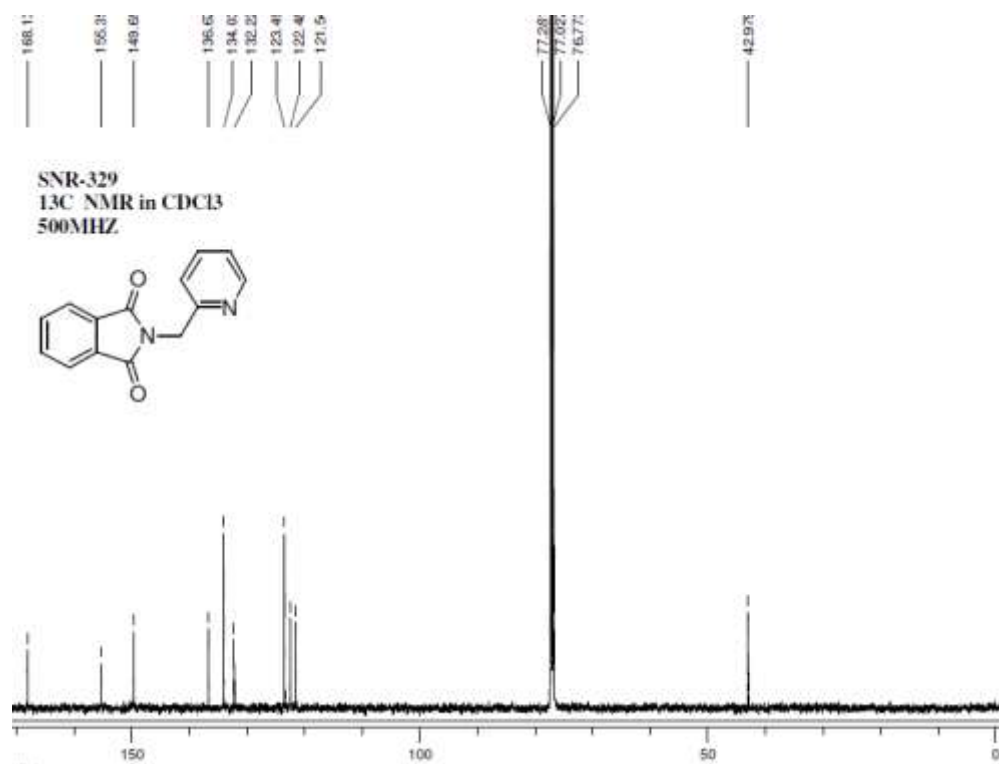
1H NMR of **4k**



13C NMR of **4k**

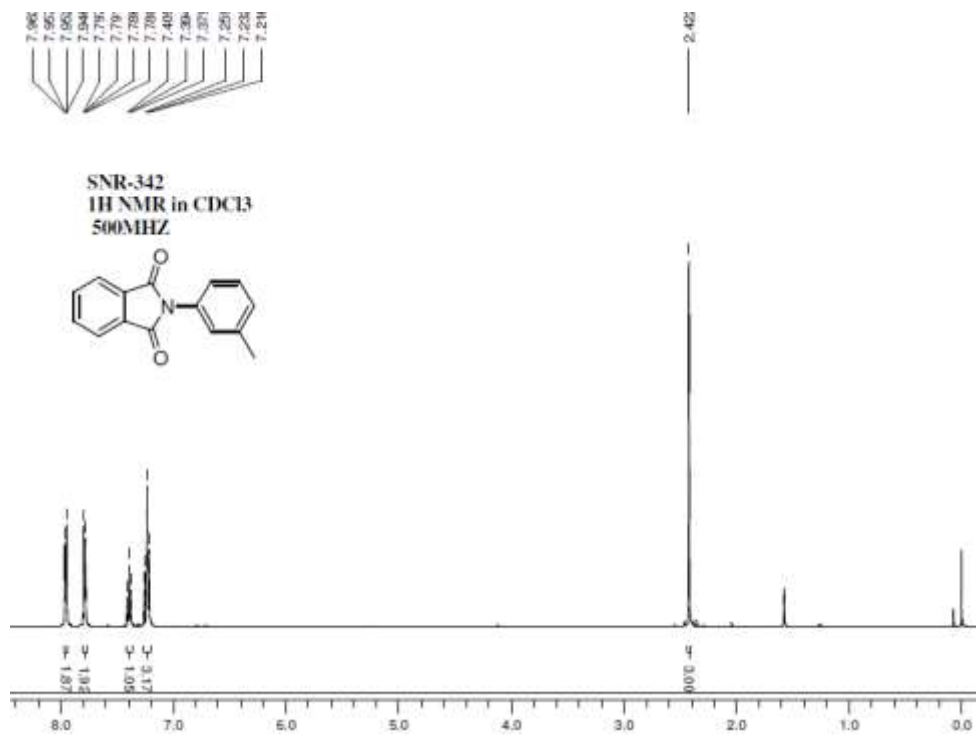


<sup>1</sup>H NMR of **4I**

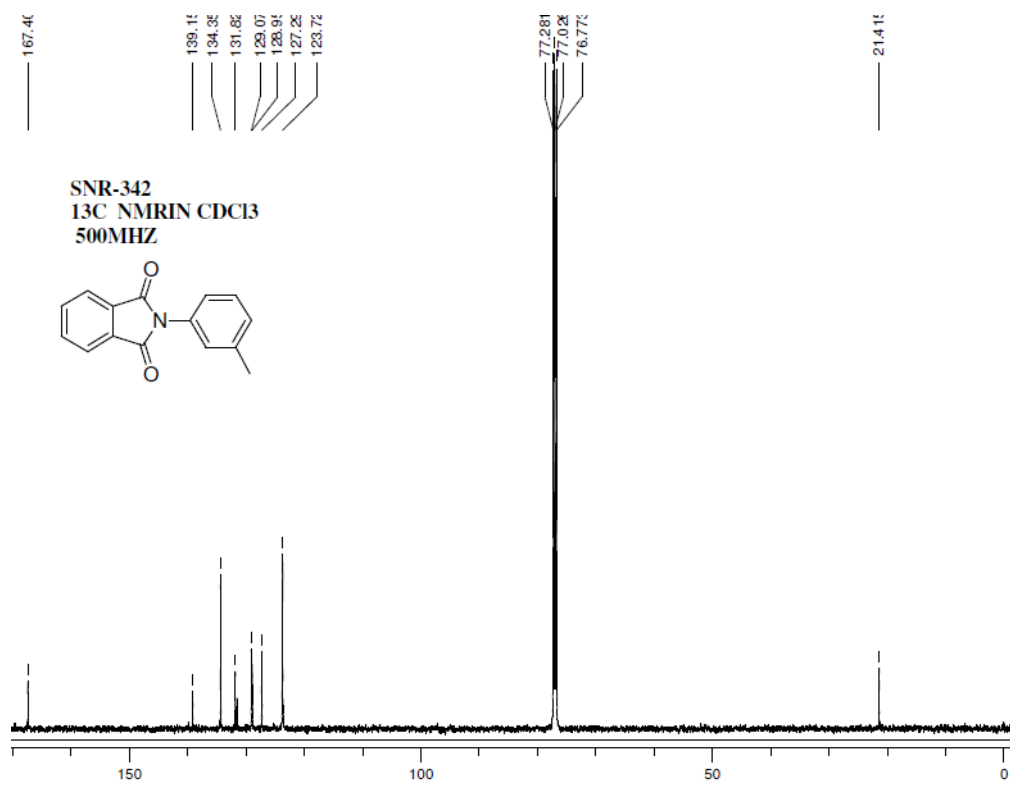


<sup>13</sup>C NMR of **4I**

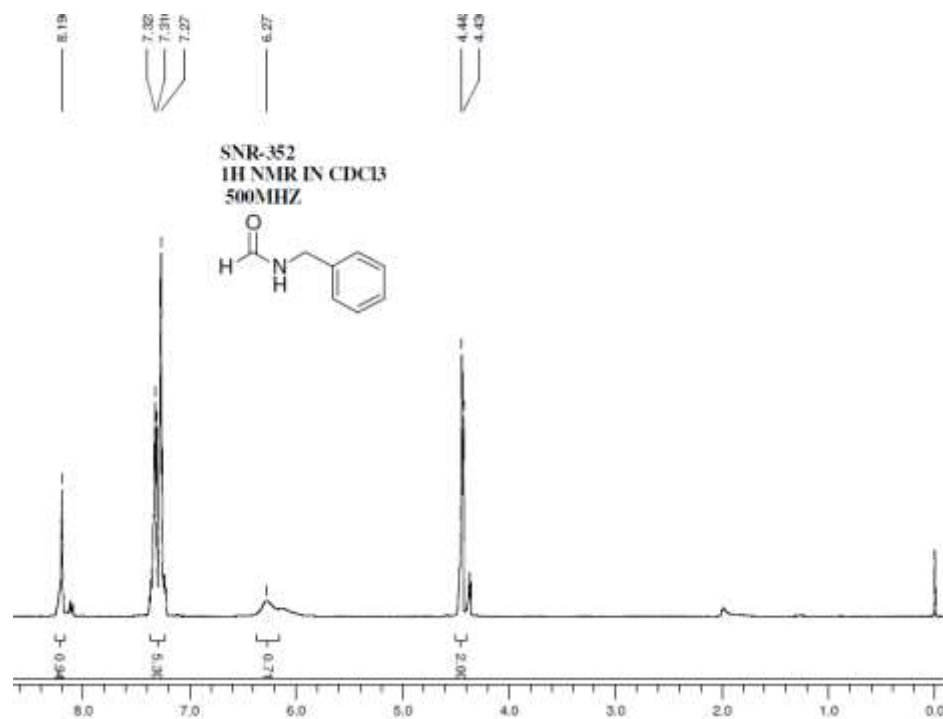




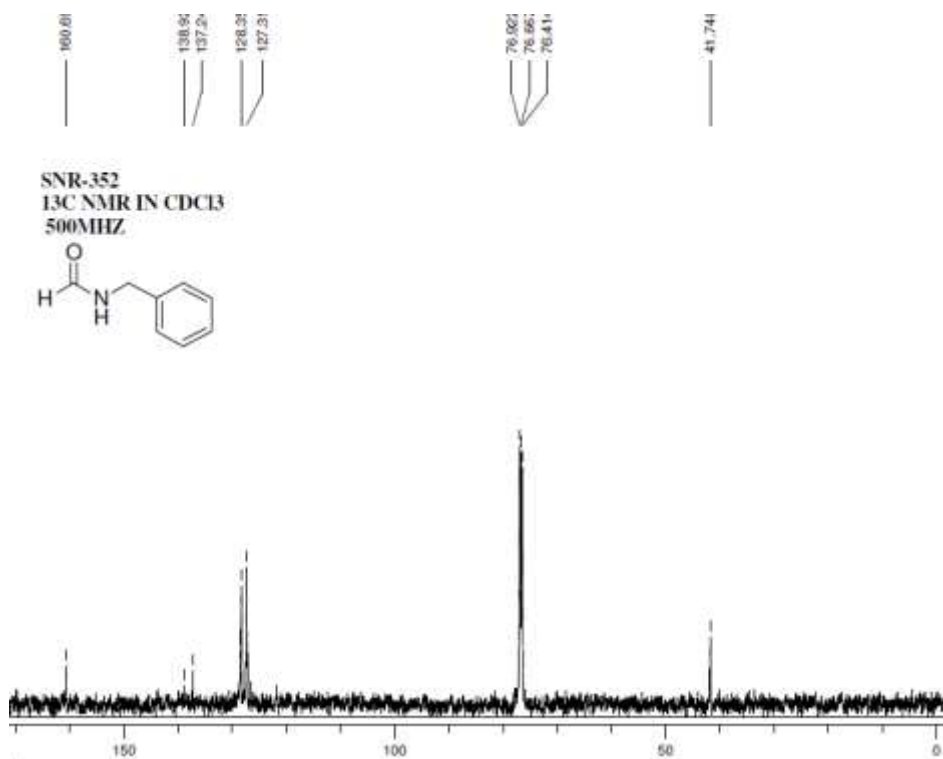
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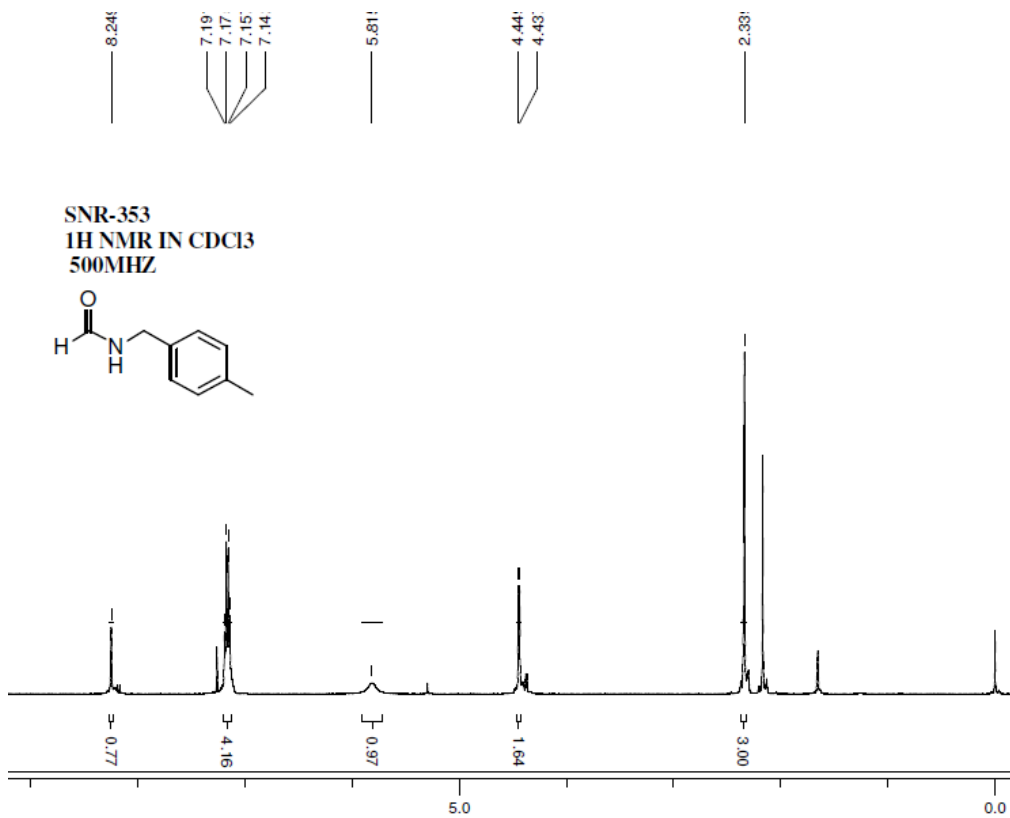
13C NMR of **4n**



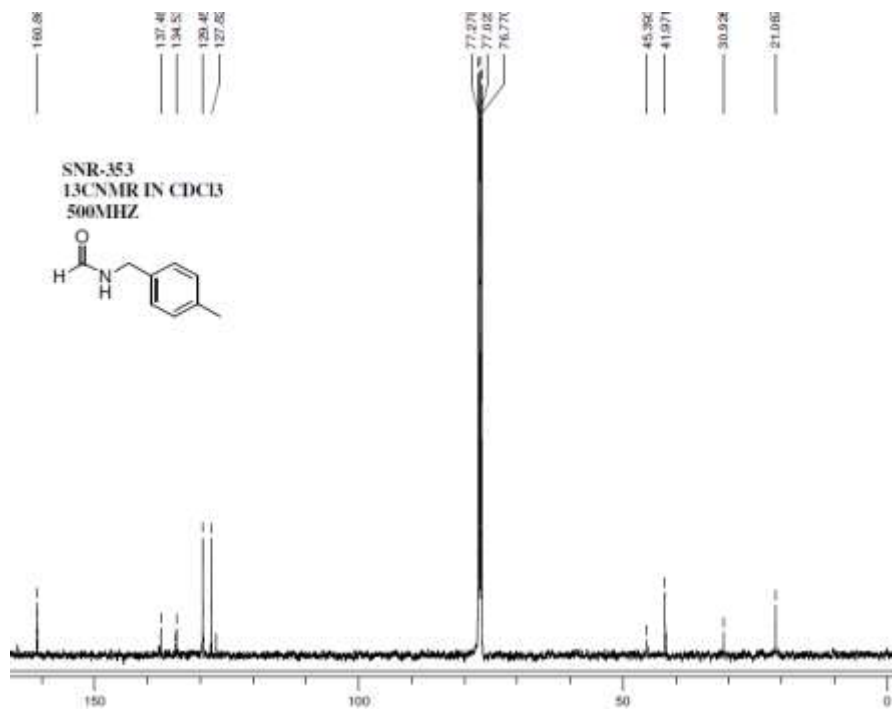
1H NMR of **4o**



13C NMR of **4o**

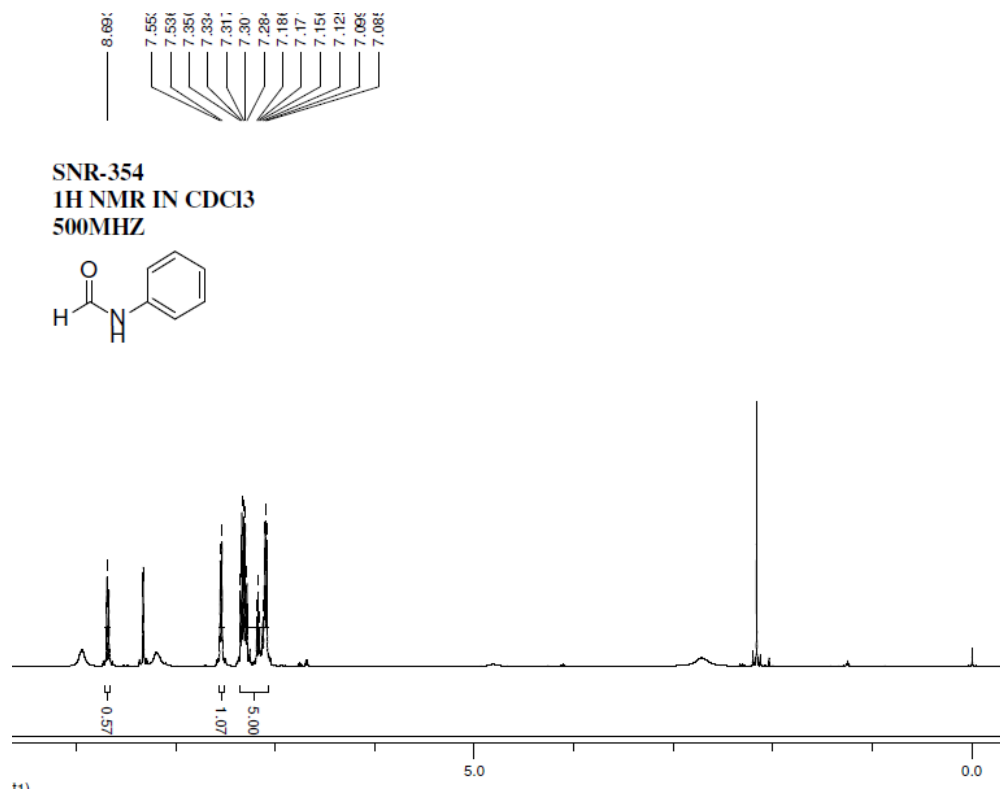


1H NMR of 4p

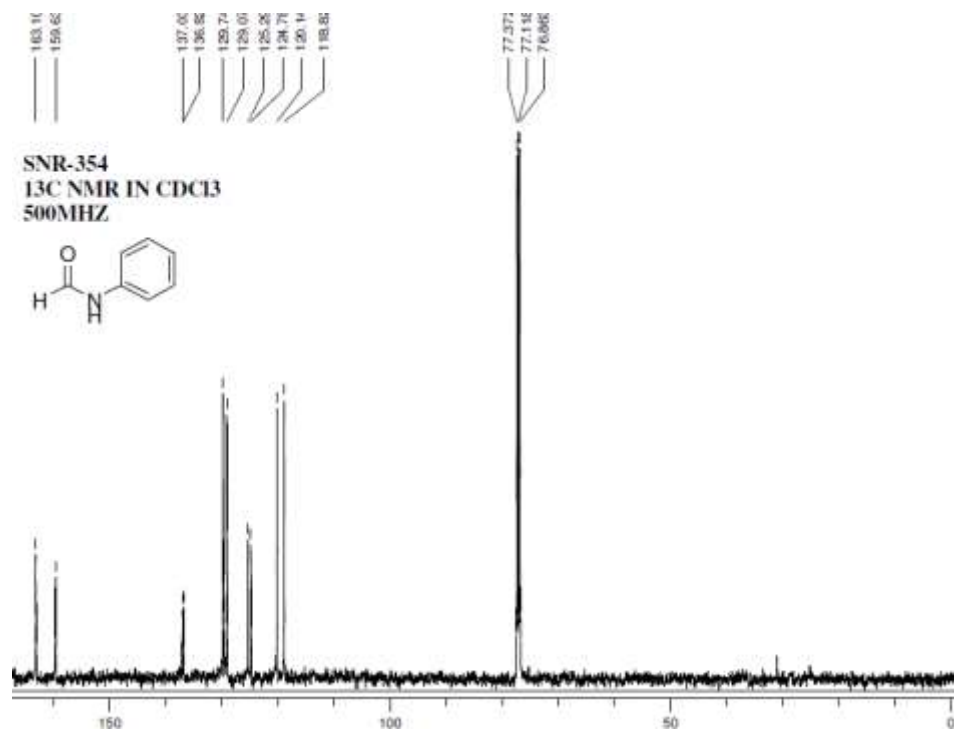


13C NMR of 4p

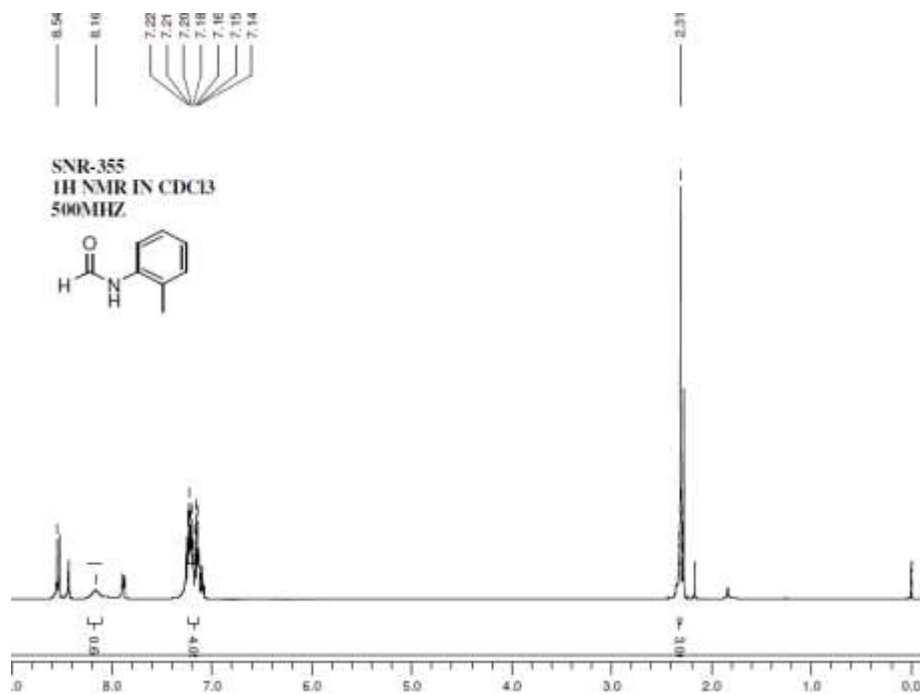




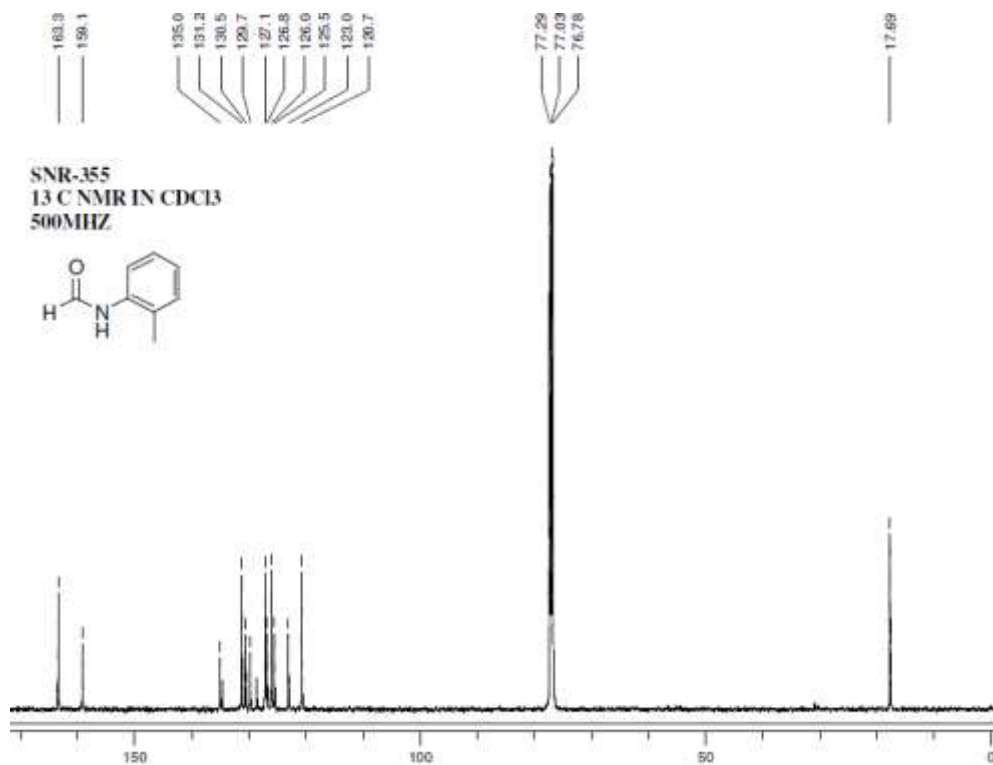
1H NMR of **4q**



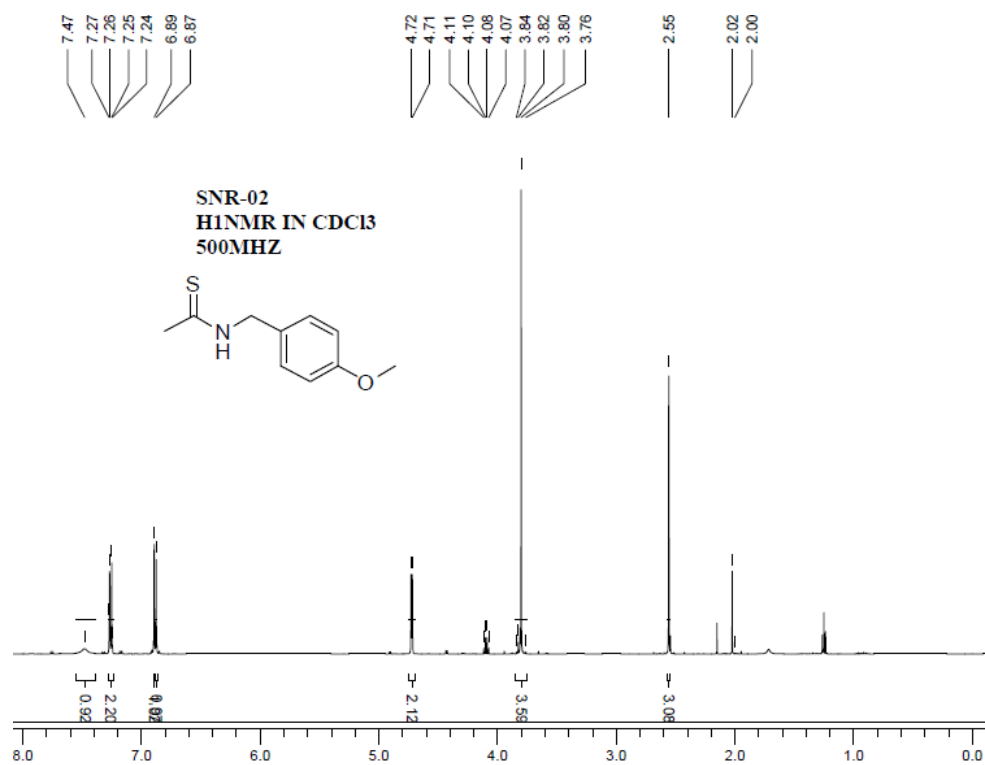
13C NMR of **4q**



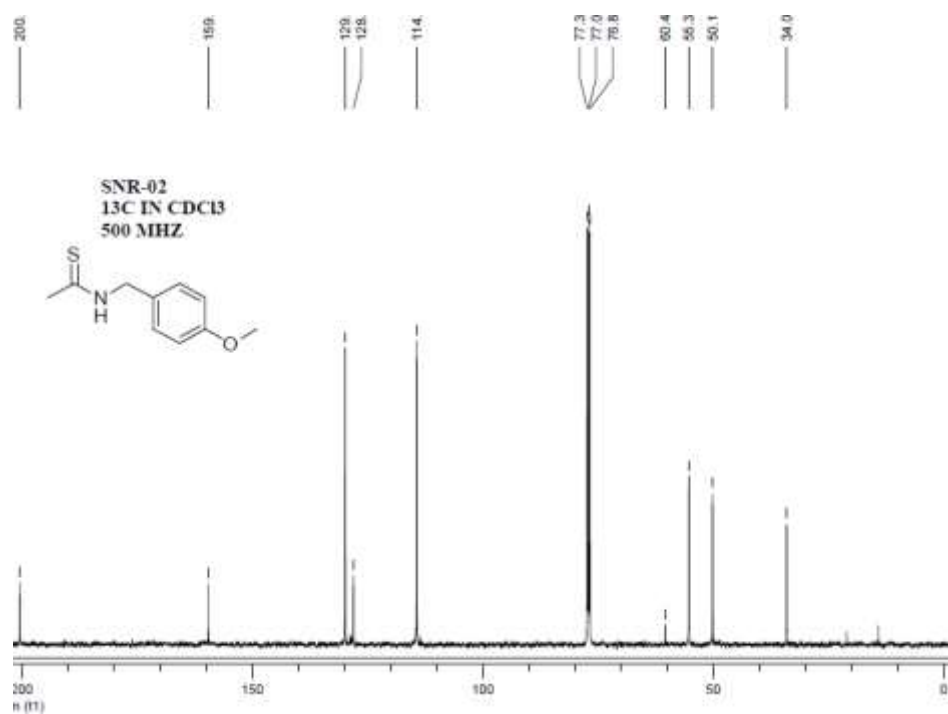
1H NMR of **4r**



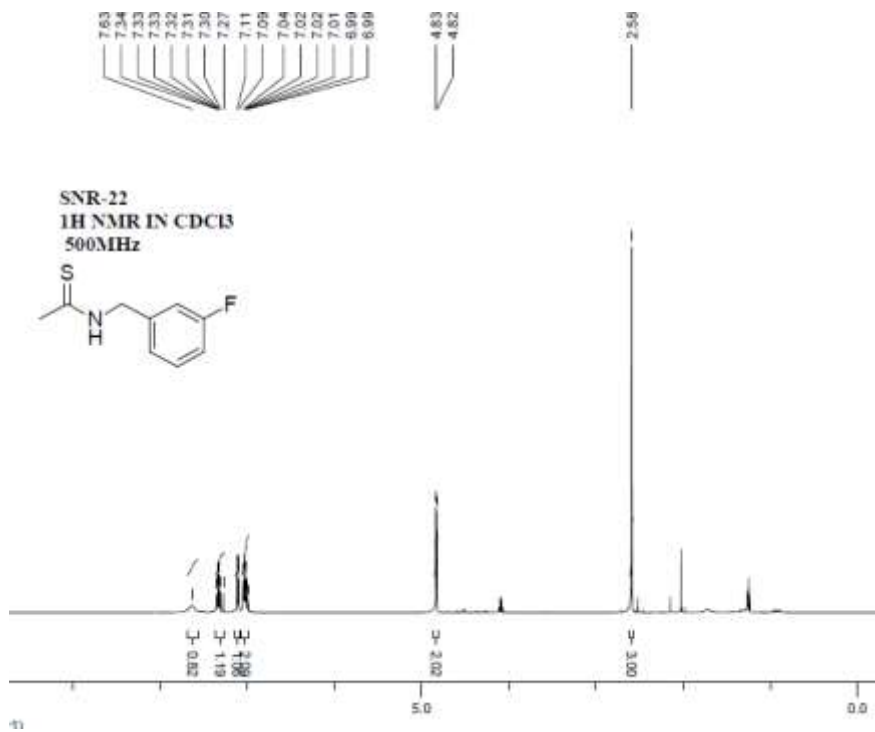
13C NMR of **4r**



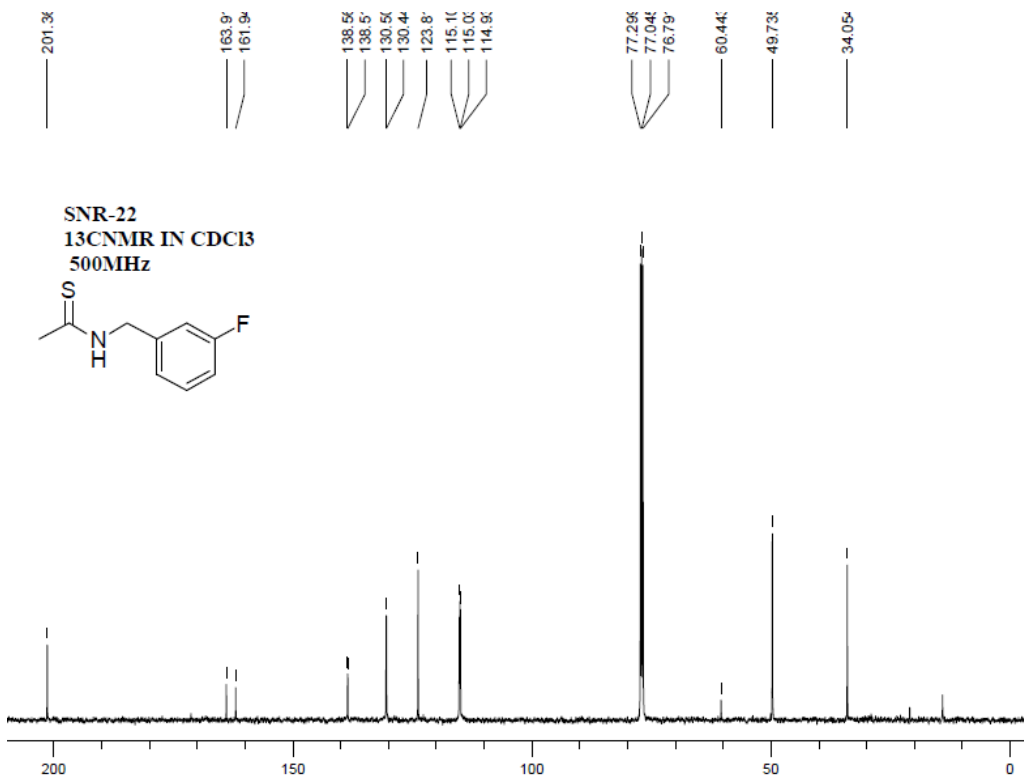
1H NMR of 5a



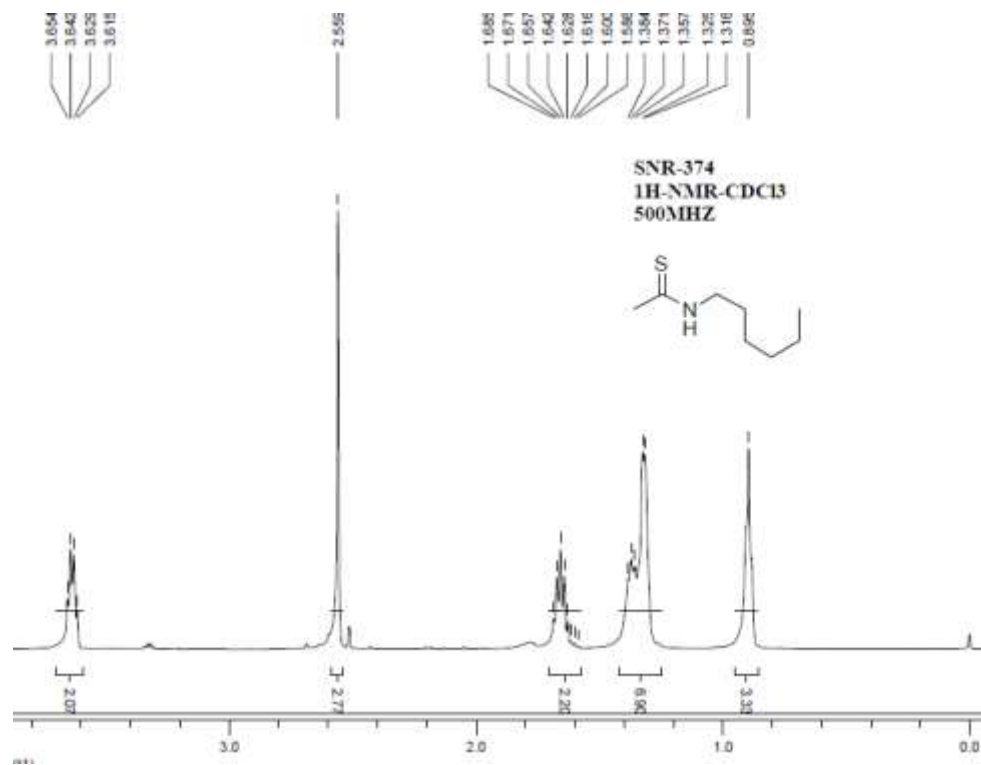
13C NMR of 5a



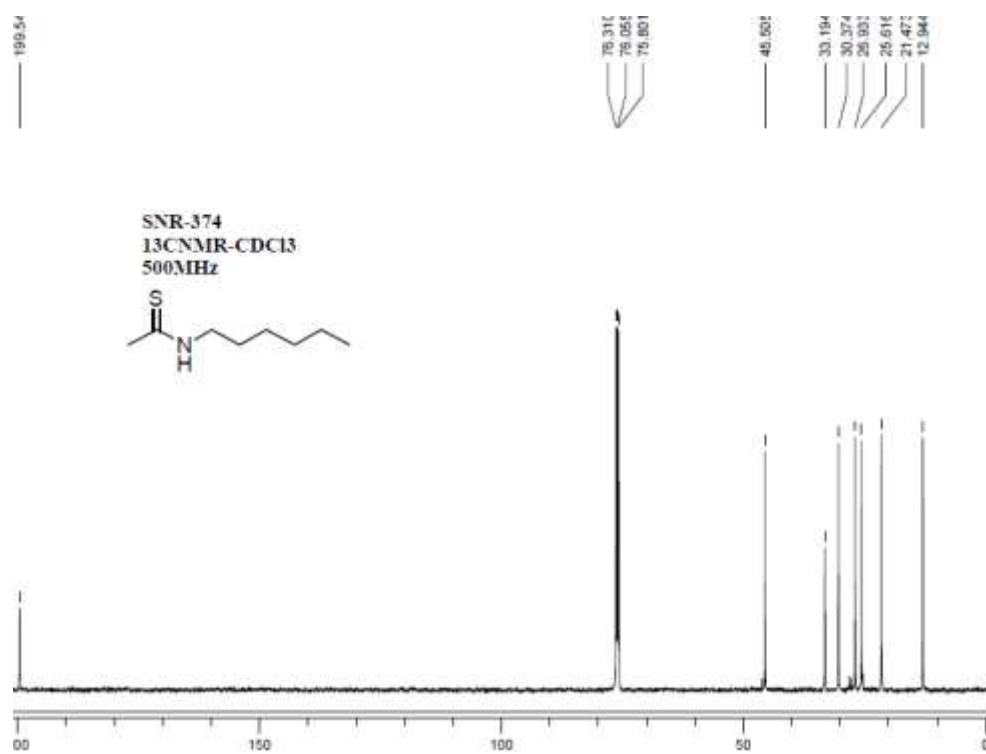
1H NMR of **5b**



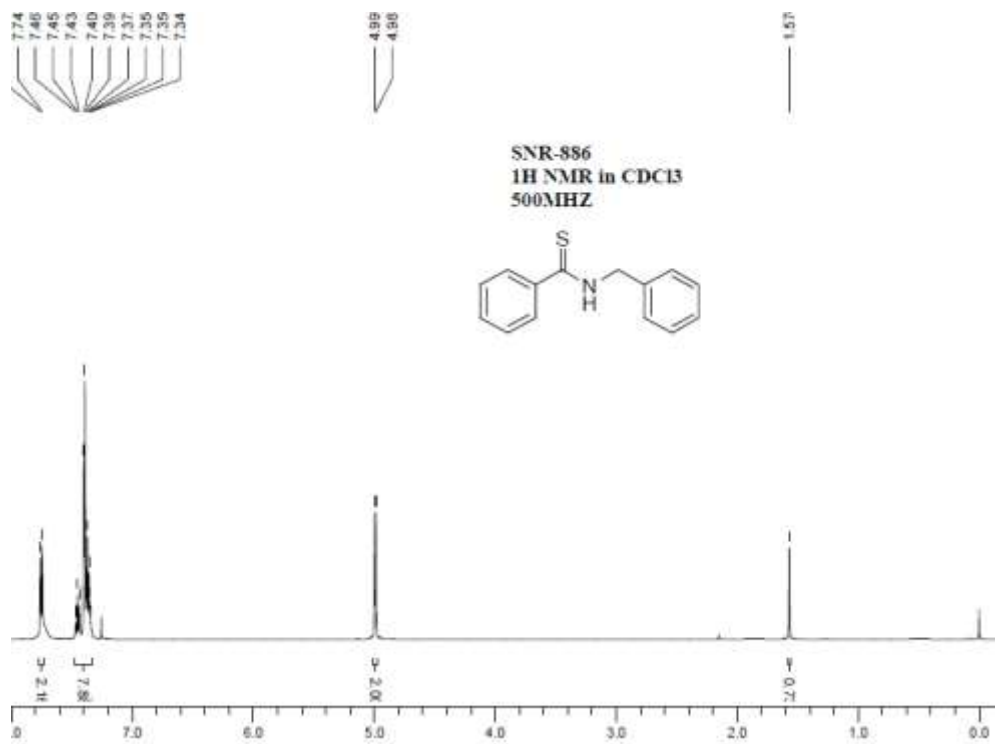
13C NMR of **5b**



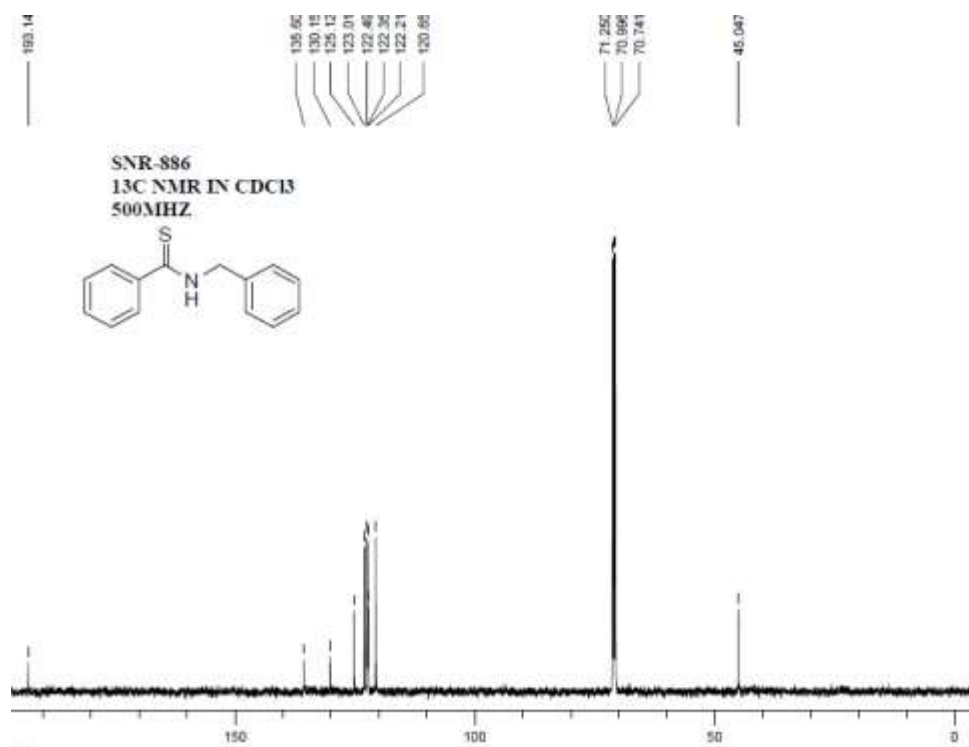
1H NMR of 5c



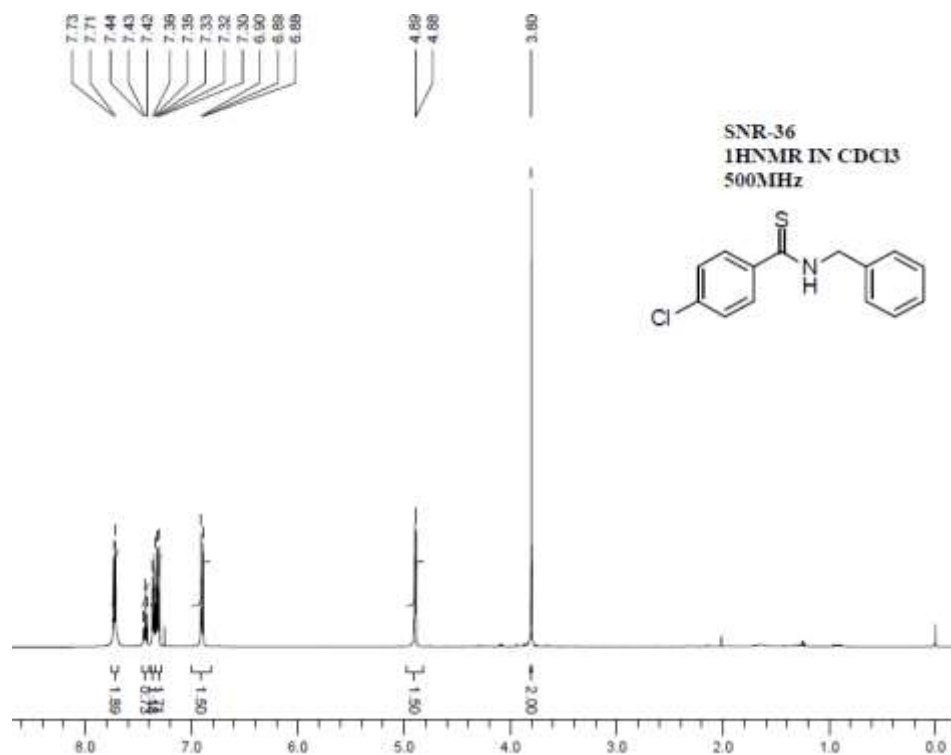
13C NMR of 5c



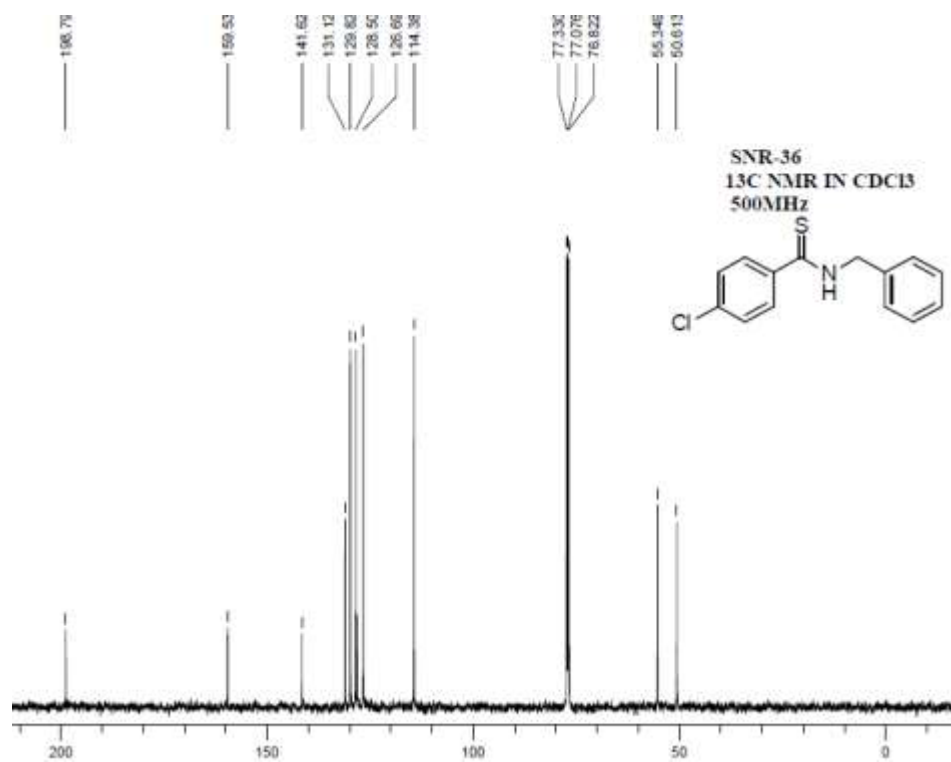
1H NMR of 5d



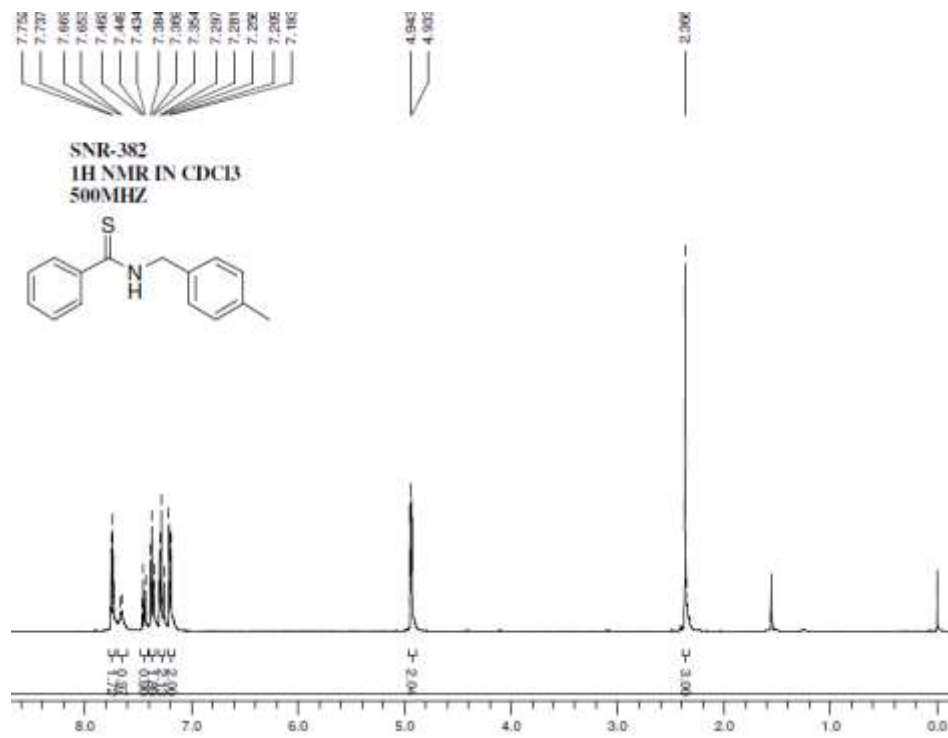
13C NMR of 5d



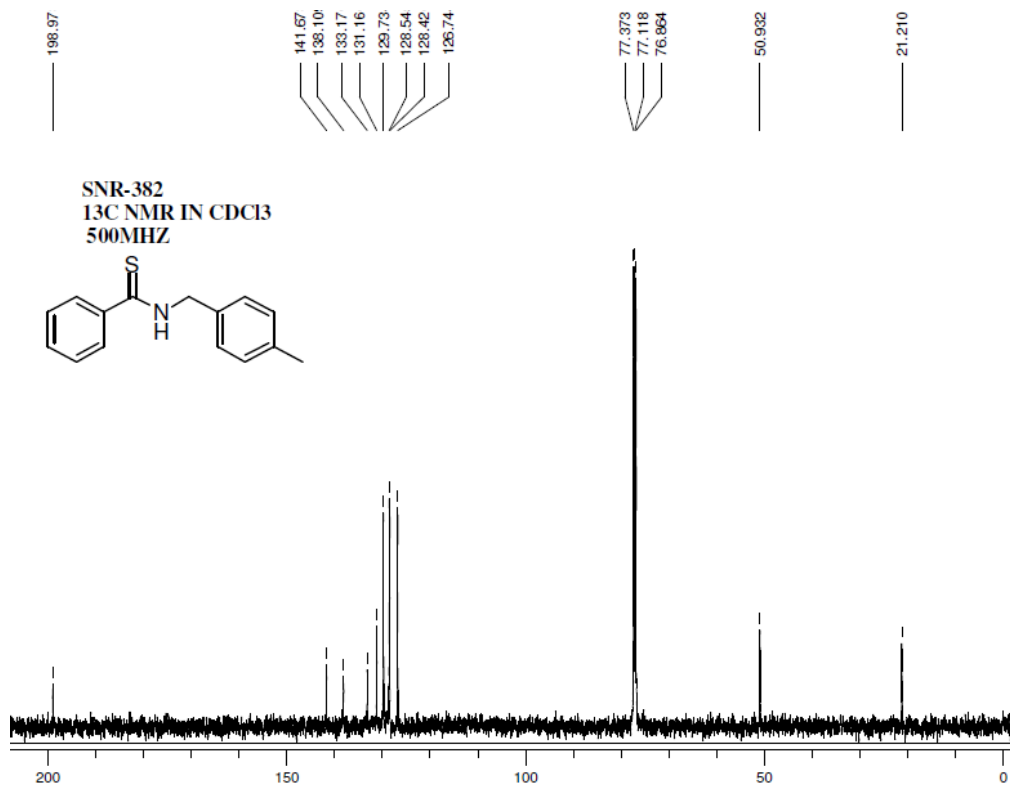
1H NMR of **5e**



13C NMR of **5e**



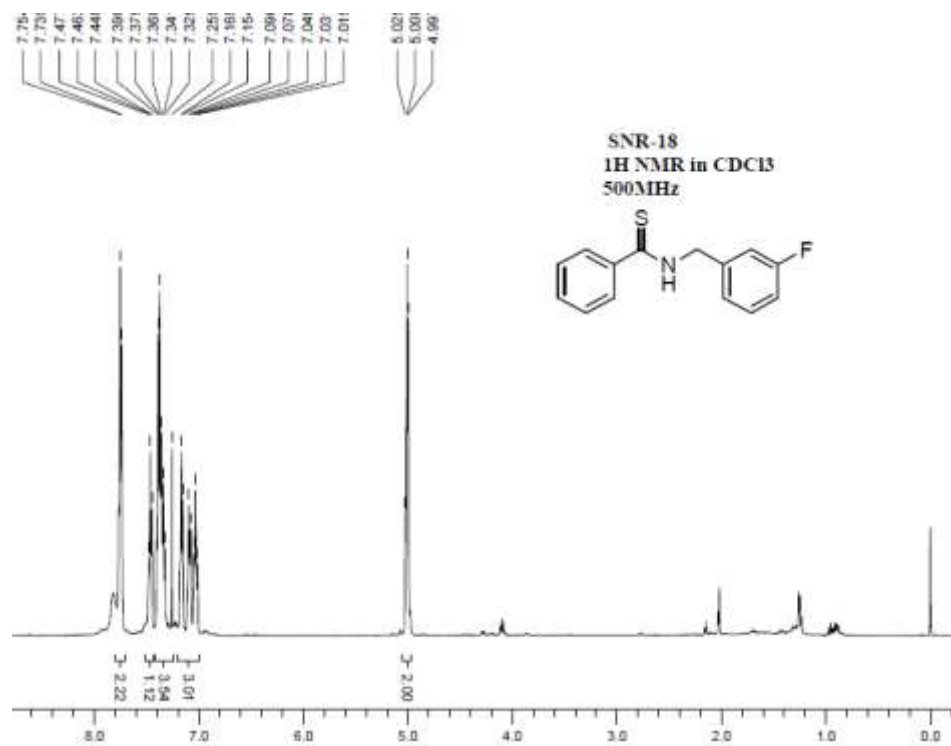
1H NMR of **5f**



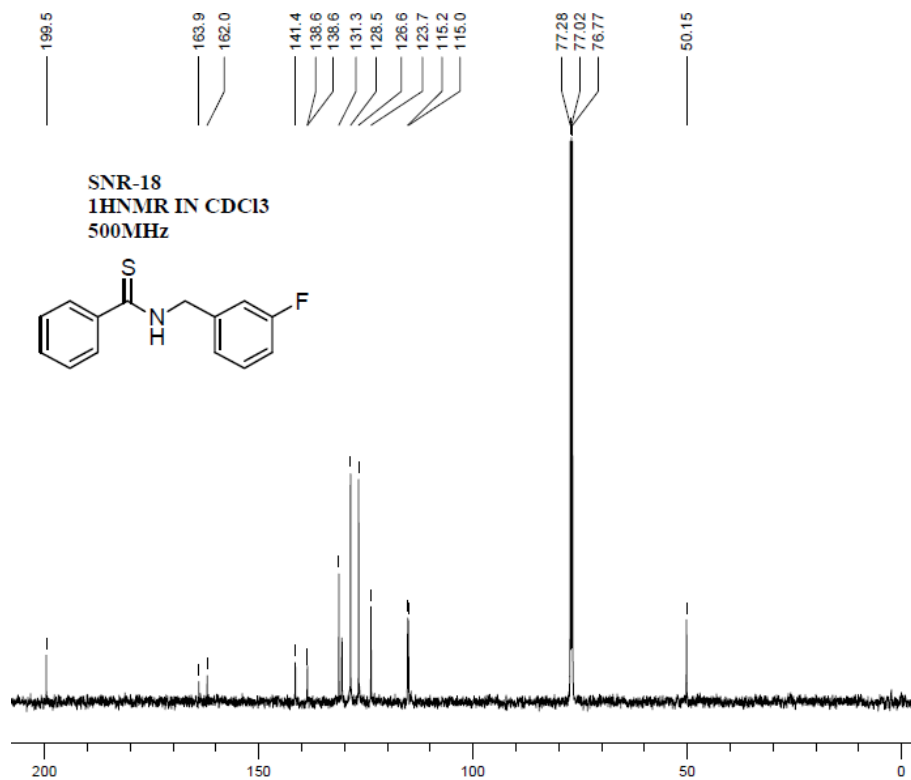
13C NMR of **5f**





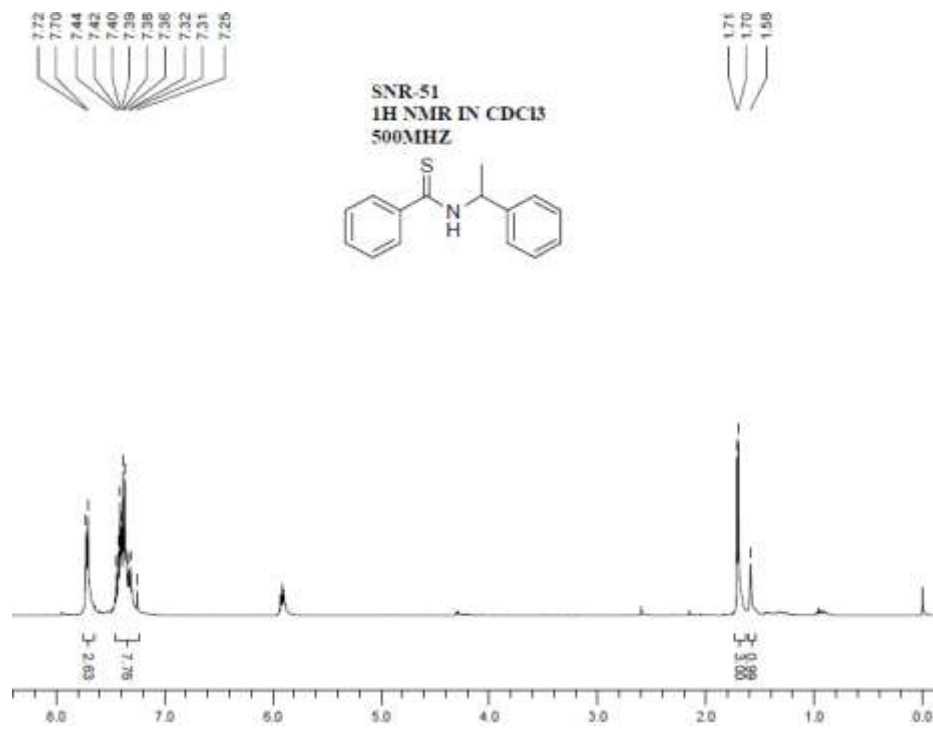


1H NMR of 5h

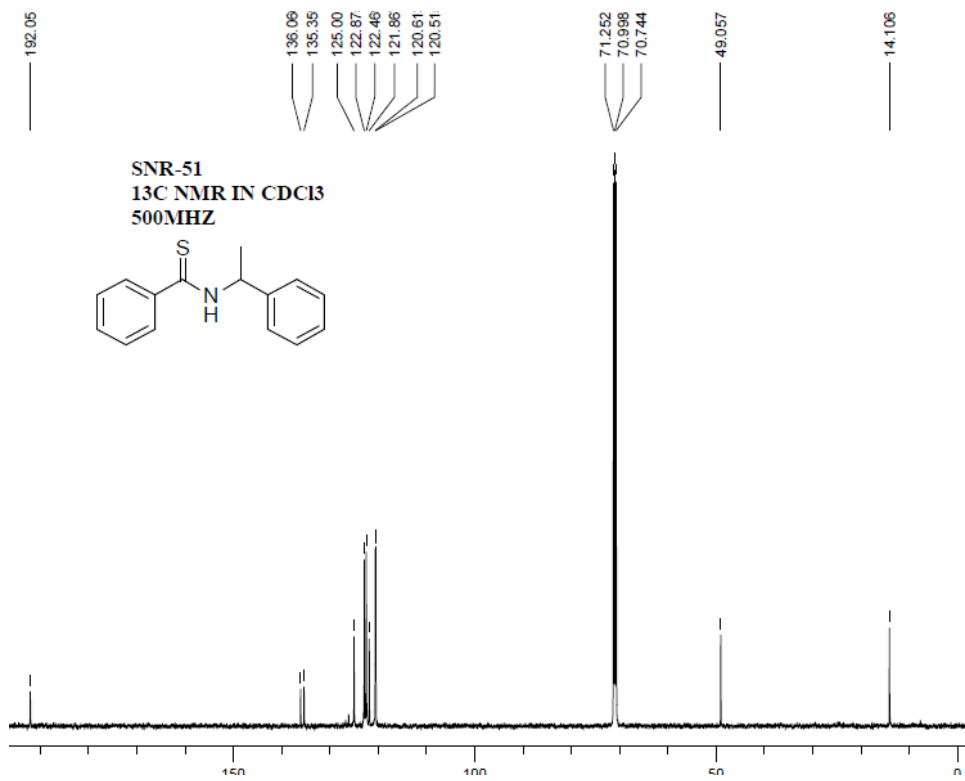


13C NMR of 5h

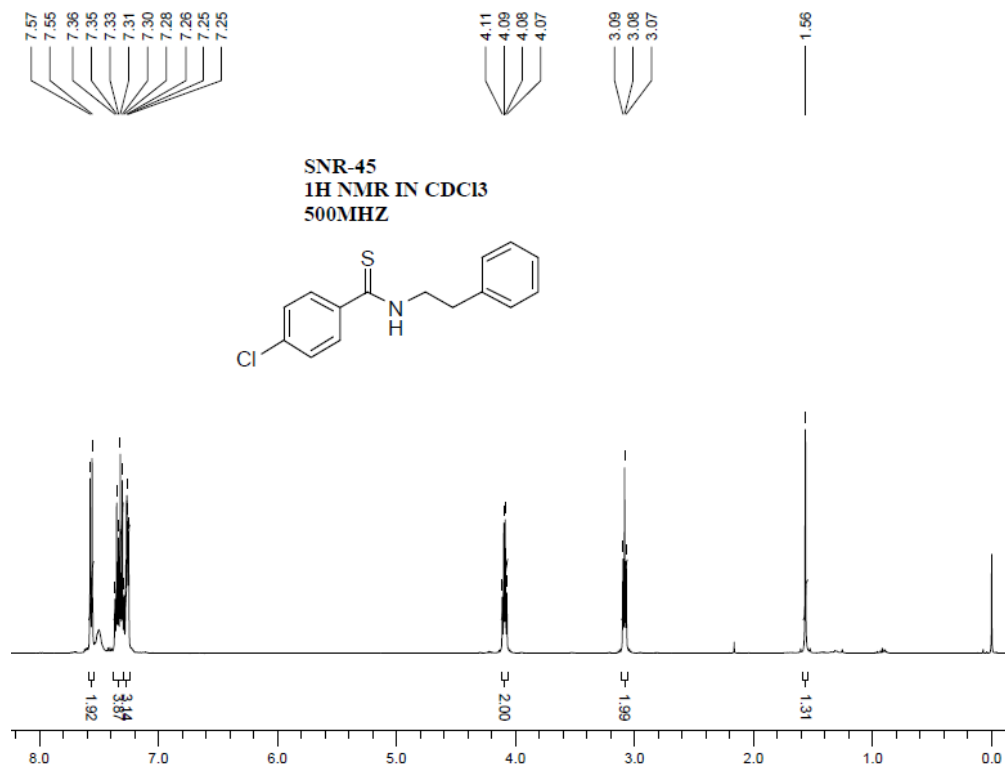




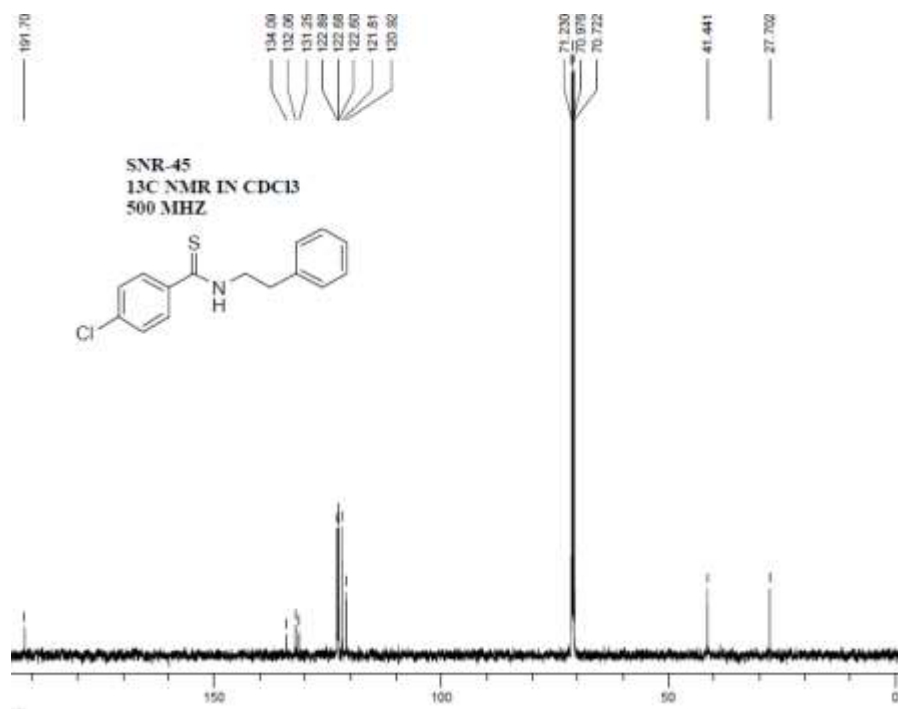
1H NMR of **5j**



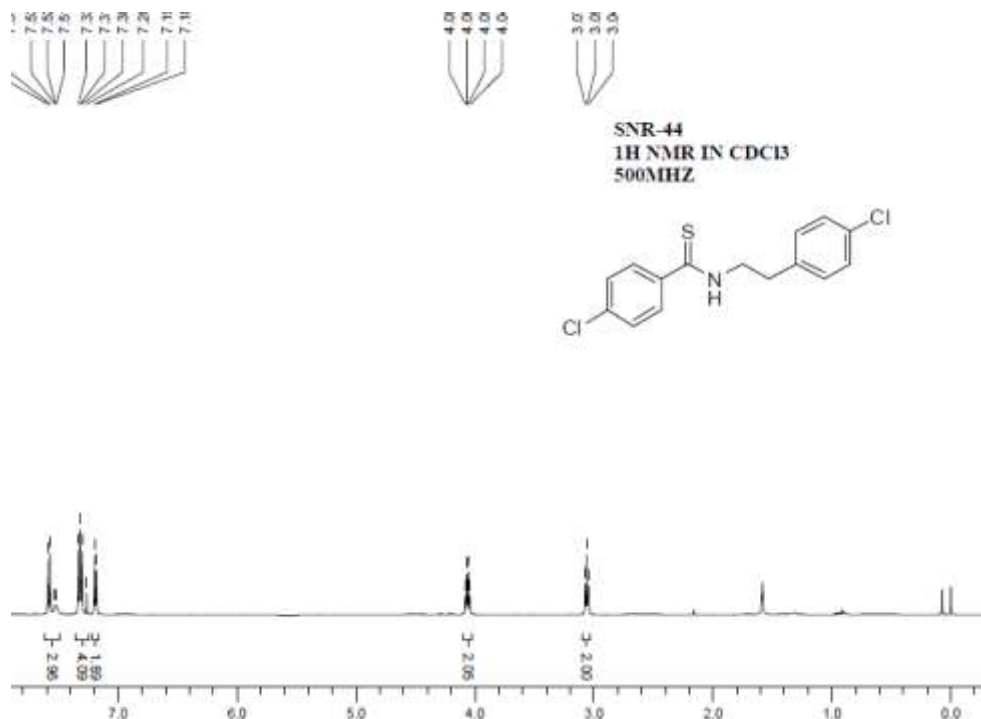
13C NMR of **5j**



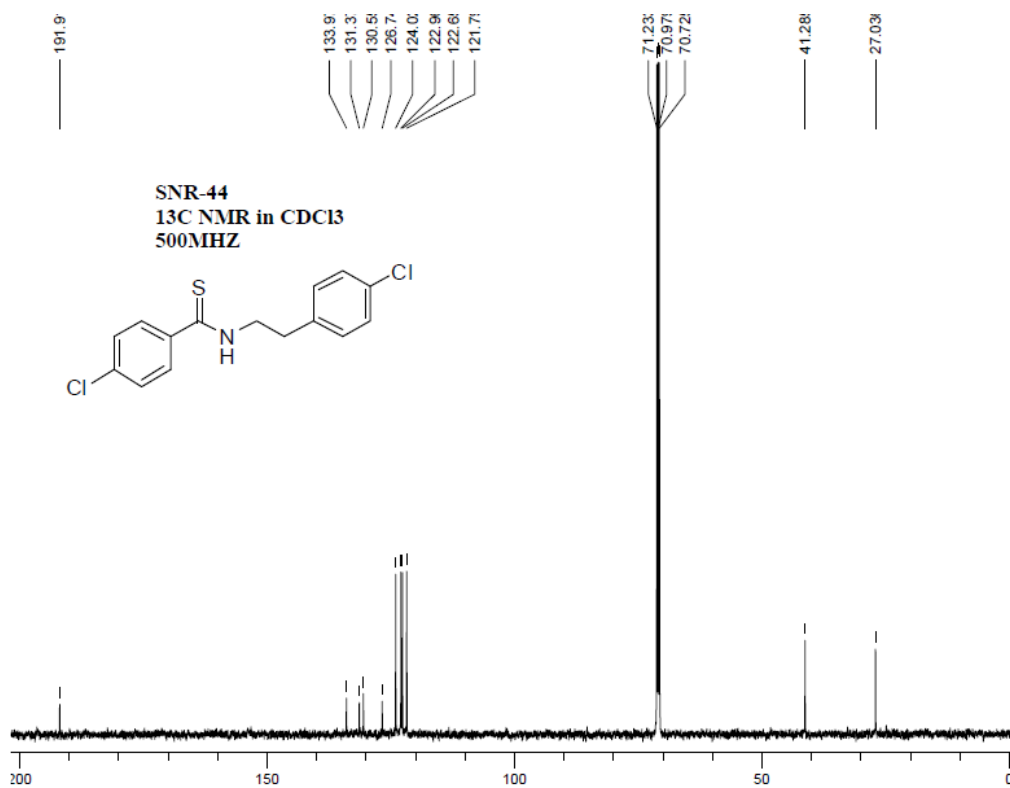
1H NMR of 5k



13C NMR of 5k

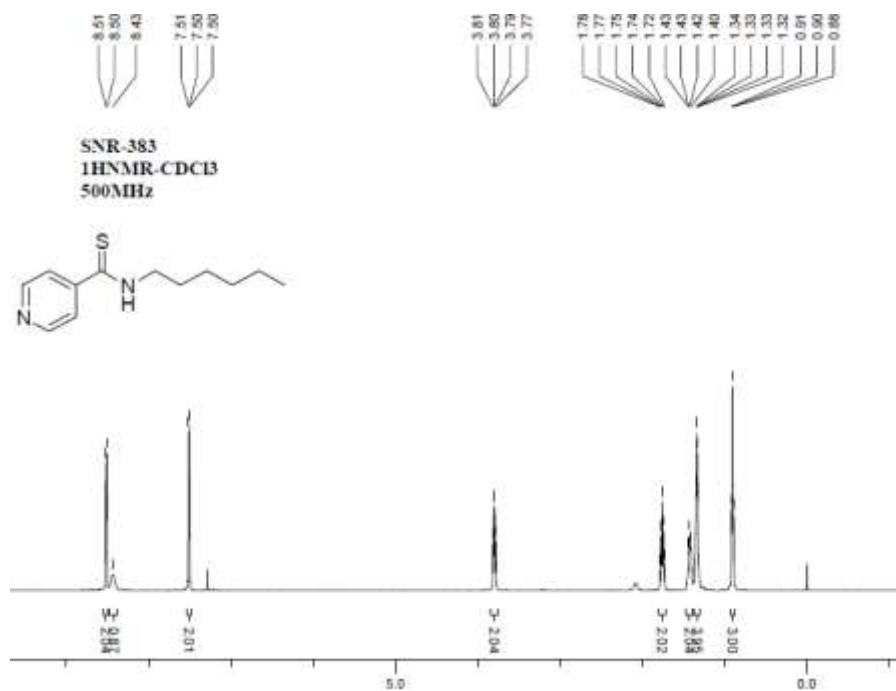


**<sup>1</sup>H NMR of 51**

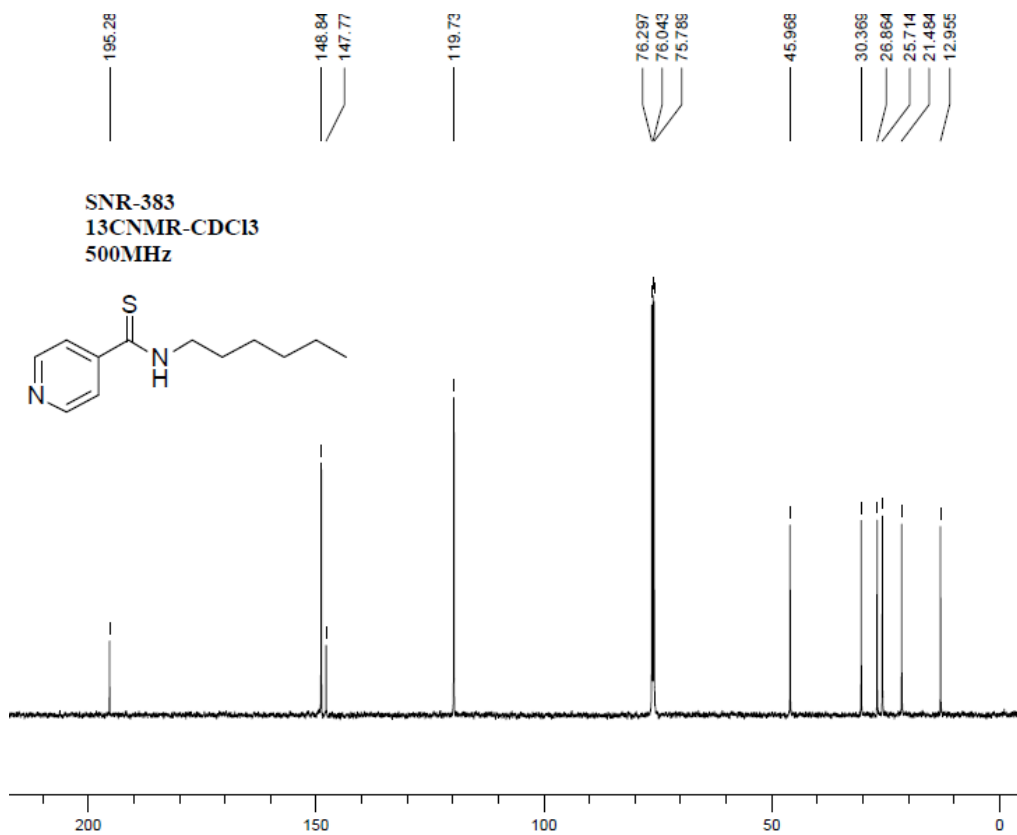


**<sup>13</sup>C NMR of 51**





$^1\text{H NMR}$  of **5n**



$^{13}\text{C NMR}$  of **5n**



