

# **Mechanistic studies on a Cu-catalyzed aerobic oxidative coupling reaction with *N*-phenyl tetrahydroisoquinolines: structure of intermediates and the role of methanol as a solvent**

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## **Supporting Information**

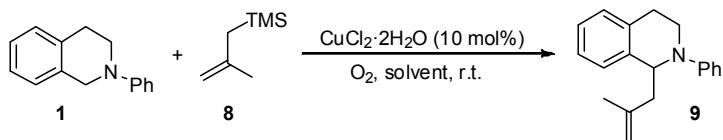
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## **Experimental procedures**

### **General methods**

Unless otherwise indicated, all reagents and solvents were purchased from commercial distributors and used as received. Solvents (ethyl acetate, pentane) used for column chromatography were of technical grade and used after distillation in a rotary evaporator. TLC was used to check the reactions for full conversion and was performed on Macherey-Nagel Polygram Sil G/UV<sub>254</sub> thin layer plates. TLC spots were visualized by UV-light irradiation and staining with KMnO<sub>4</sub>. Flash column chromatography was carried out using Merck Silica Gel 60 (40-63 µm). Yields refer to pure isolated compounds. <sup>1</sup>H and <sup>13</sup>C NMR spectra were measured with Bruker Avance 600, AV 500 and AV 400 spectrometers. All chemical shifts are given in ppm downfield relative to TMS and were referenced to TMS or the solvent residual peaks. <sup>1</sup>H NMR chemical shifts are designated using the following abbreviations as well as their combinations: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad signal. High resolution mass spectra were recorded with a Bruker APEX III FTICR-MS or a Finnigan SSQ 7000 quadrupole MS or a Finnigan MAT 95 double focusing sector field MS instrument.

### Oxidative allylation - Optimisation study



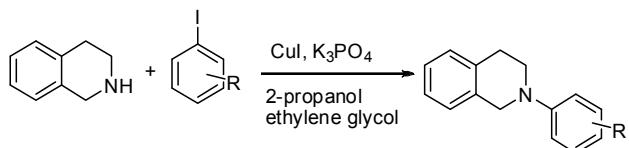
**Table S1:** Screening of solvents for the copper-catalyzed allylation.<sup>[a]</sup>

Entry	Solvent	Time (h)	Conversion <sup>[b]</sup>	Yield (%) <sup>[c]</sup>
1	MeOH	15	100%	89
2	Acetone	24	100%	76
3	DMF	24	100%	70
4	CHCl <sub>3</sub>	24	<50%	n.d.
5	CH <sub>2</sub> Cl <sub>2</sub>	24	<50%	n.d.
6	Toluene	24	<50%	n.d.

[a] 1 (0.12 mmol), 8 (0.36 mmol), CuCl<sub>2</sub>·2H<sub>2</sub>O (10 mol%), 0.5 ml solvent, O<sub>2</sub> (balloon), stirring at room temperature. [b] Monitored and estimated by TLC. [c] Isolated yields.

In acetone and DMF, the yields were still good but several side products were observed and the reaction time had to be increased to 24 hours to achieve full conversion (Table S1, entries 2 and 3). Performing the reaction in toluene, dichloromethane and chloroform resulted in less than 50% conversion of starting material, significant amounts of side products and only traces of product after one day (Table S1, entries 4-6). Alternative Cu and Fe salts were also tested as catalysts but without giving the same or better yield than CuCl<sub>2</sub>·2H<sub>2</sub>O. No reaction occurred if allyl-trimethylsilane was used instead of 2-methylallyl-trimethylsilane 8.

### General procedure for the preparation of starting materials



The tetrahydroisoquinolines were synthesised according to a reported method.<sup>1</sup> A representative procedure is given for 2-(4-tert-butylphenyl)-1,2,3,4-tetrahydroisoquinoline: copper(I)iodide (87.4 mg, 0.46 mmol) and potassium phosphate (2.0 g, 9.18 mmol) were placed into a Schlenk-tube equipped with a magnetic stirrer. The tube was evacuated and back filled with argon. 2-Propanol (4.6 mL), ethylene glycol (0.52 mL, 9.18 mmol), 1,2,3,4-tetrahydroisoquinoline (1.06 mL, 6.88 mmol) and 1-tert-Butyl-4-iodobenzene (0.81 mL, 4.59 mmol) were added successively by microsyringe at room temperature. The reaction mixture was heated at 85 °C for 24 h and then allowed to cool to room temperature. Diethyl ether (5 mL) and water (5 mL) were added to the reaction mixture and the organic layer was separated. The aqueous layer was further extracted by diethyl ether (2 x 5 mL) and the combined organic phases were dried over magnesium sulfate. The solvent was removed by rotary evaporation and the crude product was purified by column chromatography.

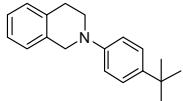
### General procedure for the oxidative allylation

To a solution of amine (0.12 mmol) in methanol (0.5 mL), was added 2-methylallyl-trimethylsilane 8 (0.36 mmol, 3 equiv.) and CuCl<sub>2</sub>-dihydrate (2 mg, 10 mol%) at room temperature. After stirring for the appropriate time under an atmosphere of oxygen, the solution was concentrated in vacuo. The crude residue was directly purified by flash column chromatography on silica gel (5-10% EtOAc/pentane) to give the corresponding allylated amine.

<sup>1</sup> Li, Z.; Li, C.-J. *J. Am. Chem. Soc.* **2005**, 127, 6968.

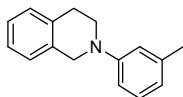
### Characterisation of starting materials

#### 2-(4-*tert*-Butylphenyl)-1,2,3,4-tetrahydroisoquinoline



Yield : 859 mg, 71%; White solid,  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.35-7.30 (m, 2H), 7.21-7.14 (m, 4H), 6.98-6.93 (m, 2H), 4.39 (s, 2H), 3.54 (t,  $J = 5.9$  Hz, 2H), 2.99 (t,  $J = 5.9$  Hz, 2H), 1.31 (s, 9H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  148.47, 141.64, 134.98, 134.72, 128.69, 126.68, 126.37, 126.11, 126.08, 115.20, 51.24, 46.88, 34.05, 31.63, 29.34; HRMS-(EI) ( $m/z$ ):  $M^+$  calcd for  $\text{C}_{19}\text{H}_{23}\text{N}_1$ , 265.183051; found 265.183112.

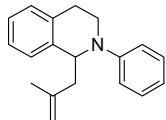
#### 2-*m*-Tolyl-1,2,3,4-tetrahydroisoquinoline



Yield : 738 mg, 72%; Clear oil,  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.22-7.15 (m, 5H), 6.86-6.79 (m, 2H), 6.67-6.65 (m, 1H), 4.41 (s, 2H), 3.56 (t,  $J = 5.85$  Hz, 2H), 3.0 (t,  $J = 5.85$  Hz, 2H), 2.36 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  150.77, 139.02, 135.02, 134.67, 129.18, 128.65, 126.67, 126.42, 126.13, 119.77, 116.10, 112.47, 50.98, 46.72, 29.33, 22.0; HRMS-(EI) ( $m/z$ ):  $M^+$  calcd for  $\text{C}_{16}\text{H}_{17}\text{N}_1$ , 223.136096; found 223.135852.

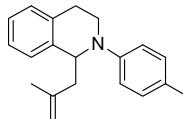
### Characterisation of products

#### 1-(2-Methylallyl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (9)



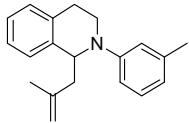
Reaction time 15h,  $r_f = 0.70$  (EtOAc/pentane, 0.5 : 9.5); Yield : 28 mg, 89%;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.17-7.14 (m, 2H), 7.09-7.01 (m, 4H), 6.85 (bs, 2H), 6.67 (bs, 1H), 4.78 (t,  $J = 7.1$  Hz, 1H), 4.73 (bs, 1H), 4.59 (bs, 1H), 3.56 (t,  $J = 5.6$  Hz, 1H), 2.98-2.92 (m, 1H), 2.79-2.76 (m, 1H), 2.63 (dd,  $J = 5.5, 13.4$  Hz, 1H), 2.33 (dd,  $J = 7.2, 13.4$  Hz, 1H), 1.71 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  149.67, 143.05, 138.62, 134.88, 129.37, 128.67, 127.58, 126.61, 125.72, 117.51, 114.36, 113.65, 58.34, 44.75, 41.82, 27.09, 23.07; HR - MS m/z: calcd for  $\text{C}_{19}\text{H}_{21}\text{N}[\text{M}^+]$ : 263.1674; found: 263.1671.

#### 1-(2-Methylallyl)-2-(*p*-tolyl)-1,2,3,4-tetrahydroisoquinoline (10):



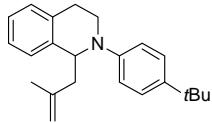
Reaction time 12h,  $r_f = 0.70$  (EtOAc/pentane, 0.5 : 9.5); Yield : 26 mg, 78%;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.15-7.07 (m, 4H), 7.03 (d,  $J = 8.3$  Hz, 2H), 6.82 (d,  $J = 8.3$  Hz, 2H), 4.79 (t,  $J = 6.6$  Hz, 2H), 4.66 (bs, 1H), 3.62-3.59 (m, 2H), 3.03-2.97 (m, 1H), 2.77 (td,  $J = 4.3, 16.2$  Hz, 1H), 2.67 (dd,  $J = 7.2, 13.8$  Hz, 1H), 2.39 (dd,  $J = 6.9, 13.8$  Hz, 1H), 2.24 (s, 3H), 1.77 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  147.67, 143.22, 138.73, 134.84, 129.85, 128.76, 127.55, 127.04, 126.48, 125.64, 115.12, 113.43, 58.38, 44.69, 41.89, 26.81, 22.99, 20.43; HR - MS m/z: calcd for  $\text{C}_{20}\text{H}_{23}\text{N}[\text{M}^+]$ : 277.1830; found: 277.1829.

**1-(2-Methylallyl)-2-(m-tolyl)-1,2,3,4-tetrahydroisoquinoline (13):**



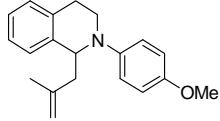
Reaction time 12h,  $r_f = 0.70$  (EtOAc/pentane, 0.5 : 9.5); Yield : 25 mg, 75%;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.14-7.06 (m, 5H), 6.73 (bs, 1H), 6.71 (d,  $J = 7.4$  Hz, 1H), 6.56 (d,  $J = 7.4$  Hz, 1H), 4.84 (t,  $J = 7.1$  Hz, 1H), 4.81-4.79 (m, 1H), 4.66-4.65 (m, 1H), 3.62 (dd,  $J = 5.1, 7.2$  Hz, 2H), 3.05-2.98 (m, 1H), 2.82 (td,  $J = 5.0, 16.1$  Hz, 1H), 2.69 (dd,  $J = 6.7, 13.6$  Hz, 1H), 2.39 (dd,  $J = 6.8, 13.6$  Hz, 1H), 2.30 (s, 3H), 1.78 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  149.70, 143.08, 138.96, 138.67, 134.91, 129.20, 128.64, 127.57, 126.56, 125.66, 118.43, 115.11, 113.61, 111.52, 58.29, 44.75, 41.82, 27.14, 23.05, 22.10; HR - MS m/z: calcd for  $\text{C}_{20}\text{H}_{23}\text{N}[\text{M}^+]$ : 277.1830; found: 277.1831.

**2-(4-(tert-Butyl)phenyl)-1-(2-methylallyl)-1,2,3,4-tetrahydroisoquinoline (11):**



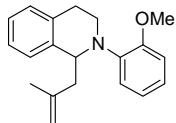
Reaction time 12h,  $r_f = 0.80$  (EtOAc/pentane, 0.5 : 9.5); Yield : 28 mg, 73%;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.25 (d,  $J = 8.9$  Hz, 2H), 7.13-7.05 (m, 4H), 6.85 (d,  $J = 8.9$  Hz, 2H), 4.81-4.79 (m, 2H), 4.66-4.65 (m, 1H), 3.63-3.59 (m, 2H), 3.06-2.98 (m, 1H), 2.80 (td,  $J = 3.7, 16.1$  Hz, 1H), 2.69 (dd,  $J = 6.6, 13.3$  Hz, 1H), 2.39 (dd,  $J = 7.1, 13.3$  Hz, 1H), 1.79 (s, 3H), 1.27 (s, 9H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  147.37, 143.17, 140.11, 138.77, 134.95, 128.66, 127.58, 126.51, 126.13, 125.64, 114.02, 113.54, 58.57, 44.90, 41.80, 33.91, 31.65, 26.98, 23.12; HR - MS m/z: calcd for  $\text{C}_{23}\text{H}_{29}\text{N}[\text{M}^+]$ : 319.2299; found: 319.2297.

**2-(4-Methoxyphenyl)-1-(2-methylallyl)-1,2,3,4-tetrahydroisoquinoline (12):**



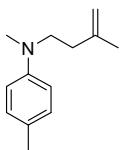
Reaction time 24h,  $r_f = 0.65$  (EtOAc/pentane, 1 : 9); Yield : 20 mg, 56%;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.13-7.08 (m, 4H), 6.87 (d,  $J = 8.9$  Hz, 2H), 6.80 (d,  $J = 8.9$  Hz, 2H), 4.79 (bs, 1H), 4.69 (t,  $J = 7.0$  Hz, 1H), 4.66 (bs, 1H), 3.75 (s, 3H), 3.58-3.55 (m, 2H), 3.00-2.94 (m, 1H), 2.72 (td,  $J = 3.8, 16.3$  Hz, 1H), 2.63 (dd,  $J = 7.5, 13.9$  Hz, 1H), 2.39 (dd,  $J = 6.6, 13.9$  Hz, 1H), 1.76 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  152.70, 144.56, 143.37, 138.84, 134.74, 128.92, 127.54, 126.42, 125.65, 117.74, 114.69, 113.23, 58.50, 55.77, 44.02, 42.53, 26.56, 22.88; HR - MS m/z: calcd for  $\text{C}_{20}\text{H}_{23}\text{NONa}[\text{M}^++\text{Na}]$ : 316.1672; found: 316.1670.

**2-(2-Methoxyphenyl)-1-(2-methylallyl)-1,2,3,4-tetrahydroisoquinoline (14):**



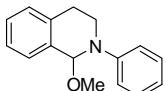
Reaction time 24h,  $r_f = 0.60$  (EtOAc/pentane, 1 : 9); Yield : 29 mg, 82%;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.27-7.19 (m, 4H), 7.09-7.05 (m, 1H), 6.98 (d,  $J = 8.3$  Hz, 1H), 6.97-6.92 (m, 2H), 4.90 (t,  $J = 6.6$  Hz, 1H), 4.84 (bs, 1H), 4.72 (bs, 1H), 3.97 (s, 3H), 3.72-3.62 (m, 2H), 3.09-3.02 (m, 1H), 2.80 (dd,  $J = 2.4, 16.9$  Hz, 1H), 2.68 (dd,  $J = 6.5, 13.9$  Hz, 1H), 2.50 (dd,  $J = 6.7, 13.9$  Hz, 1H), 1.79 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  152.84, 143.76, 140.77, 139.53, 134.38, 129.14, 127.46, 126.18, 125.43, 122.58, 121.60, 121.18, 112.87, 112.57, 57.74, 55.90, 44.12, 42.19, 27.35, 22.51; HR - MS m/z: calcd for  $\text{C}_{20}\text{H}_{23}\text{NONa}[\text{M}^++\text{Na}]$ : 316.1672; found: 316.1672.

**N,4-Dimethyl-N-(3-methylbut-3-en-1-yl)aniline (15):**



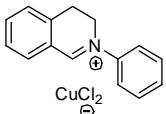
Reaction time 3 d,  $r_f = 0.70$  (EtOAc/pentane, 0.5 : 9.5); Yield : 13 mg, 57%;  $^1\text{H}$  NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  7.04 (d,  $J = 7.5$  Hz, 2H), 6.64 (d,  $J = 7.5$  Hz, 2H), 4.78 (bs, 1H), 4.72 (bs, 1H), 3.41 (t,  $J = 7.8$  Hz, 2H), 2.90 (s, 3H), 2.25 (s, 5H), 1.77 (s, 3H);  $^{13}\text{C}$  NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  147.18, 143.96, 129.86, 125.53, 112.78, 111.38, 52.03, 38.47, 34.17, 22.95, 20.36; HR - MS m/z: calcd for C<sub>15</sub>H<sub>19</sub>N[M<sup>+</sup>]: 189.1517; found: 189.1516.

**1-Methoxy-2-phenyl-1,2,3,4-tetrahydroisoquinoline (5):**



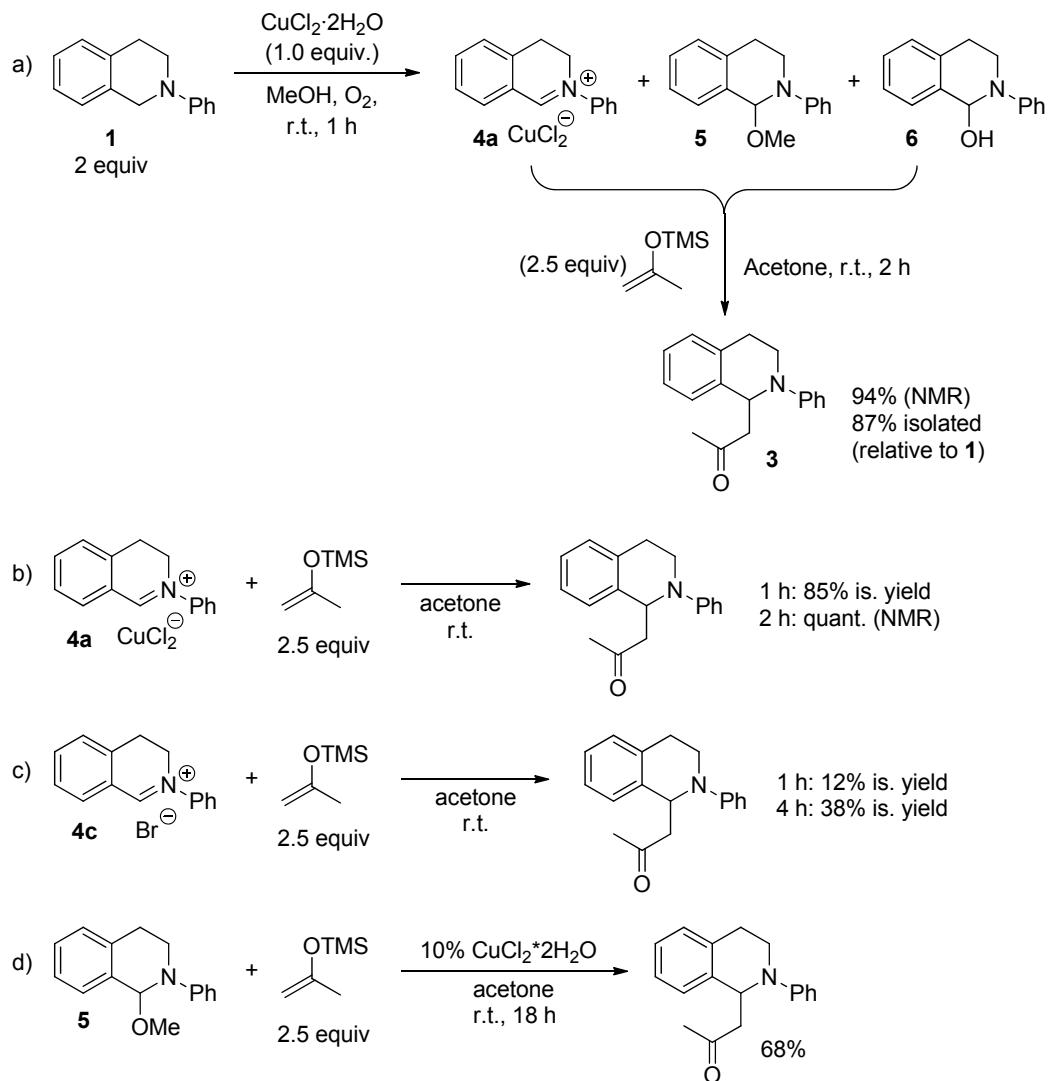
To a solution of *N*-phenyl tetrahydroisoquinoline (0.12 mmol) in methanol (0.5 mL) was added CuCl<sub>2</sub>·2H<sub>2</sub>O (2 mg, 10 mol%) at room temperature. The reaction was carried out under an oxygen atmosphere. After stirring for 60-72 hours, the reaction mixture was passed through a pad of celite and washed with dry MeOH. The solvent of the combined filtrates was removed in high vacuum, giving a crude product which is pure enough for characterization but decomposes when subjected to column chromatography on silica.  $R_f = 0.50$  (EtOAc/pentane, 2 : 8); Yield : 26 mg, 91%;  $^1\text{H}$  NMR (500 MHz, MeOH-D<sub>4</sub>):  $\delta$  7.19-7.09 (m, 6H), 6.90 (d,  $J = 10.0$  Hz, 2H), 6.69 (t,  $J = 5.0$  Hz, 1H), 5.56 (s, 1H), 3.54-3.50 (m, 1H), 3.40-3.35 (m, 1H), 3.24 (s, 3H), 2.93-2.78 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz, MeOH-D<sub>4</sub>):  $\delta$  150.25, 137.42, 135.35, 130.12, 129.50, 129.22, 128.99, 127.04, 120.06, 115.99, 89.57, 49.84, 43.97, 28.71; HR - MS m/z: calcd for C<sub>16</sub>H<sub>17</sub>NO[M<sup>+</sup>]: 239.1310; found: 239.1311.

**2-Phenyl-3,4-dihydroisoquinolinium dichlorocuprate (4a)**



To a solution of *N*-phenyl tetrahydroisoquinoline (300mg, 1.43 mmol) in methanol (1.44 mL) was added CuCl<sub>2</sub>·2H<sub>2</sub>O (122.2 mg, 0.72 mmol) at room temperature. The reaction was carried out under an oxygen atmosphere. After stirring for 1 h, the reaction mixture was passed through a pad of cotton wool and washed with a small amount of MeOH. The crude residue was crystallized from MeOH. Yield : 63 mg, 26%;  $^1\text{H}$  NMR (500 MHz, DMSO):  $\delta$  9.64 (s, 1H), 8.04-8.0 (m, 1H), 7.92-7.85 (m, 3H), 7.72-7.59 (m, 5H), 4.60 (t,  $J = 7.82$  Hz, 2H), 3.42 (t,  $J = 7.82$  Hz, 2H);  $^{13}\text{C}$  NMR (125 MHz, DMSO):  $\delta$  167.19, 142.97, 138.44, 137.10, 134.82, 130.91, 130.0, 128.35, 128.33, 125.58, 122.75, 50.67, 24.88; HR-MS m/z: calculated for C<sub>15</sub>H<sub>14</sub>N[M<sup>+</sup>]: 208.11208, found: 208.11204 (M - CuCl<sub>2</sub><sup>-</sup>).

**Reactivity studies**



**Scheme S1:** Overview of reactivity studies between amine species and silyl enolate **2**.

Iminium bromide **4c** was synthesized according to a literature procedure.<sup>2</sup>

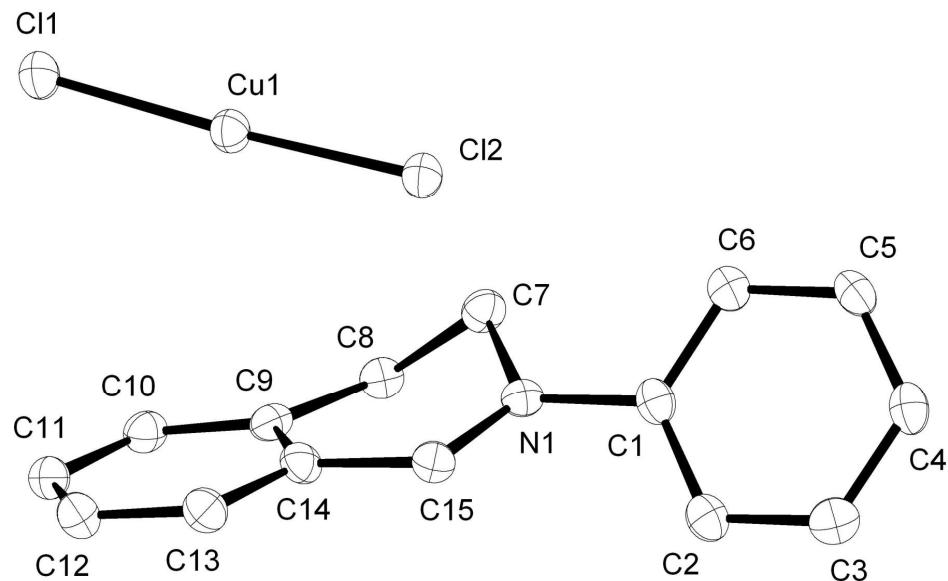
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<sup>2</sup> Bulman Page, P. C.; Buckley, B. R.; Appleby, L. F.; Alsters, P. A. *Synthesis* **2005**, 3405.

### X-ray studies

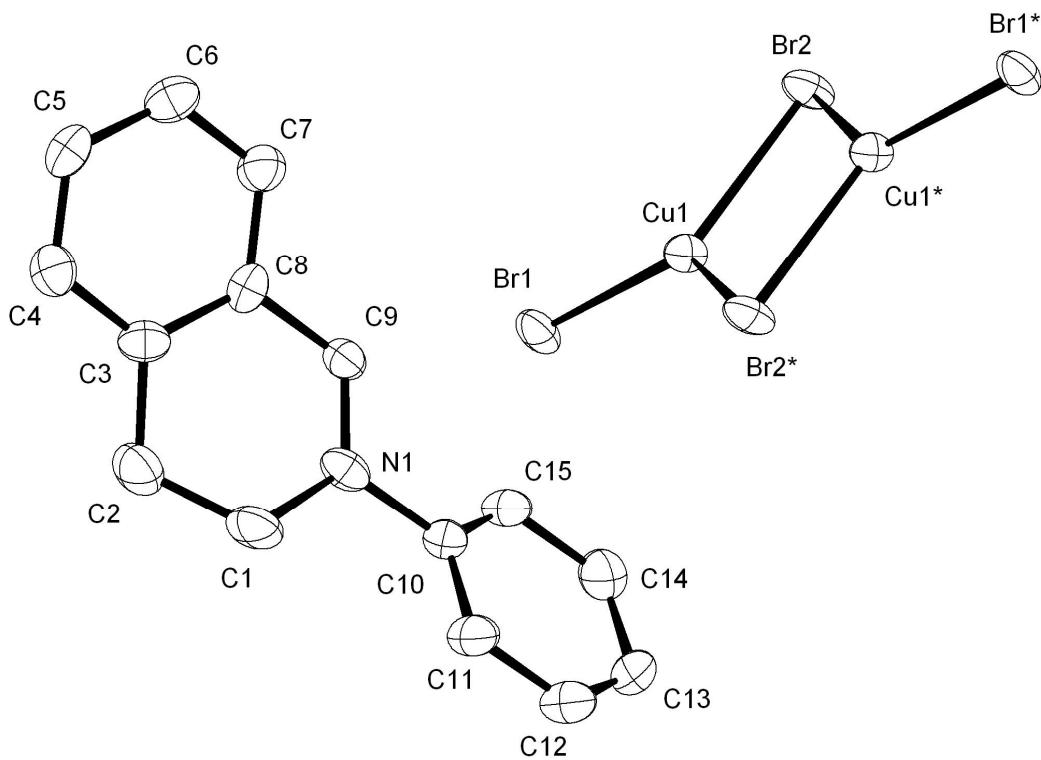
Single crystals of **4a** and **4b** were crystallised from MeOH.

#### X-ray structure of **4a**



Sum formula:  $C_{15} H_{14} Cl_2 Cu N$ . CCDC reference number: 807625

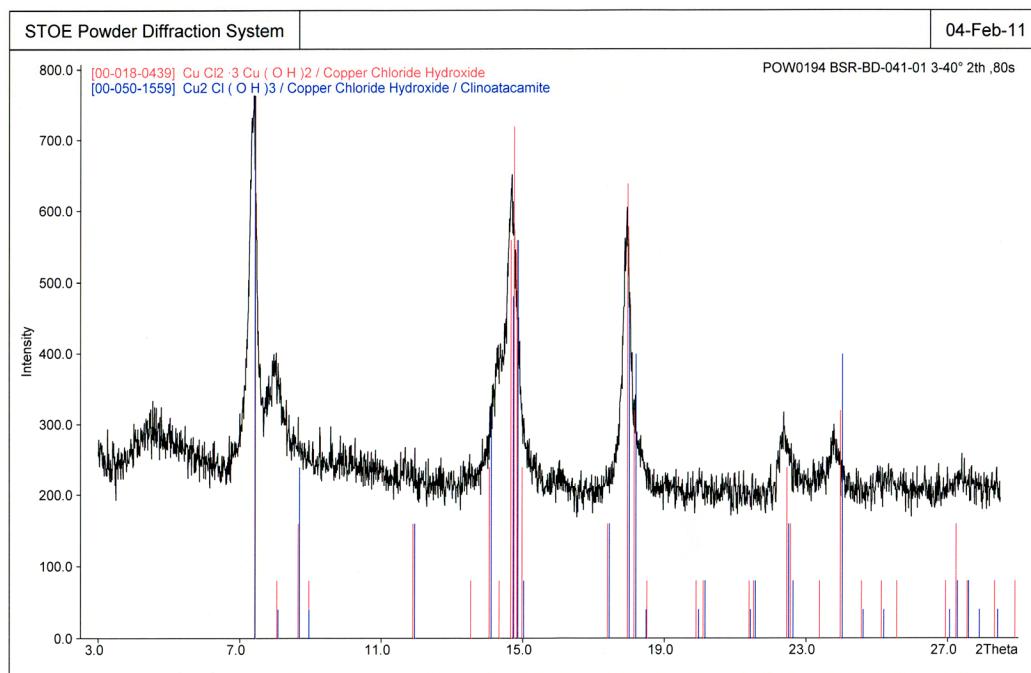
#### X-ray structure of **4b**



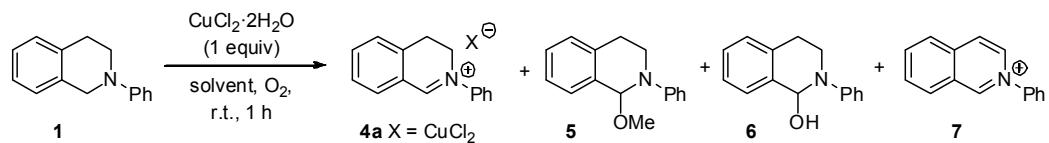
Sum formula:  $C_{15} H_{14} Br_2 Cu N$ . CCDC reference number: 813339

### Powder-X-ray of the CuCl(OH)-precipitate

Shown is the powder X-ray of the precipitate collected from the reaction mixture of Scheme 3, in comparison with two calculated X-ray patterns of  $\text{CuCl}_2 \cdot 3\text{Cu(OH)}_2$  and  $\text{Cu}_2\text{Cl}(\text{OH})_3$ .

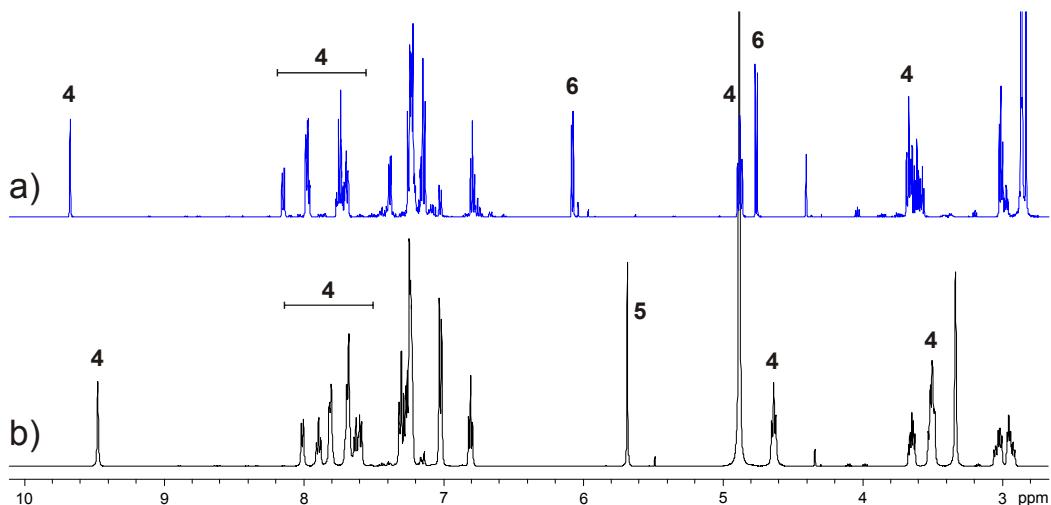


## NMR studies



### Reactions between amine 1 and CuCl<sub>2</sub> dihydrate in CD<sub>3</sub>OD and in acetone-d<sub>6</sub>, direct analysis

Typical procedure: to a solution of N-phenyl tetrahydroisoquinoline (300mg, 1.43 mmol) in CD<sub>3</sub>OD or acetone-d<sub>6</sub> (1.44 mL) was added CuCl<sub>2</sub>·2H<sub>2</sub>O (122.2 mg, 0.72 mmol) at room temperature. The reaction was carried out under an oxygen atmosphere. After stirring for 1 h, the reaction mixture was filtered over cotton and the filtrate was directly analyzed by NMR (Figure S1).



**Figure S1:** NMR analysis of the compound mixtures resulting from reactions of amine **1** with CuCl<sub>2</sub> dihydrate, indicated are characteristic peaks of compounds **4**, **5** and **6**; a) reaction performed and analyzed in acetone-d<sub>6</sub>, b) reaction performed and analyzed in CD<sub>3</sub>OD.

In MeOD, compounds **4** and **5** were formed in a ratio of ca. 40:60. In acetone-d<sub>6</sub>, compounds **4** and **6** were formed in a ratio of ca. 40:60.

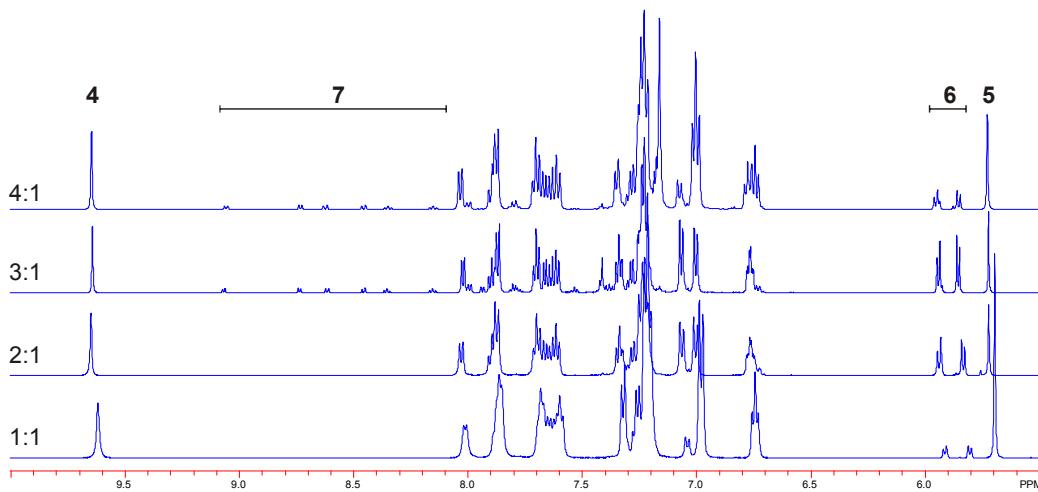
Further experiments have been performed in MeOH and analyzed in DMSO-d<sub>6</sub> which gives a better resolution, less overlapping of peaks and allowed the full characterization of compounds **4-7**.

### Compound mixtures from reactions between amine 1 and CuCl<sub>2</sub> dihydrate in different ratios, analysis in DMSO-d<sub>6</sub>

Typical procedure: to a solution of N-phenyl tetrahydroisoquinoline (300mg, 1.43 mmol) in methanol (1.44 mL) was added CuCl<sub>2</sub>·2H<sub>2</sub>O (122.2 mg, 0.72 mmol) at room temperature. The reaction was carried out under an oxygen atmosphere. After stirring for 1 h, the reaction mixture was filtered over cotton or a fritted glass funnel. The green precipitate was washed with methanol and analyzed by powder X-ray diffraction (see below). The solvent of the filtrate was removed in vacuo and the residue analyzed by NMR in DMSO-d<sub>6</sub> (Table S2, Figure S2).

**Table S2:** Ratio of products formed in the mixture as observed by  $^1\text{H}$ -NMR in DMSO-d<sub>6</sub> (Figure S3).

#	eq. amine	4	5	6	7
1	1	1.0	1.6	0.2	0.0
2	2	1.0	0.7	0.7	0.0
3	3	1.0	1.0	1.0	0.1
4	4	1.0	1.0	0.3	0.1



**Figure S2:**  $^1\text{H}$ -NMR spectra of mixtures resulting from reacting amine **1** and  $\text{CuCl}_2$  dihydrate in MeOH at various ratios (NMR's measured in DMSO-d<sub>6</sub>).

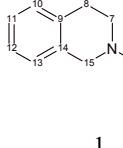
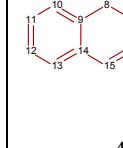
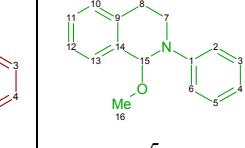
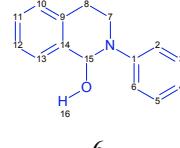
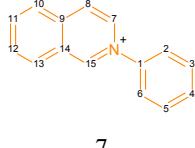
### Characterization of compounds **4-7** in DMSO, results and discussion

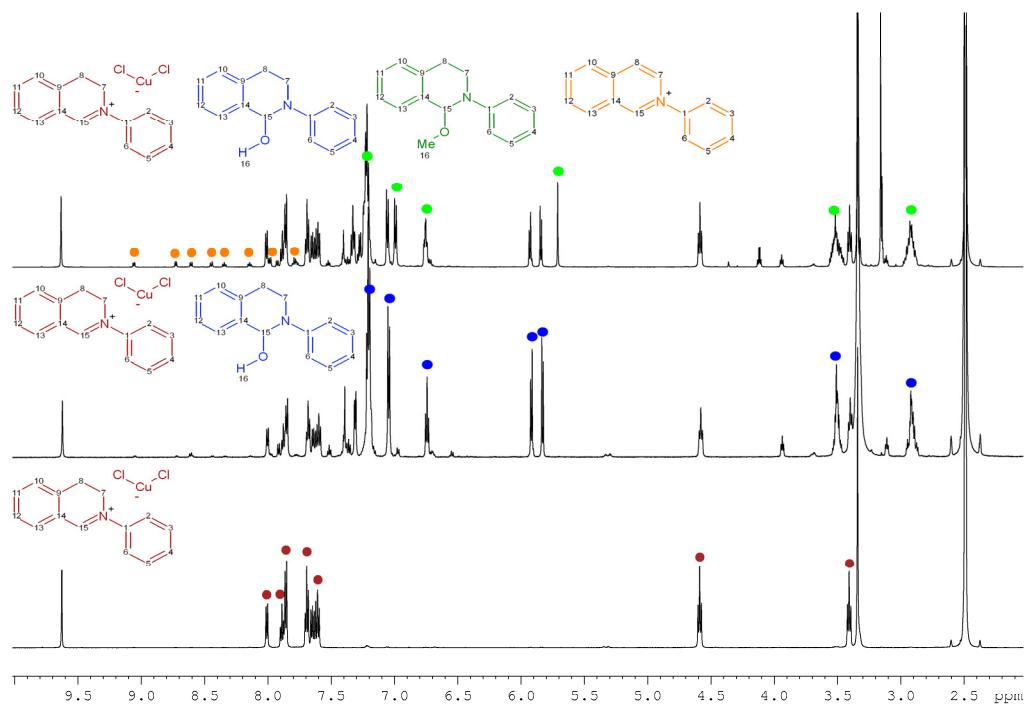
NMR spectra were recorded at 291 K on a Bruker Avance 600 (600.22 MHz) spectrometer. For the characterization of the components, 1D  $^1\text{H}$ , 1D  $^{13}\text{C}$ , 1D  $^{13}\text{C}$ -dept135,  $^1\text{H}$ , $^1\text{H}$ -COSY,  $^1\text{H}$ , $^1\text{H}$ -NOESY/EXSY,  $^1\text{H}$ , $^{13}\text{C}$ -HSQC,  $^1\text{H}$ , $^{13}\text{C}$ -HMBC and  $^1\text{H}$ , $^{15}\text{N}$ -HMBC were measured. For the NOESY/EXSY spectrum a mixing time of 700 ms was used. NMR data were processed and evaluated with Bruker's TOPSPIN 2.1.

- a) The  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{15}\text{N}$  chemical shift assignments (see Table S3) of the 4 product species (**4**, **5**, **6**, and **7**) of **Scheme 3** were completed on the reaction mixture after solvent replacement with DMSO-d<sub>6</sub>. The relative concentration of **4**, **5** and **6** close to 1:1:1 initially complicated their identification as three distinct components as shown in the  $^1\text{H}$  spectrum in Figure S3, top trace. Individual components can however be identified by comparing the  $^1\text{H}$  spectra measured on a pure iminium salt **4a** sample (Figure S3, bottom trace) and on a mixture of the salt **4** with hemiaminal **6** in DMSO-d<sub>6</sub> (Figure S3, middle trace).
- b) Trace amounts (4% relative to **4**) of the fully aromatic isoquinolinium salt (**7**) was also identified in the mixture sample and  $^1\text{H}$  and  $^{13}\text{C}$  assignment could be completed due to low field shift of a number of its protons and carbons. The assignment is also given in Table S3.
- c) No trace of amine **1** was observed in the mixture. Full assignment of **1** (Table S3) was obtained on a separate sample prepared in DMSO-d<sub>6</sub>.
- d) The NOESY/EXSY experiment conducted on the mixture revealed exchange cross-peaks between corresponding  $^1\text{H}$  from the iminium salt **4** and the hemiaminal **6** (see Figure S4). Cross-peak integration reveals that the exchange occurs at an estimated rate of approx.  $0.07 \text{ s}^{-1}$ . Exchange cross-peaks are also visible between corresponding  $^1\text{H}$  from the iminium salt **4** and the methanol adduct **5**, albeit in a much weaker regime. Cross-peak integration at position H15 reveals exchanges occurring between **4a-5** and **4a-6** at estimated rates of  $0.002 \text{ s}^{-1}$  and  $0.042 \text{ s}^{-1}$ , respectively, using a simple 3-site exchange model and first order approximations. No such

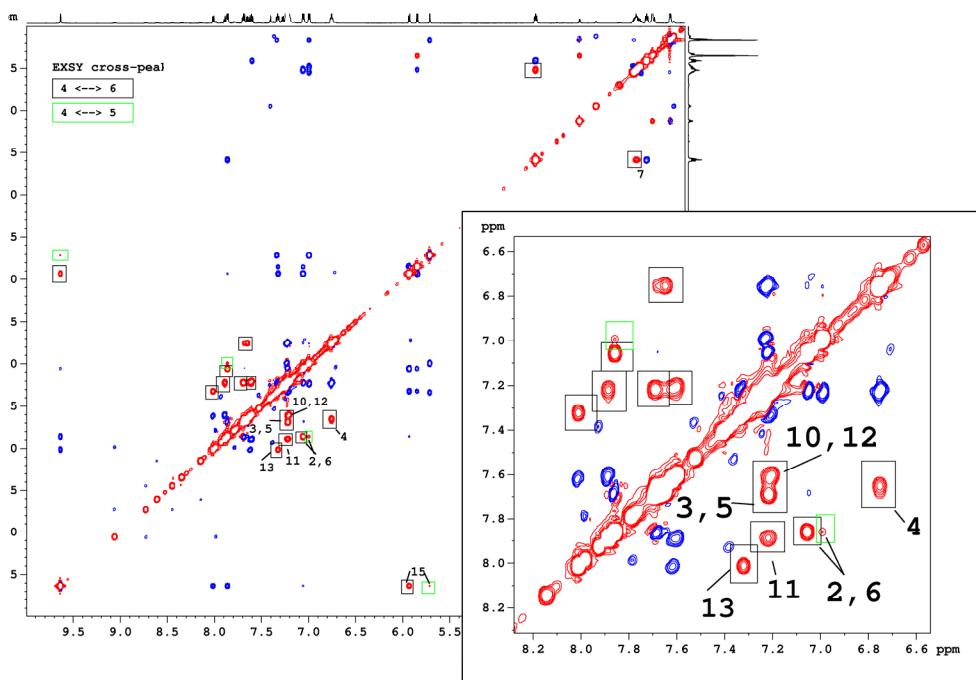
exchange cross-peaks are observed from the isoquinolinium salt (**7**) or between **5** and **6**, indicating very slow or absence of exchange.

**Table S3:**  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{15}\text{N}$  chemical shift assignments (solvent: DMSO-d<sub>6</sub>).

Atom No.										
	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$	$^{13}\text{C}$	$^1\text{H}$
1	150.11		142.93		148.46		149.02		142.99	
2	114.70	6.98	122.71	7.86	114.17	6.99	115.5	7.05	124.97	7.98
3	129.04	7.19-7.24	129.96	7.69		7.18-		7.18-	130.24	7.79
4	117.95	6.73	131.09	7.58-7.67	118.2	6.76	118.28	6.75		131.09
5	129.04	7.19-7.24	129.96	7.69		7.18-		7.18-	130.24	7.79
6	114.70	6.98	122.71	7.86	114.17	6.99	115.5	7.05	124.97	7.98
7	45.67	3.51	50.63	4.59		3.48, 3.54	40.77	3.52	134.98	9.06
8	28.09	2.88	24.84	3.41		2.86-		2.86-	125.69	8.73
9	134.61		137.05		135.7		134.82		137.23	
10	128.38	7.15	128.3*	7.58-7.67		7.18-		7.18-	127.28	8.45
11			138.39	7.89	128.18	7.28	127.43	7.26	137.68	8.34
12	125.85	7.16	128.28*	7.58-7.67		7.18-		7.18-	131.48	8.14
13	126.61	7.19-7.24	134.78	8.01	128.03	7.34	128.1	7.32	131.23	8.61
14	134.48		125.54		134.24		137.83		127.33	
15	49.78	4.36	167.14	9.63	86.72	5.71	78.68	5.93	150.57	10.39
16					53.15	3.16 (OMe)		5.84 (OH)		
	$^{15}\text{N}$		$^{15}\text{N}$		$^{15}\text{N}$		$^{15}\text{N}$		$^{15}\text{N}$	
	-319.2		-171.3		-306.0		-301.2		-184.9	



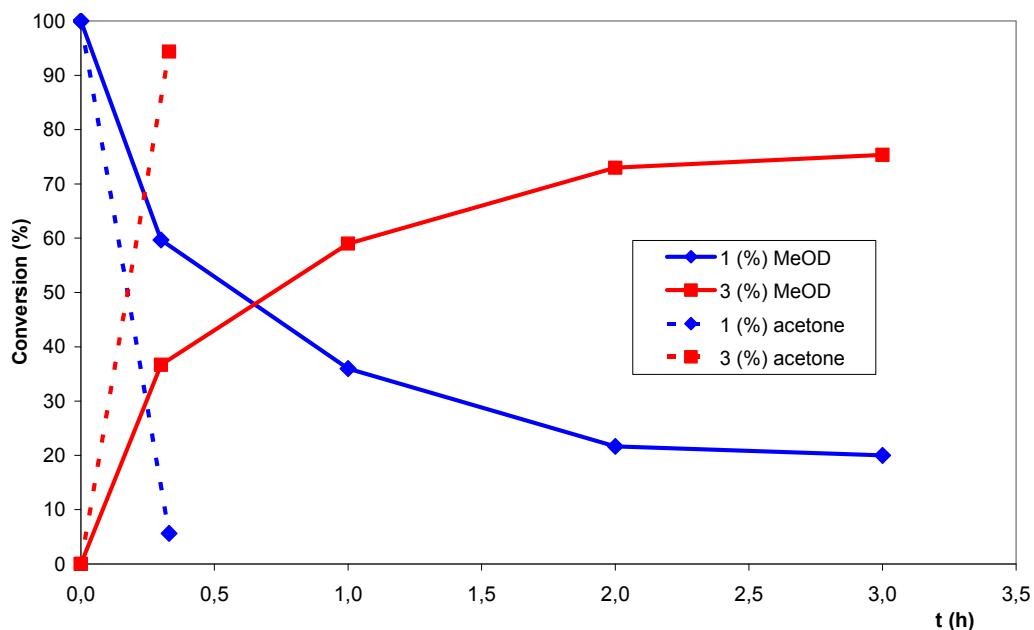
**Figure S3:**  $^1\text{H}$  spectra of 4 (bottom), mixture of 4 and 6 (middle) and mixture of 4, 5, 6 and 7 (with peak labels: red, green, blue and orange, respectively). Spectra were recorded in  $\text{DMSO-d}_6$  at 291 K on a Bruker Avance 600 (600.22 MHz) spectrometer, equipped with a TCI cryoprobe with z-gradient.



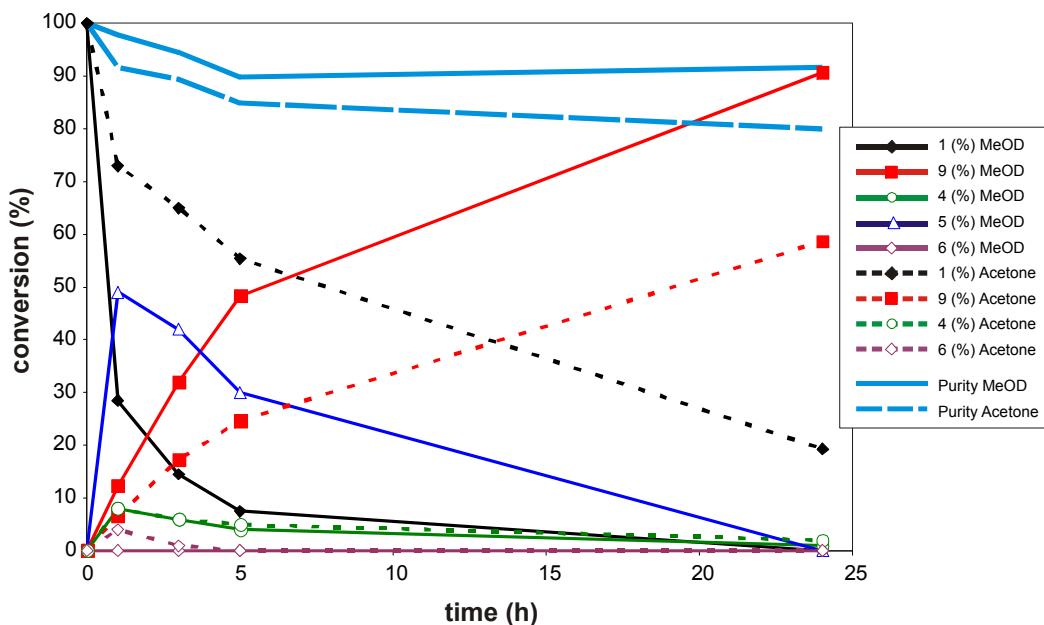
**Figure S4:** Two-dimensional  $^1\text{H}, ^1\text{H}$  NOESY/EXSY in  $\text{DMSO-d}_6$  ( $\tau_m=700\text{ms}$ ) spectrum of 4/5/6/7 mixture showing exchange cross-peaks between species 4 and 6 (black boxes) and between 4 and 5 (green boxes).

### NMR-studies of the oxidative coupling reaction

The reactions were performed according to the general procedures given for the oxidative coupling with enolat **2**<sup>3</sup> or with allyl silane **8** (see above) in methanol-d<sub>4</sub> and acetone-d<sub>6</sub>, respectively. Samples were taken at intervals and diluted with the same solvent prior to NMR-spectroscopic analysis. During the coupling with **2**, the intermediates **4**, **5** and **6** were not clearly visible by NMR.

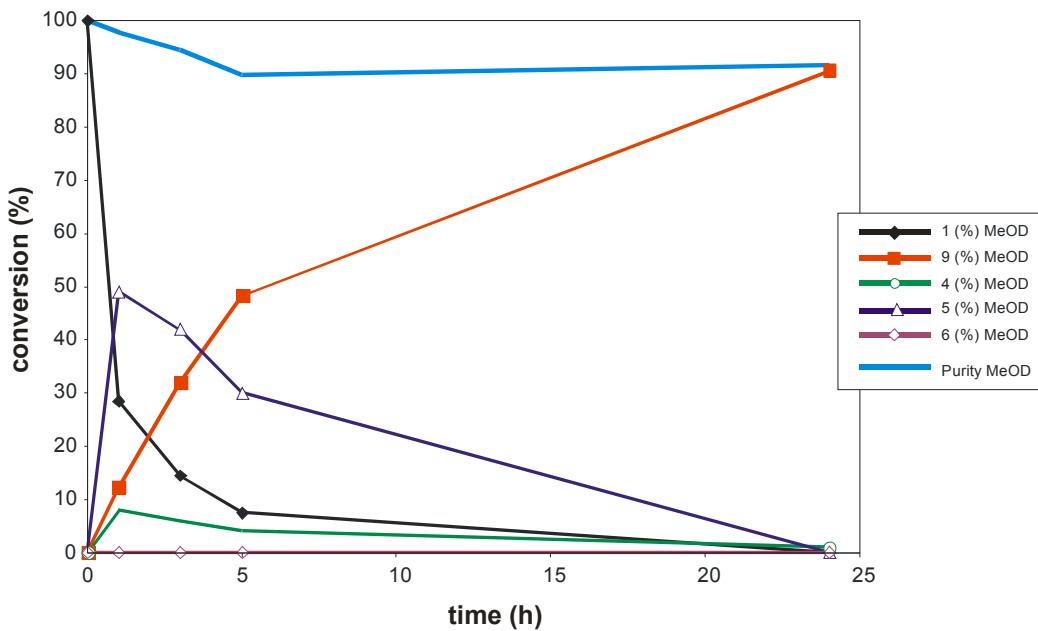


**Figure S5:** Oxidative coupling of amine **1** with enolate **2** to product **3** in methanol-d<sub>4</sub> and acetone-d<sub>6</sub>.

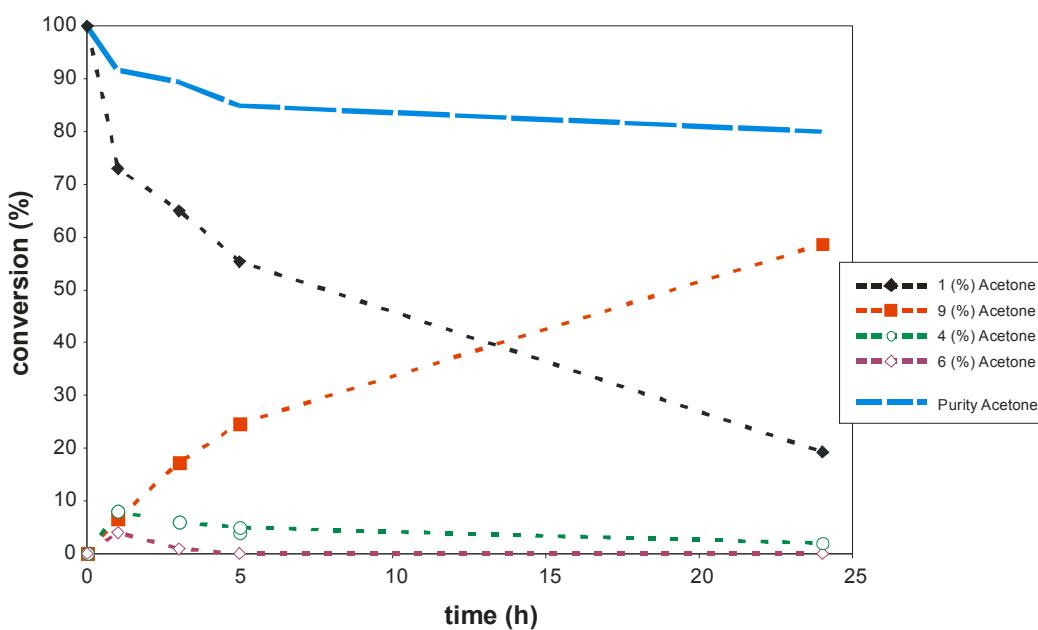


**Figure S6:** Oxidative coupling of **1** with allyl silane **8** to product **9**, comparison of solvents methanol-d<sub>4</sub> and acetone-d<sub>6</sub>. Purity: Percentage of identified species **1**, **4**, **5**, **6**, **8** and **9** in the reaction mixture.

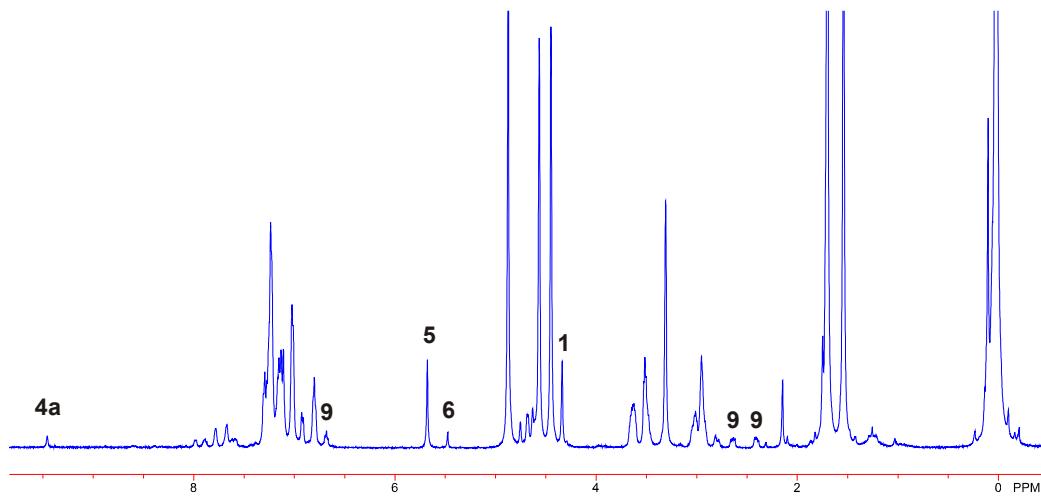
<sup>3</sup> Sureshkumar, D.; Sud, A.; Klussmann, M. *Synlett* **2009**, 1558-1561.



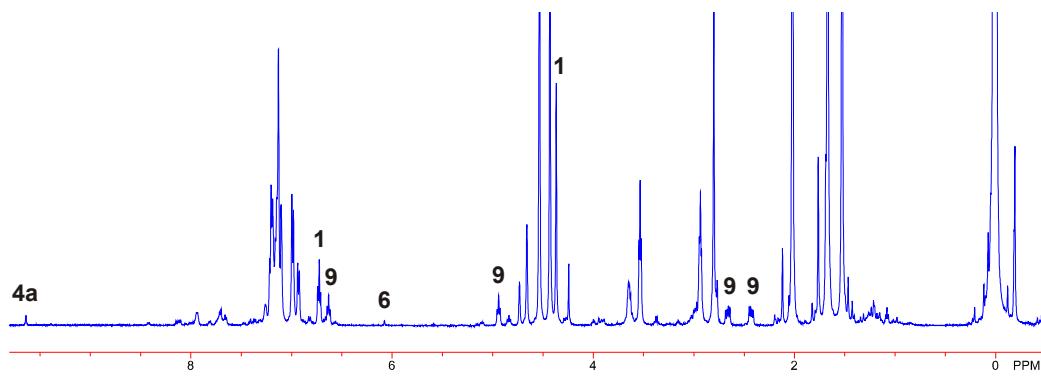
**Figure S7:** Oxidative coupling of amine **1** with allyl silane **8** to product **9** in methanol-d<sub>4</sub>. Purity: Percentage of identified species **1**, **4**, **5**, **6**, **8** and **9** in the reaction mixture.



**Figure S8:** Oxidative coupling of amine **1** with allyl silane **8** to product **9** in acetone-d<sub>6</sub>. Purity: Percentage of identified species **1**, **4**, **5**, **6**, **8** and **9** in the reaction mixture.



**Figure S9:** <sup>1</sup>H-NMR spectrum of the reaction mixture of the allylation of **1** with **8** in methanol-d<sub>4</sub>, taken after one hour; assignment of characteristic peaks of compounds **1**, **4a**, **5**, **6** and **9**.



**Figure S10:** <sup>1</sup>H-NMR spectrum of the reaction mixture of the allylation of **1** with **8** in acetone-d<sub>6</sub>, taken after five hours; assignment of characteristic peaks of compounds **1**, **4a**, **5**, **6** and **9**.

## NMR-spectra of starting materials and products

### Starting materials

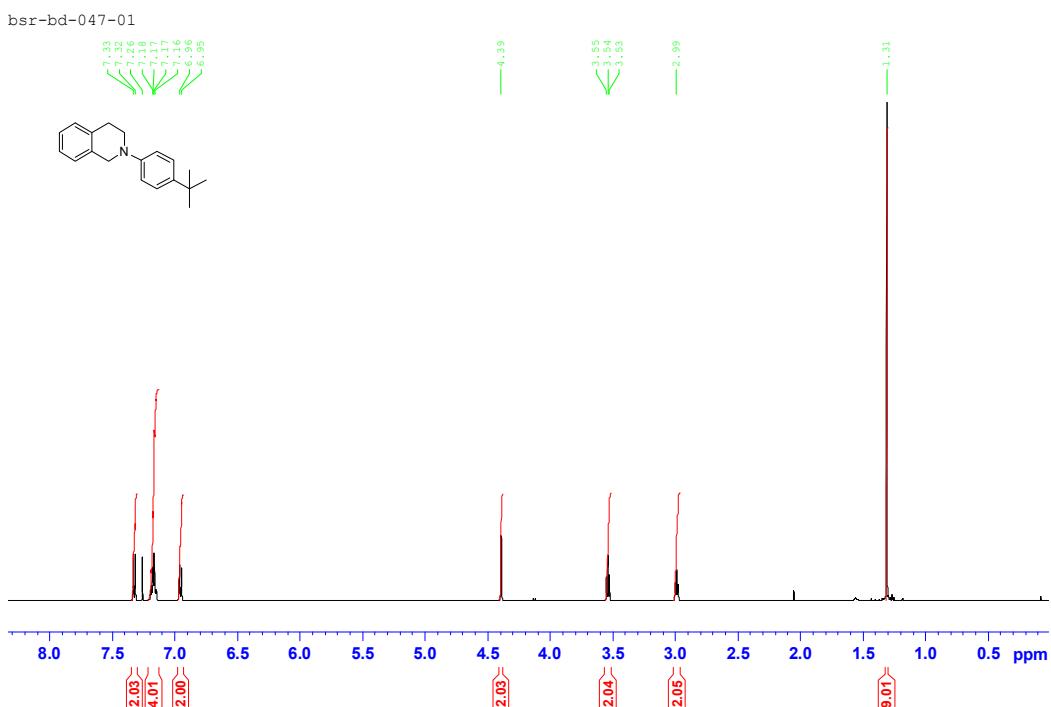


Figure S11:  $^1\text{H}$  NMR in  $\text{CDCl}_3$

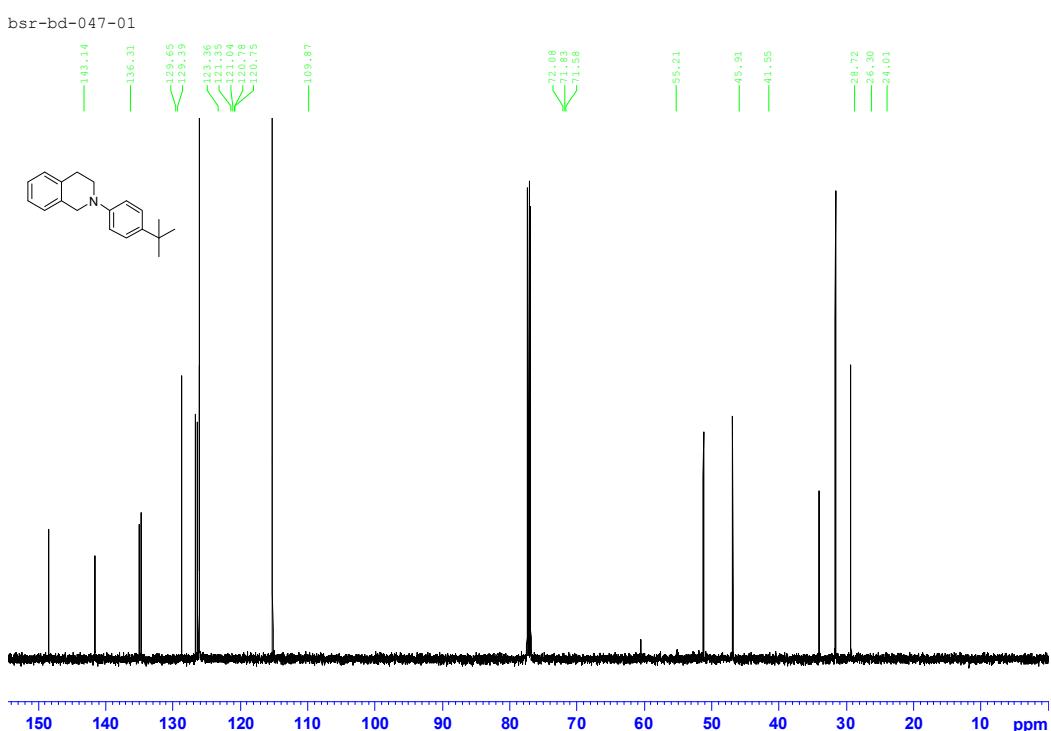
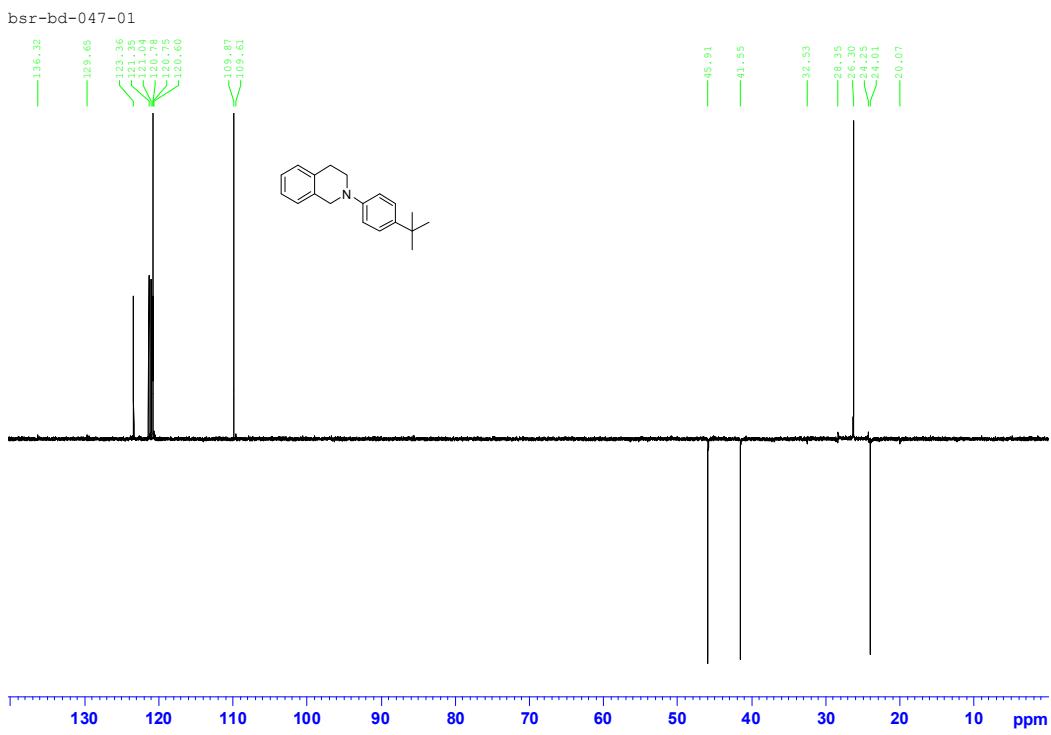
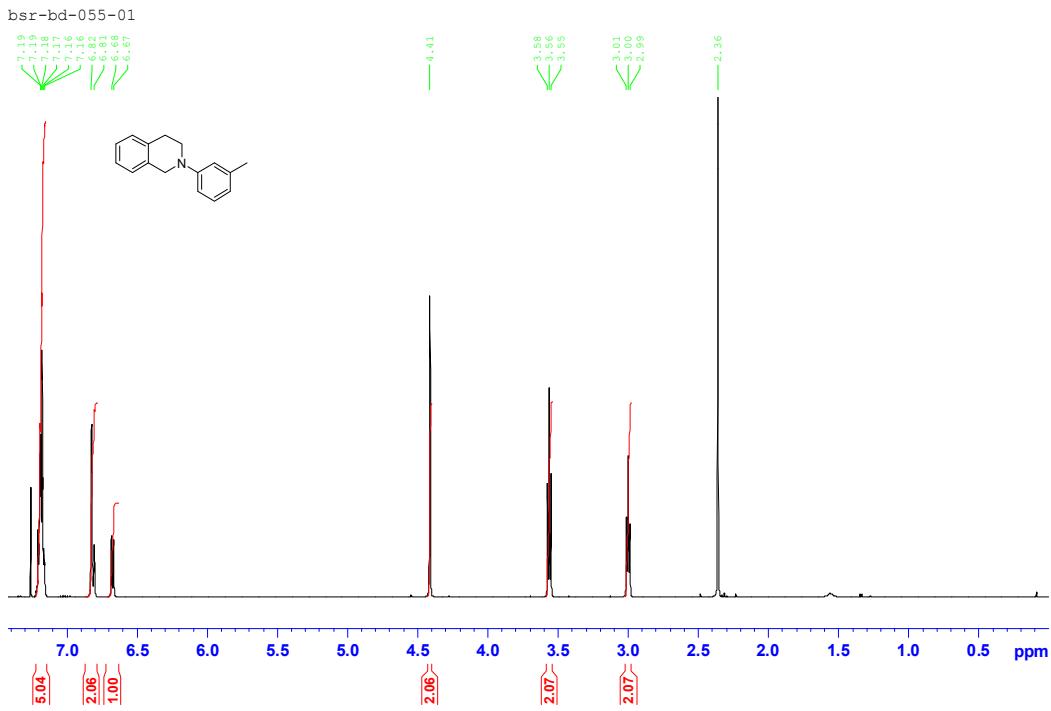


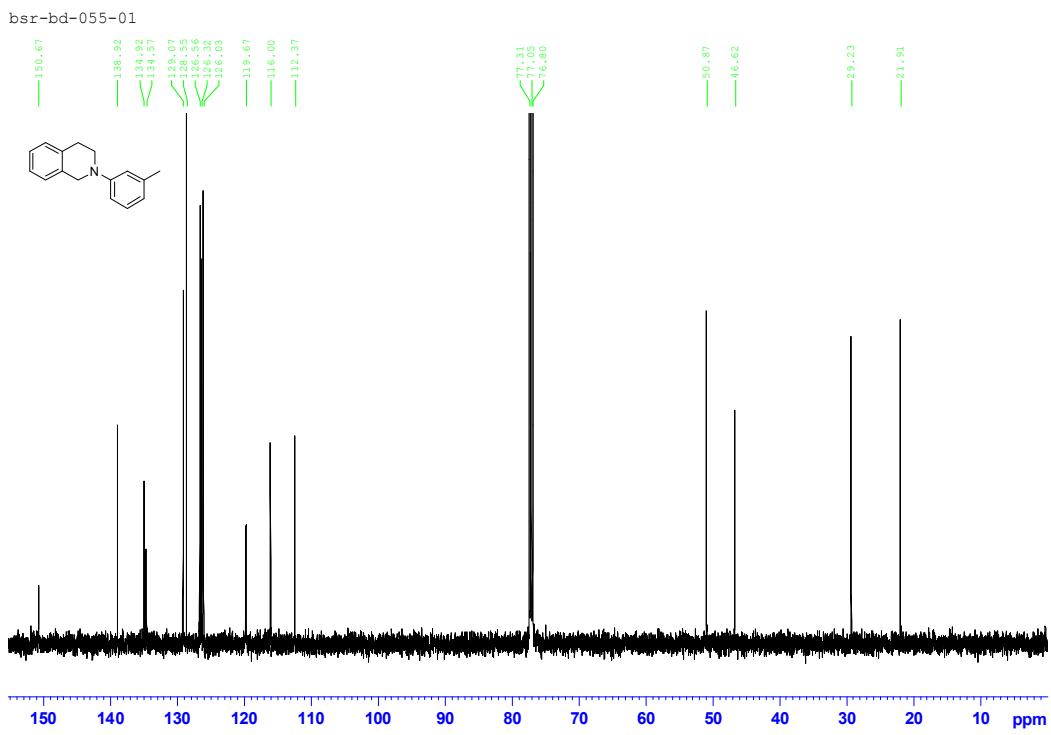
Figure S12:  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



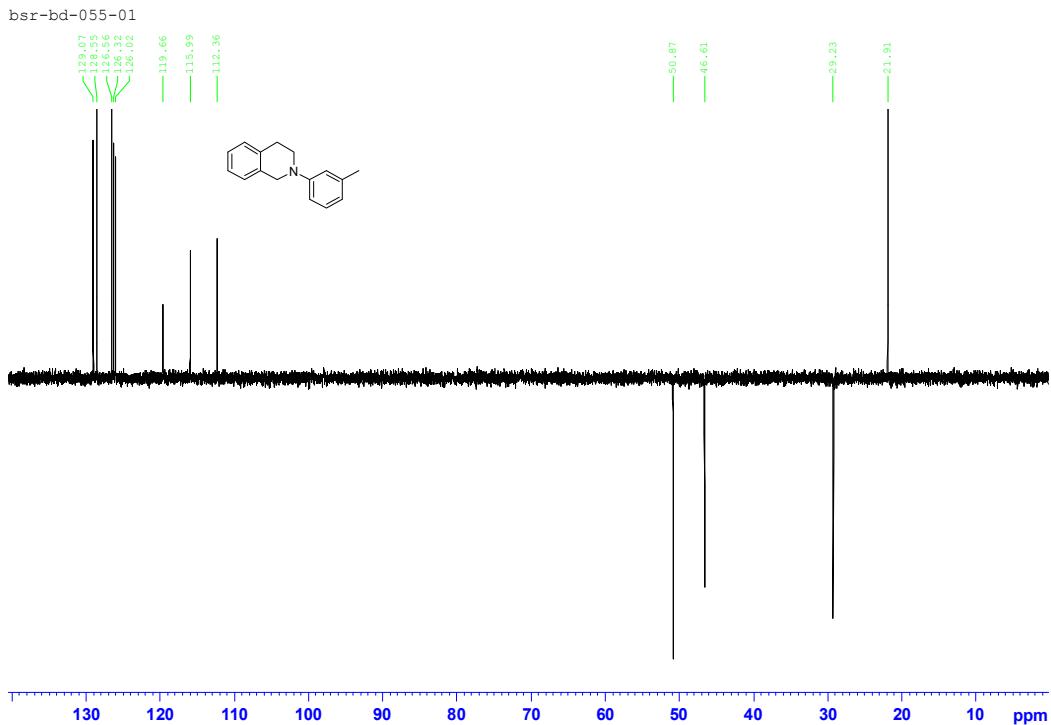
**Figure S13:** dept135 NMR in  $\text{CDCl}_3$



**Figure S14:**  $^1\text{H}$  NMR in  $\text{CDCl}_3$



**Figure S15:**  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



**Figure S16:** dept135 NMR in  $\text{CDCl}_3$

## Products

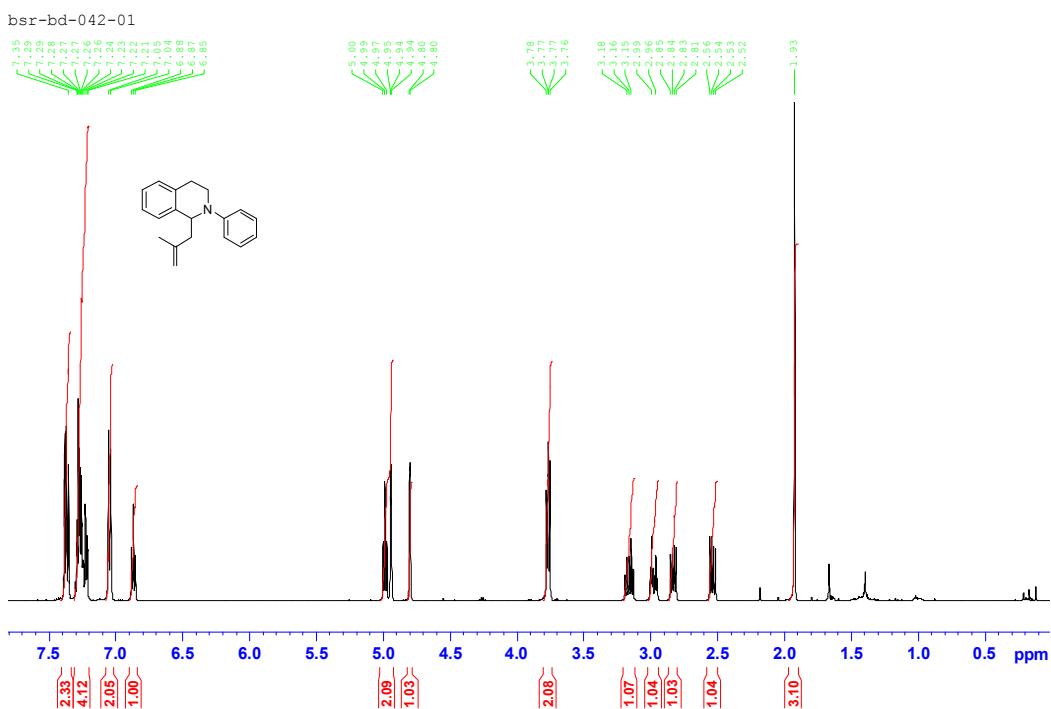


Figure S17:  $^1\text{H}$  NMR in  $\text{CDCl}_3$

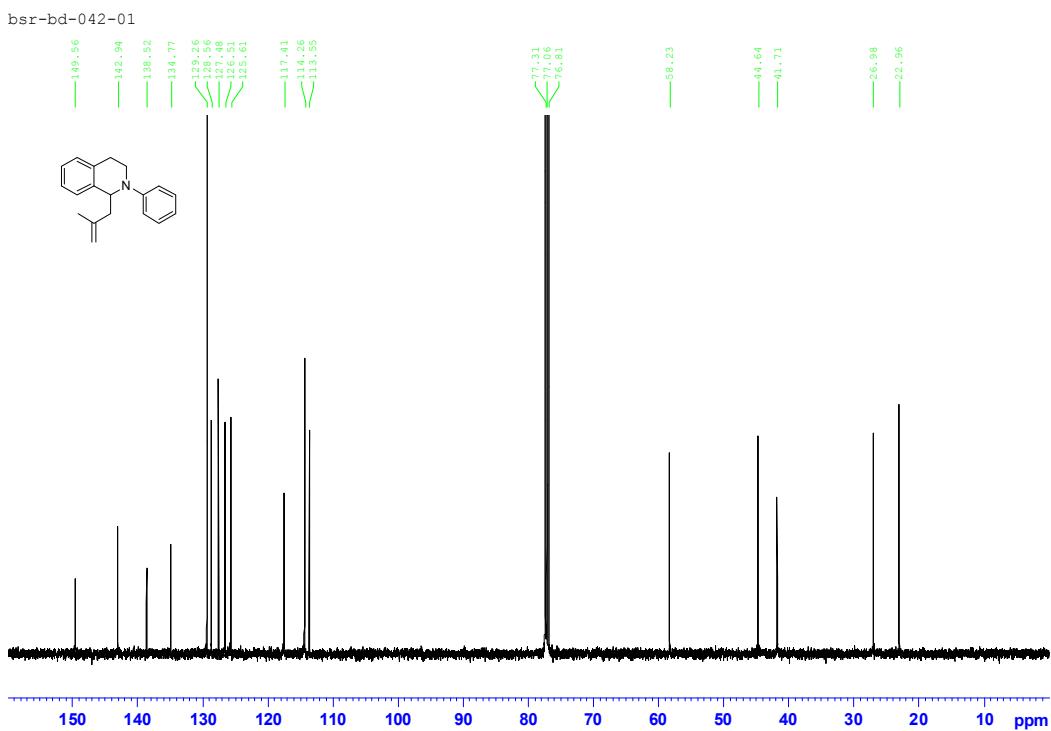
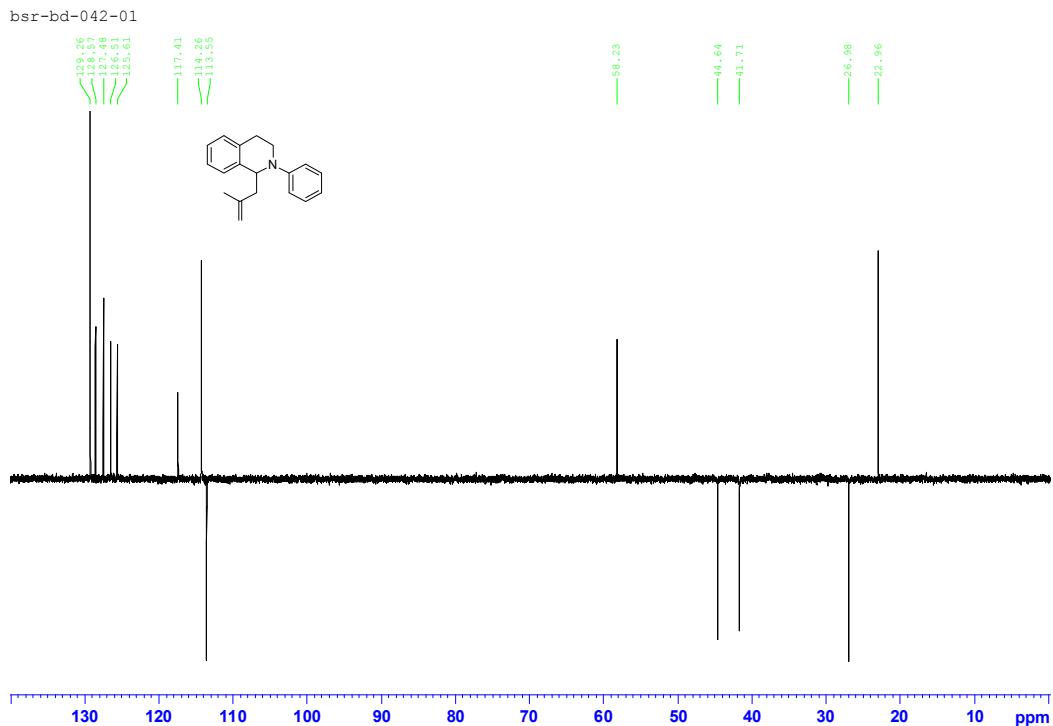
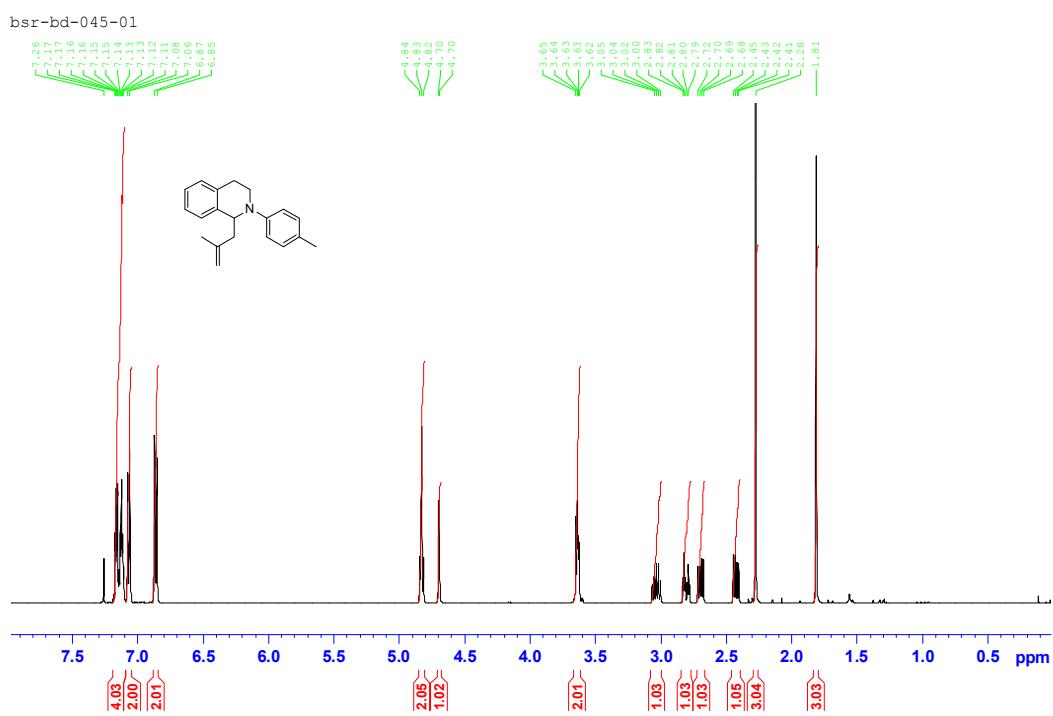


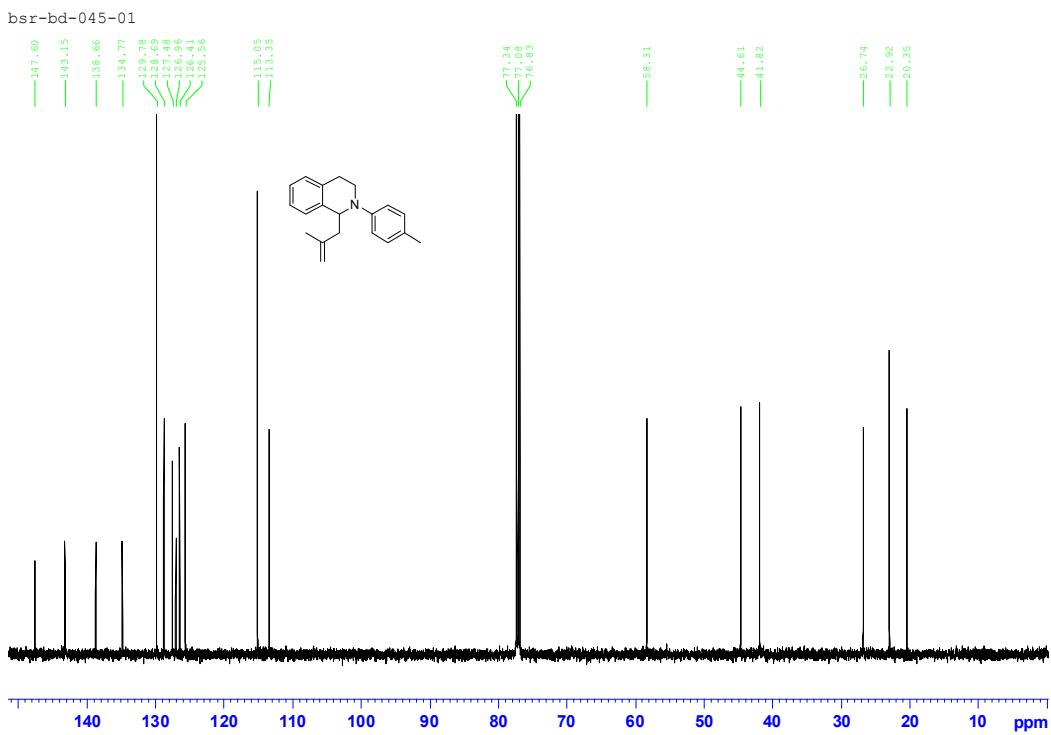
Figure S18:  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



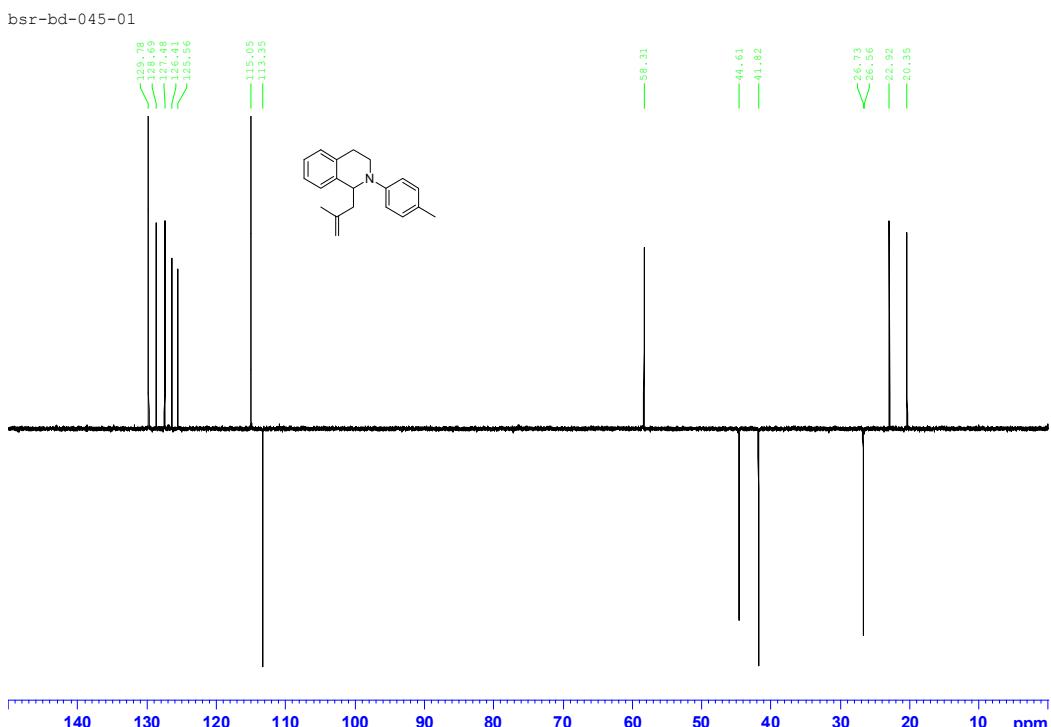
**Figure S19:** dept135 NMR in  $\text{CDCl}_3$



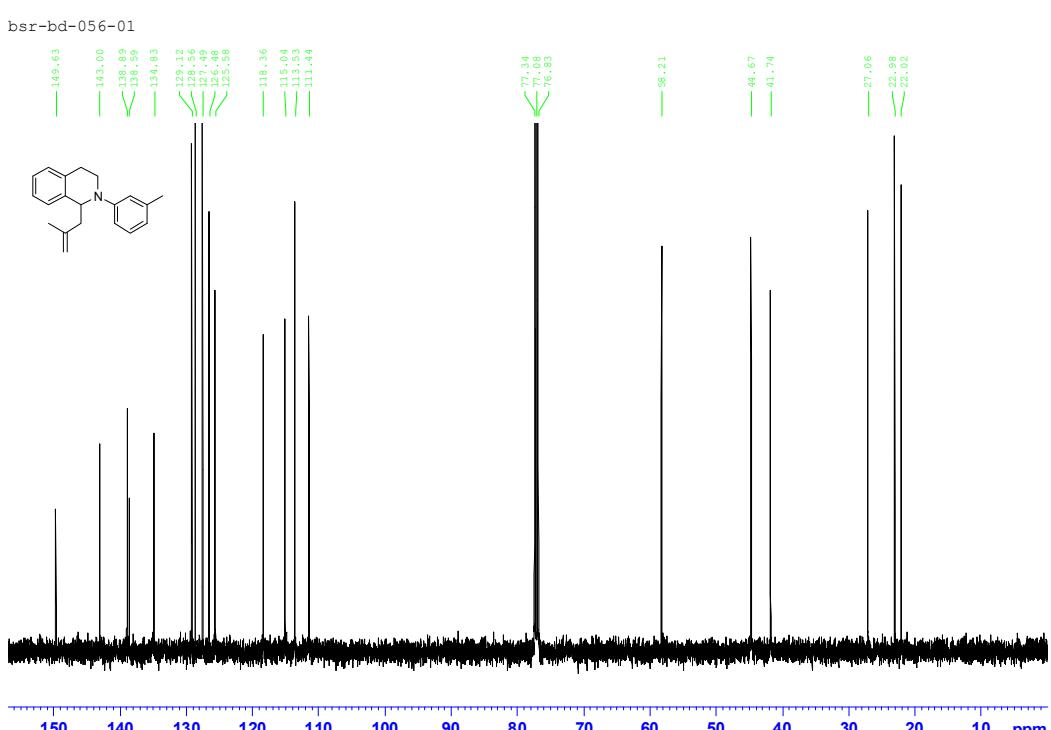
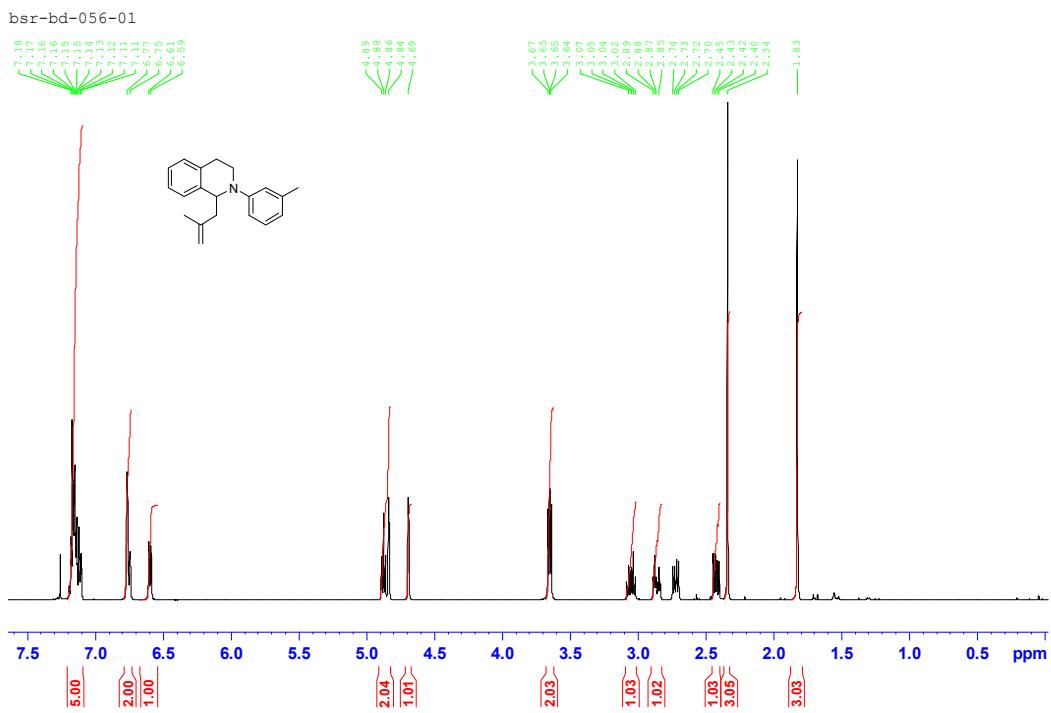
**Figure S20:**  $^1\text{H}$  NMR in  $\text{CDCl}_3$

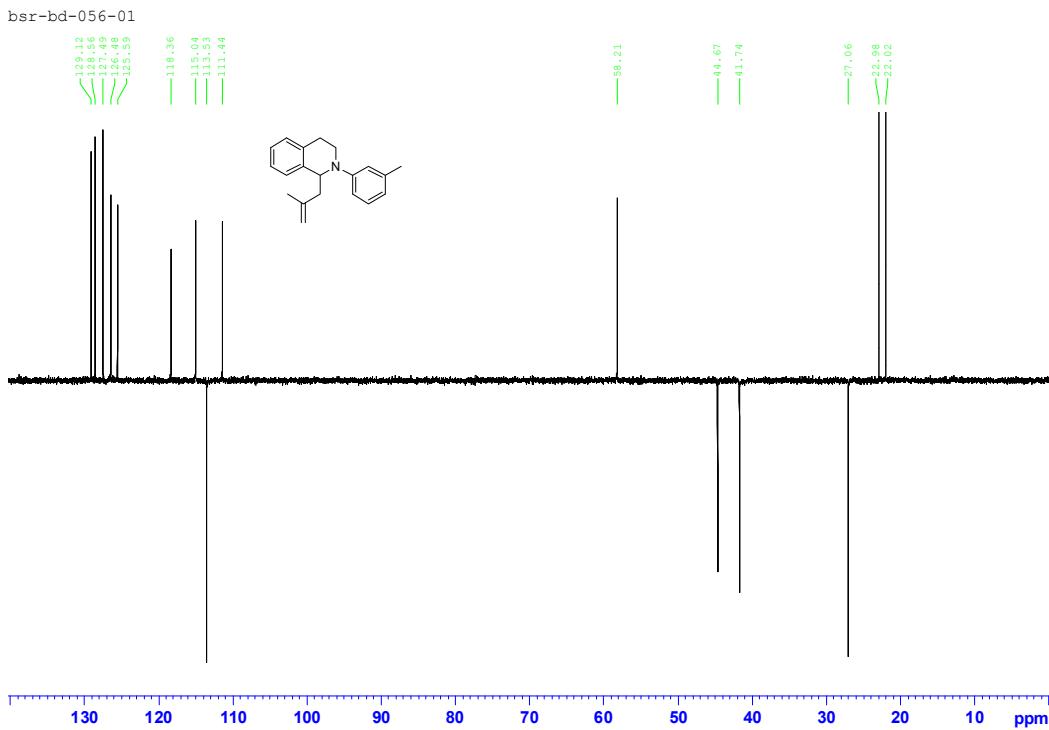


**Figure S21:**  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$

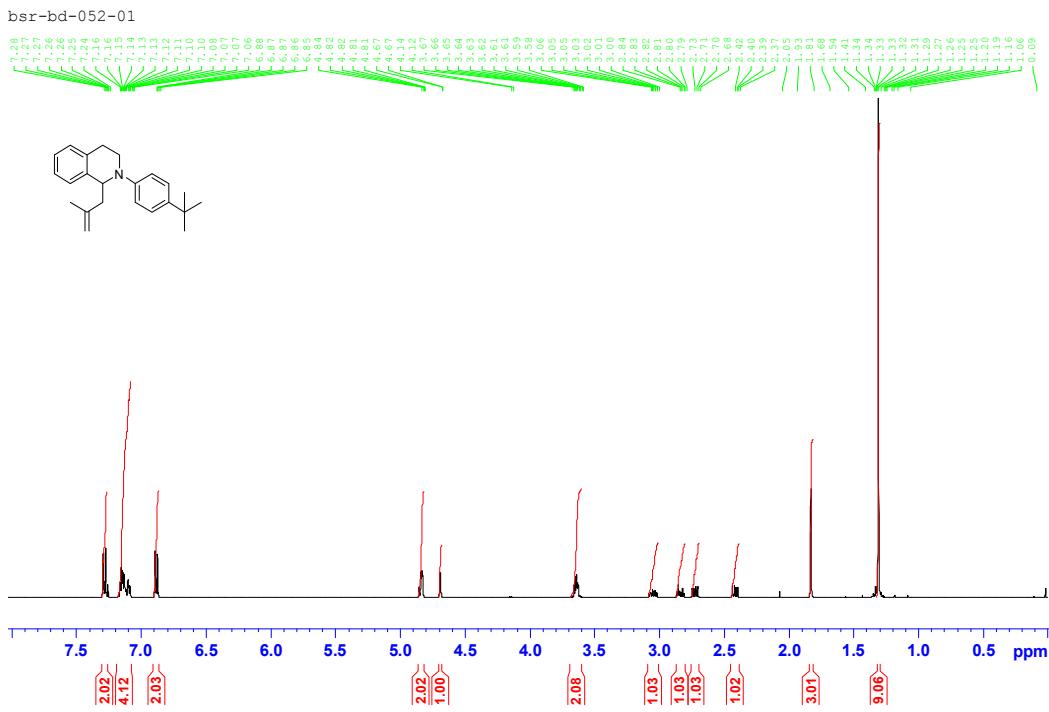


**Figure S22:** dept135 NMR in  $\text{CDCl}_3$

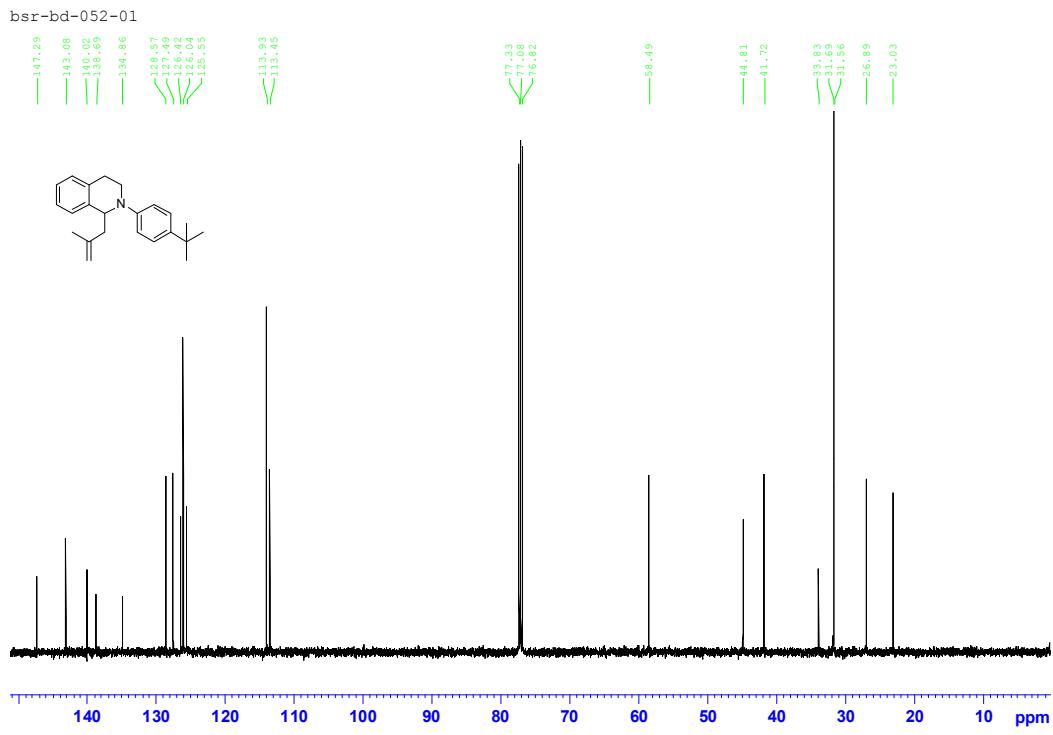




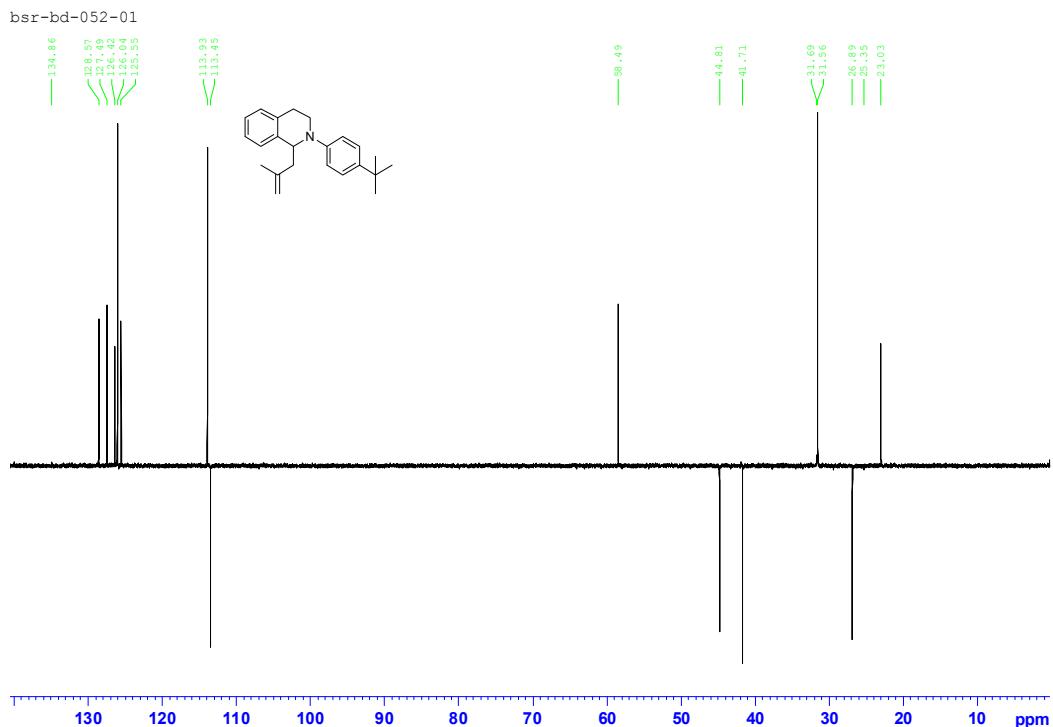
**Figure S25:** dept135 NMR in  $\text{CDCl}_3$



**Figure S26:**  $^1\text{H}$  NMR in  $\text{CDCl}_3$



**Figure S27:**  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



**Figure S28:** dept135 NMR in  $\text{CDCl}_3$

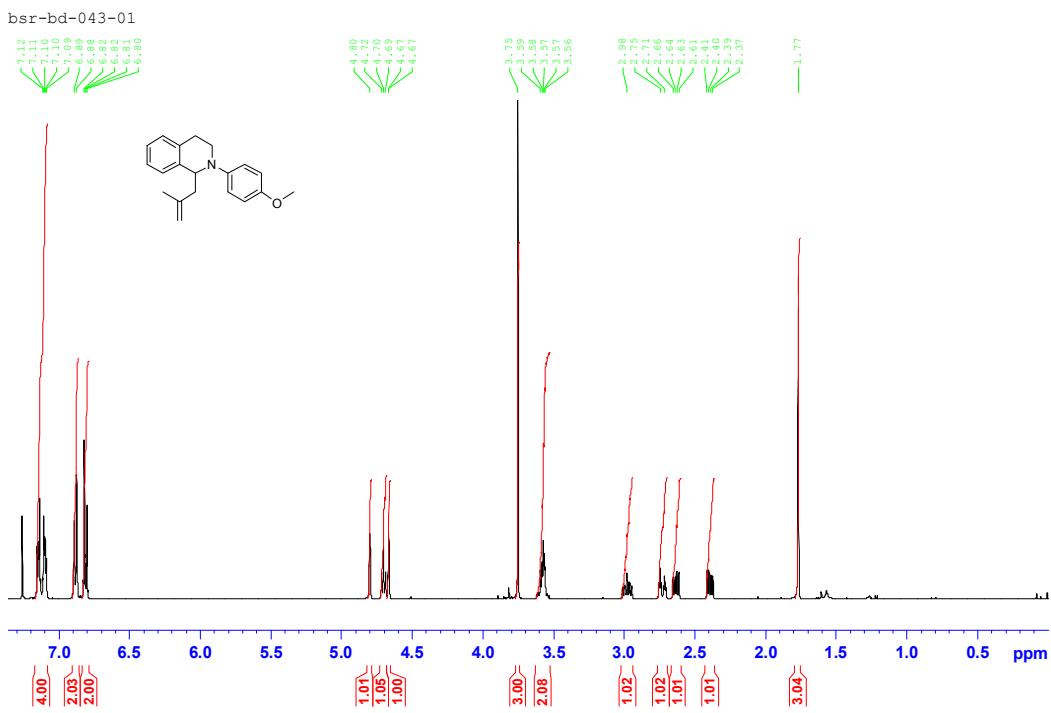


Figure S29: <sup>1</sup>H NMR in CDCl<sub>3</sub>

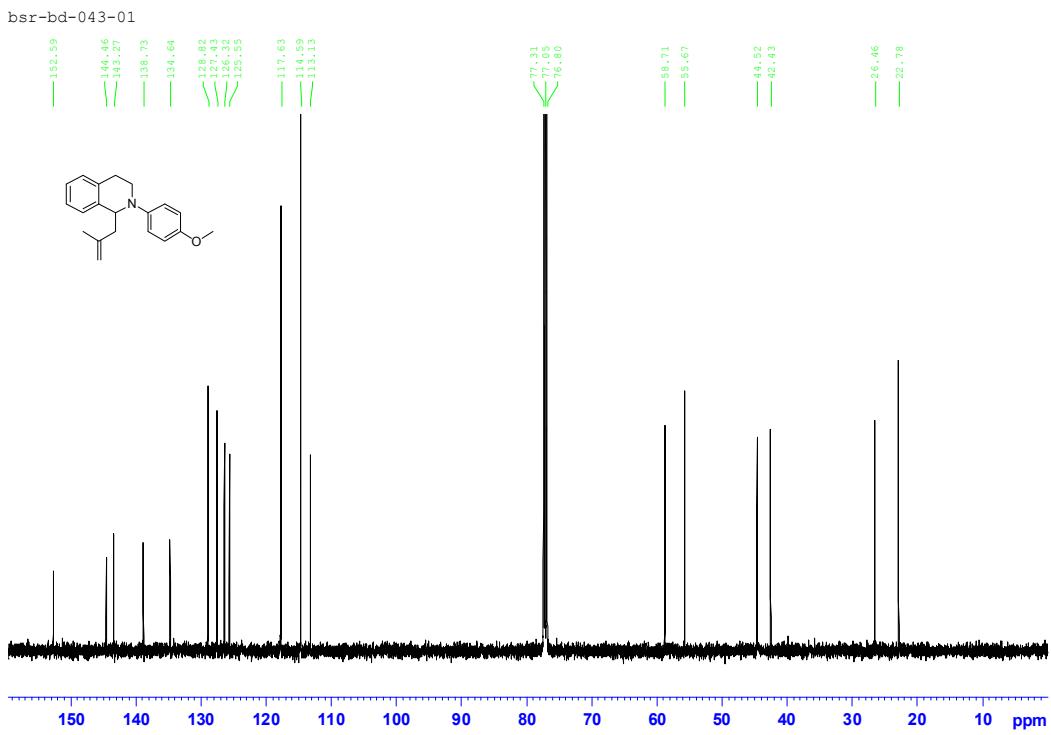
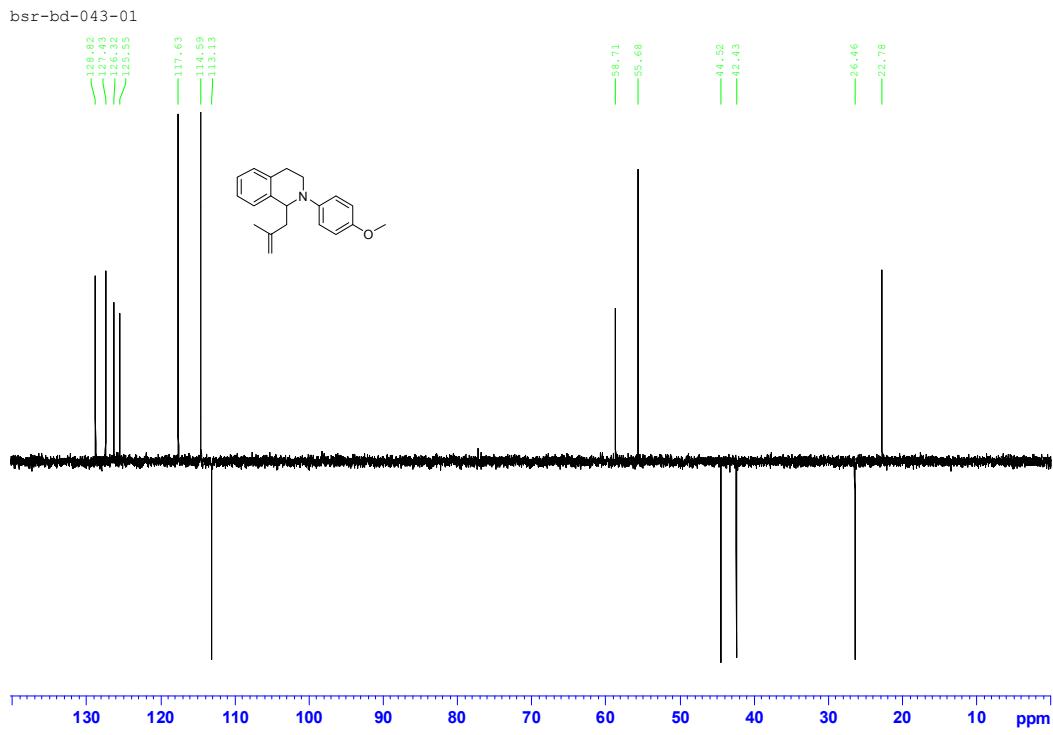
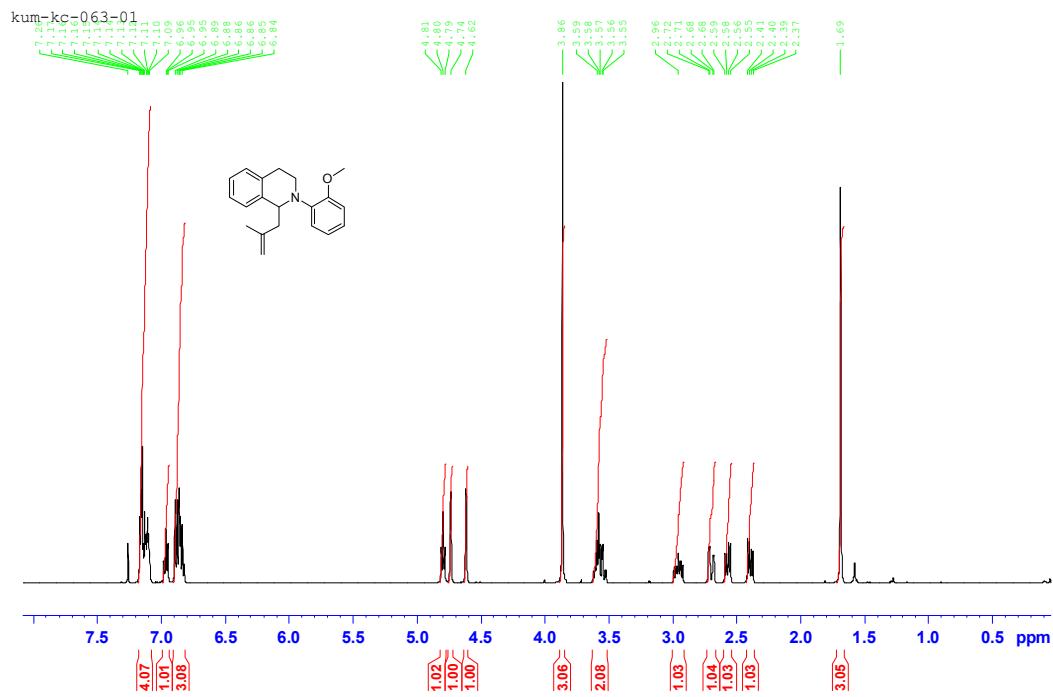


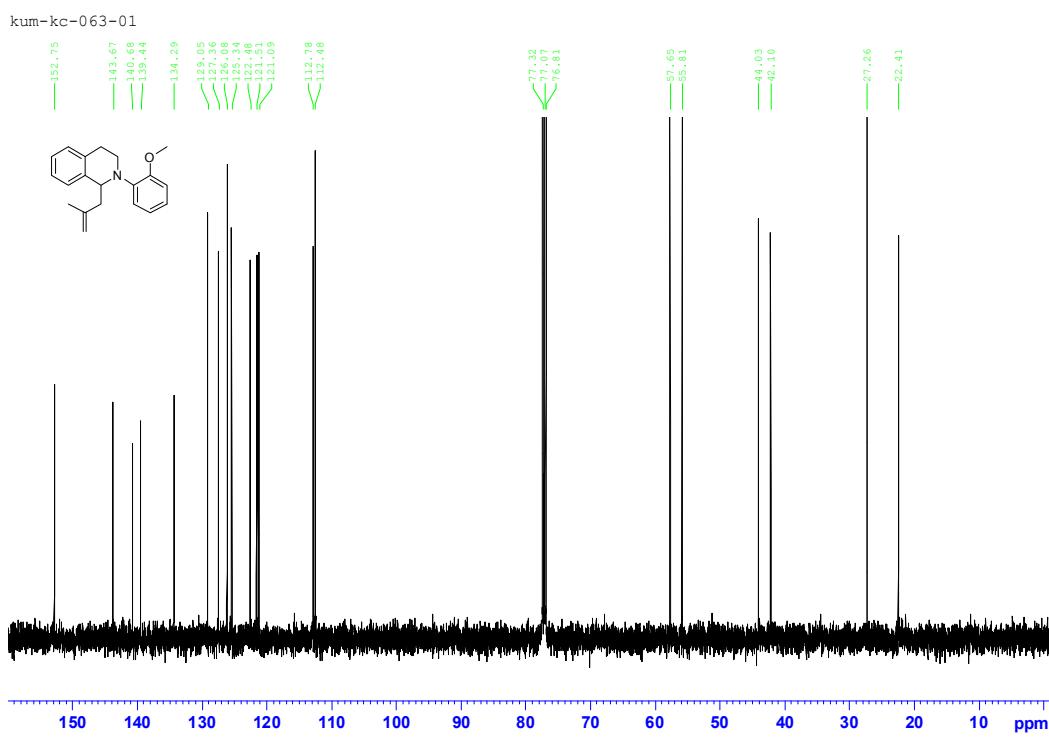
Figure S30: <sup>13</sup>C NMR in CDCl<sub>3</sub>



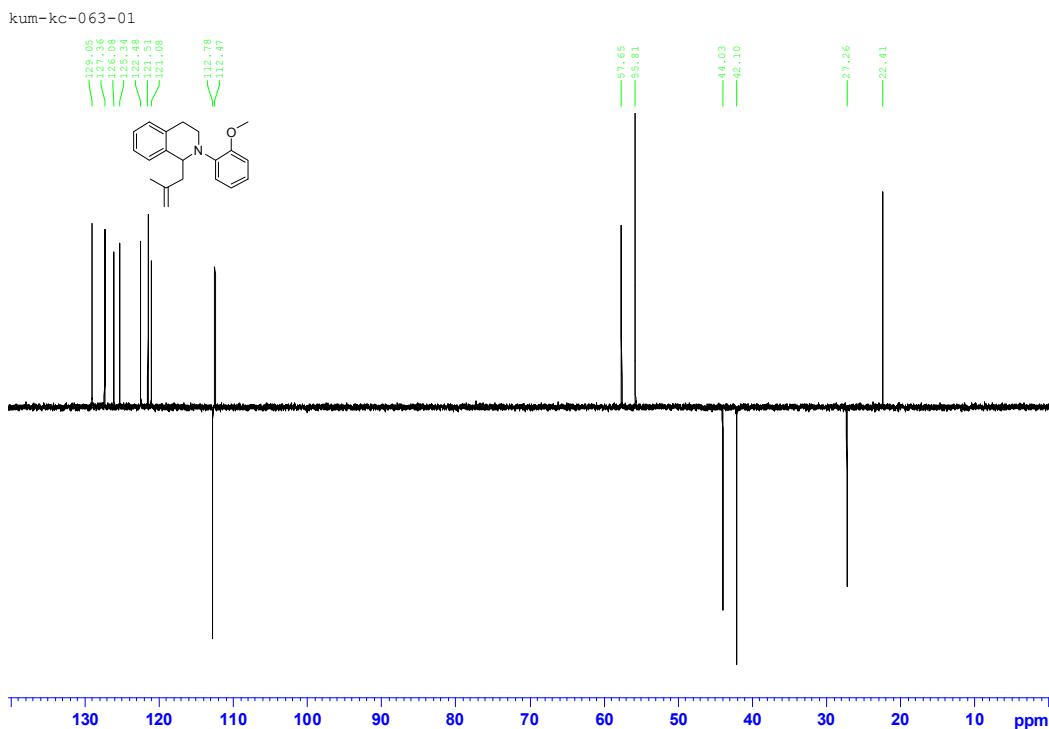
**Figure S31:** dept135 NMR in  $\text{CDCl}_3$



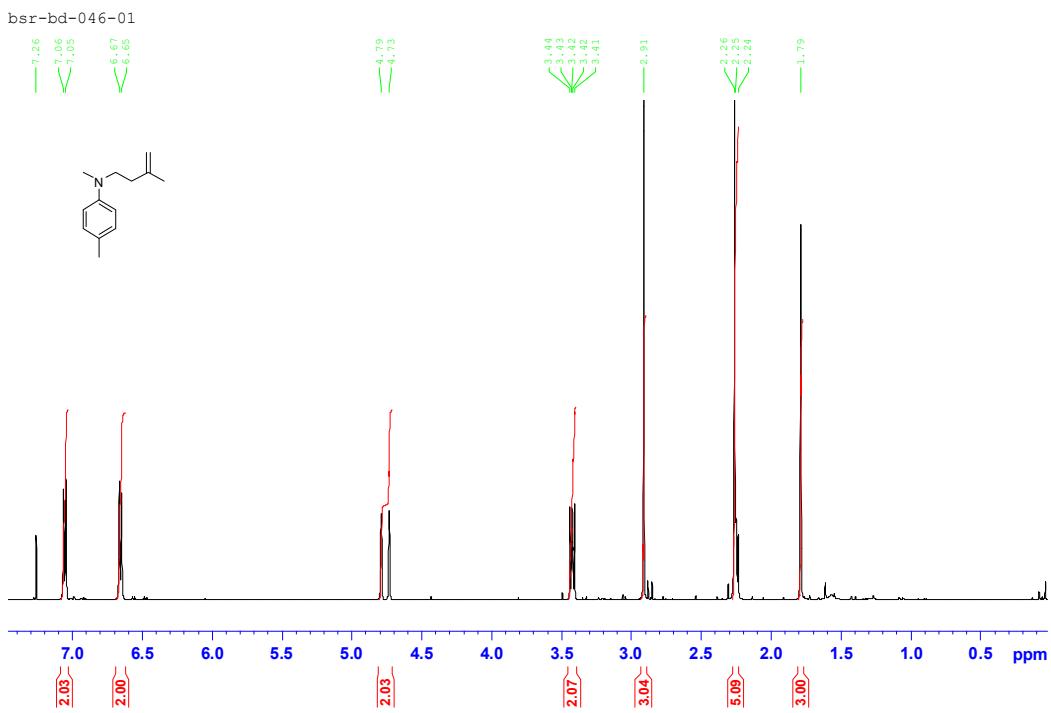
**Figure S32:**  $^1\text{H}$  NMR in  $\text{CDCl}_3$



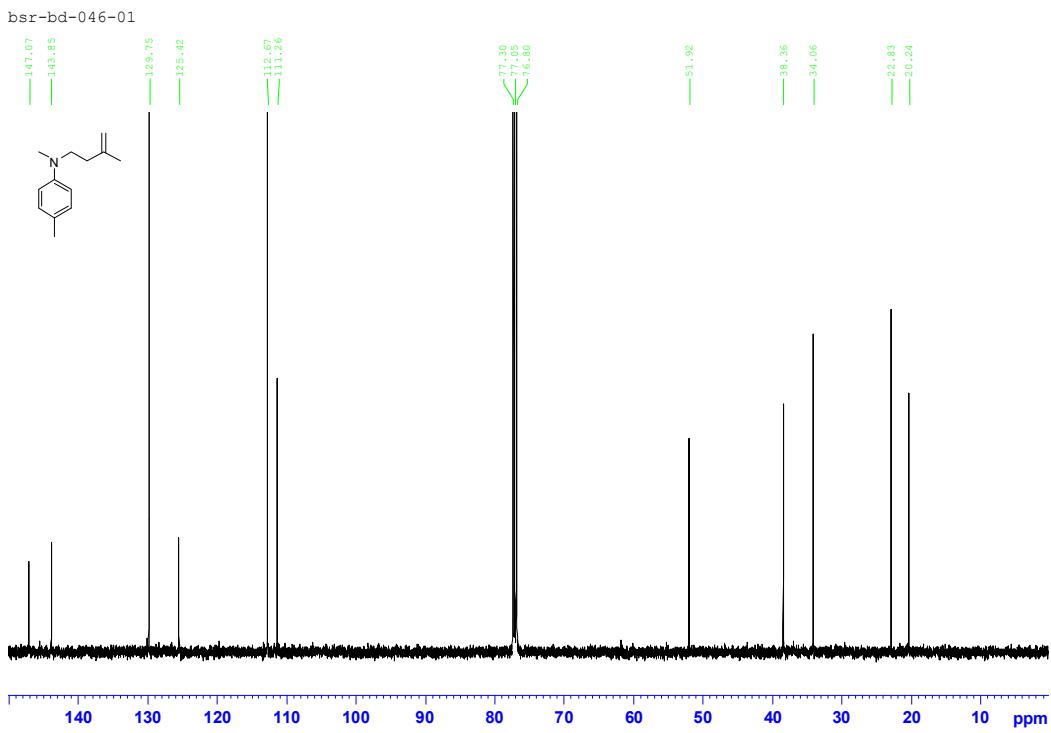
**Figure S33:**  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



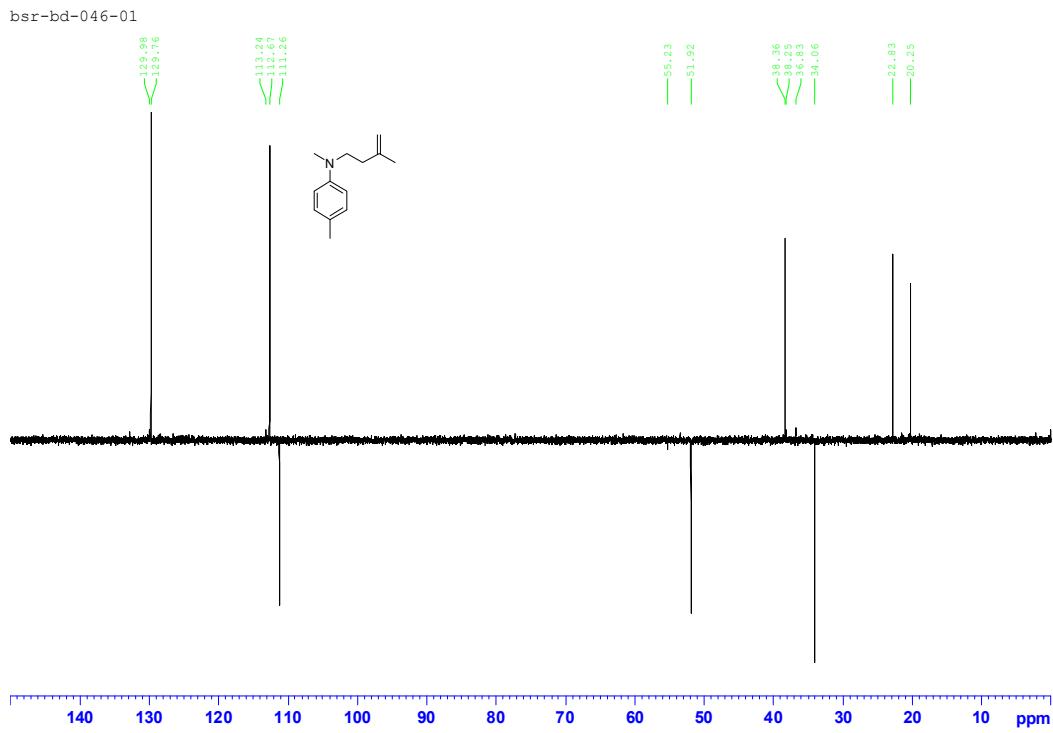
**Figure S34:** dept135 NMR in  $\text{CDCl}_3$



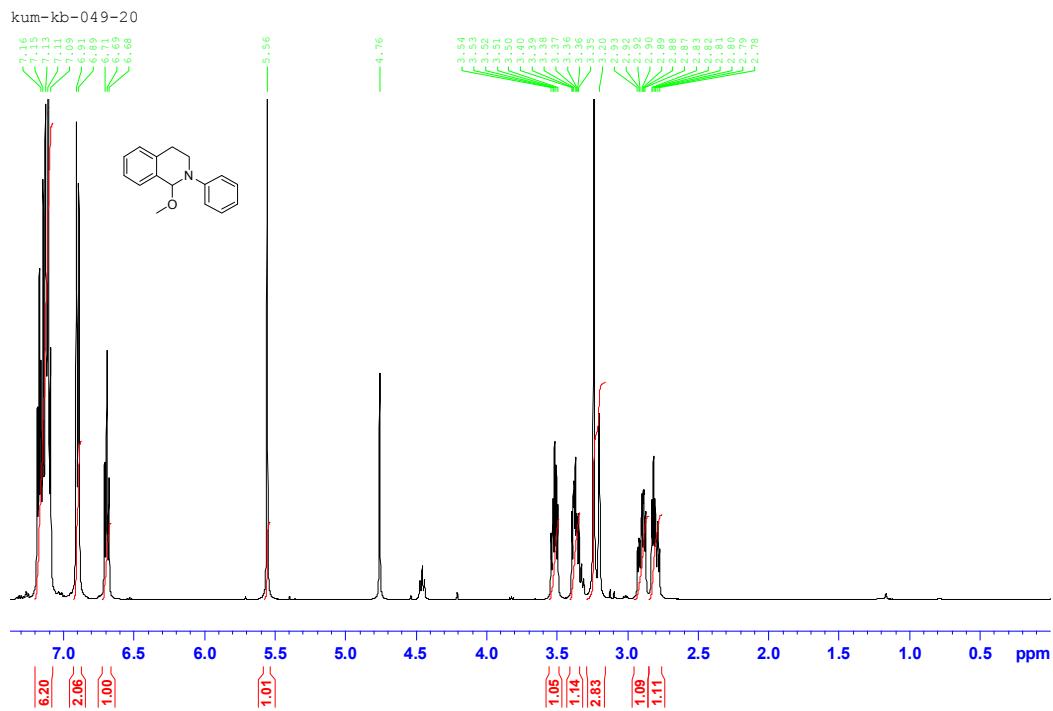
**Figure S35:**  $^1\text{H}$  NMR in  $\text{CDCl}_3$



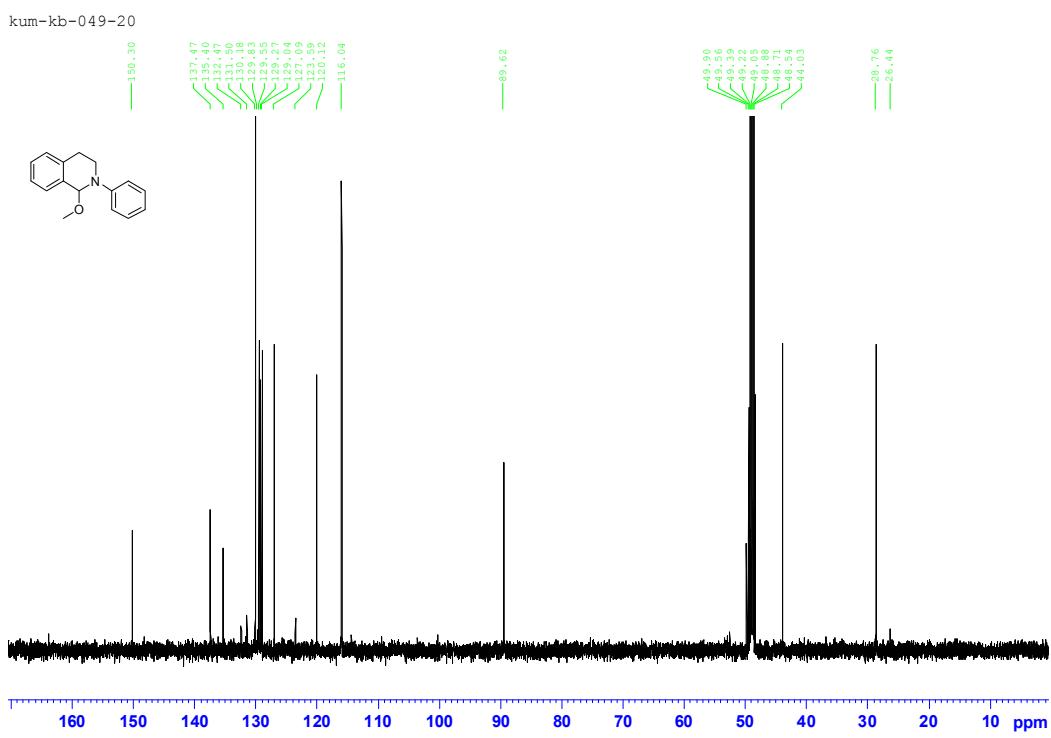
**Figure S36:**  $^{13}\text{C}$  NMR in  $\text{CDCl}_3$



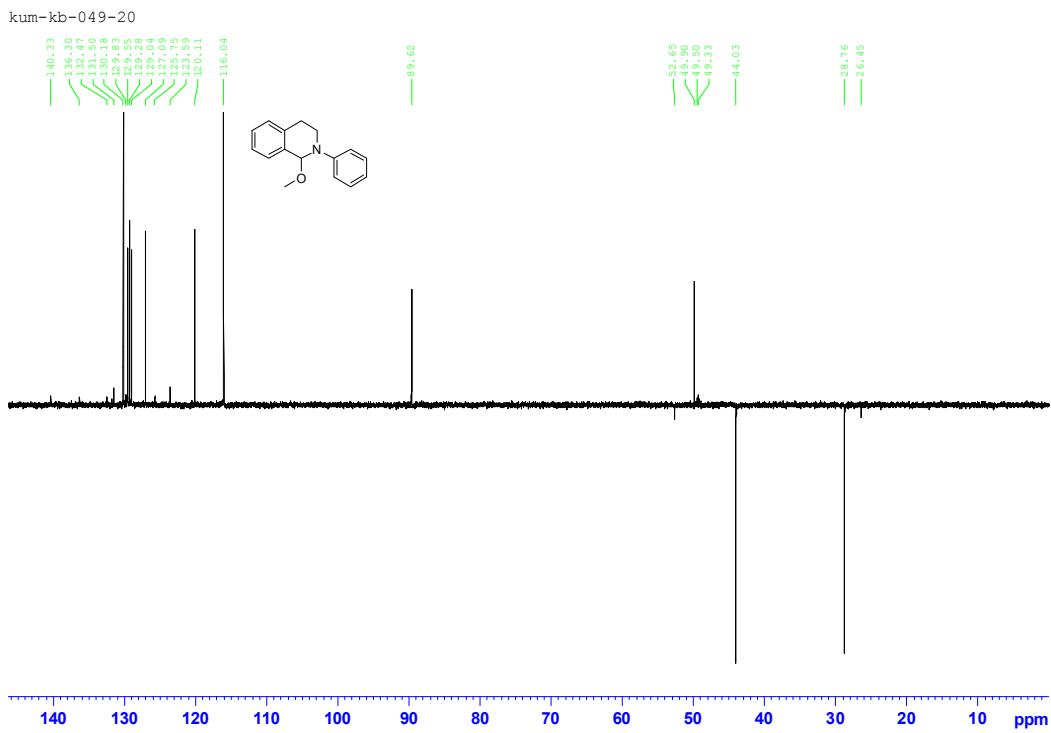
**Figure S37:** dept135 NMR in  $\text{CDCl}_3$



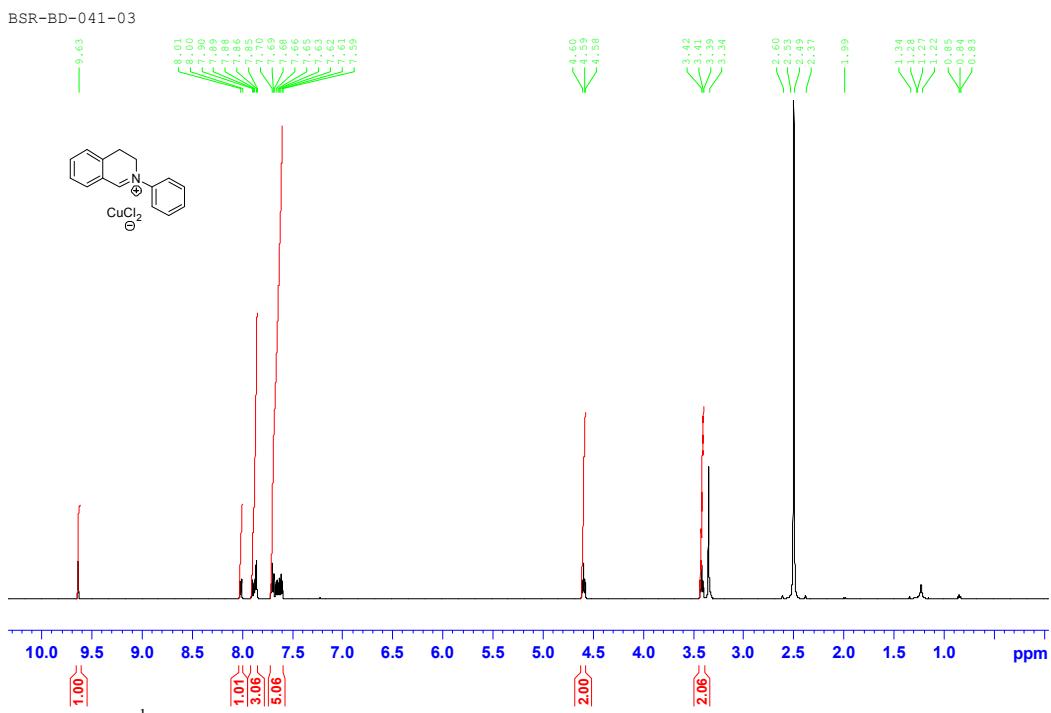
**Figure S38:**  $^1\text{H}$  NMR in  $\text{CD}_3\text{OD}$  (crude product)



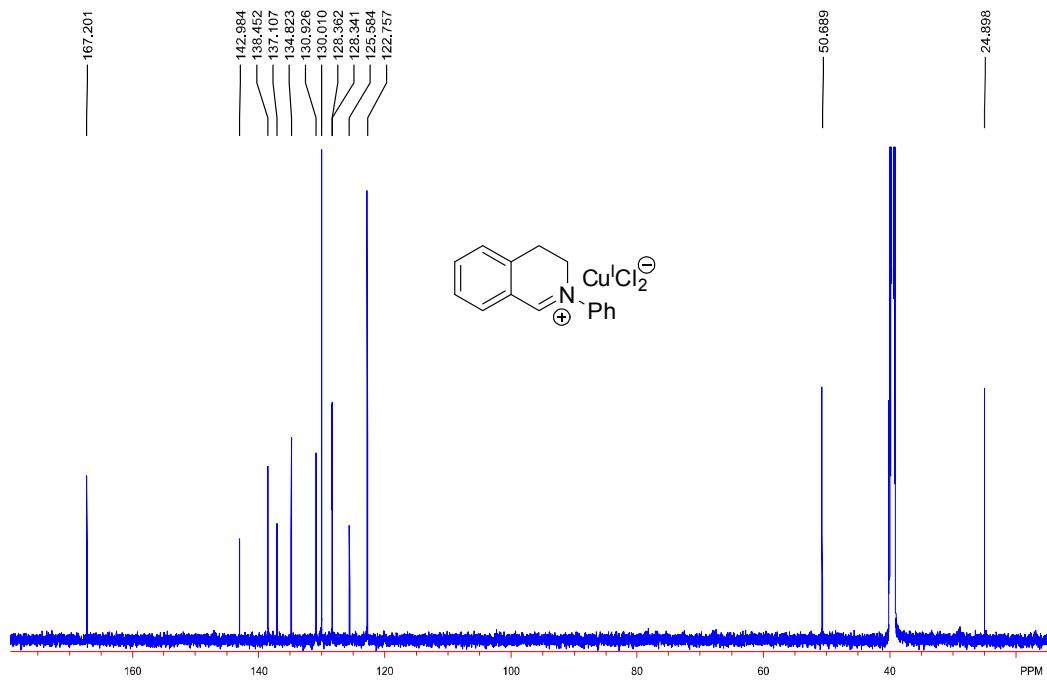
**Figure S39:**  $^{13}\text{C}$  NMR in  $\text{CD}_3\text{OD}$  (crude product)



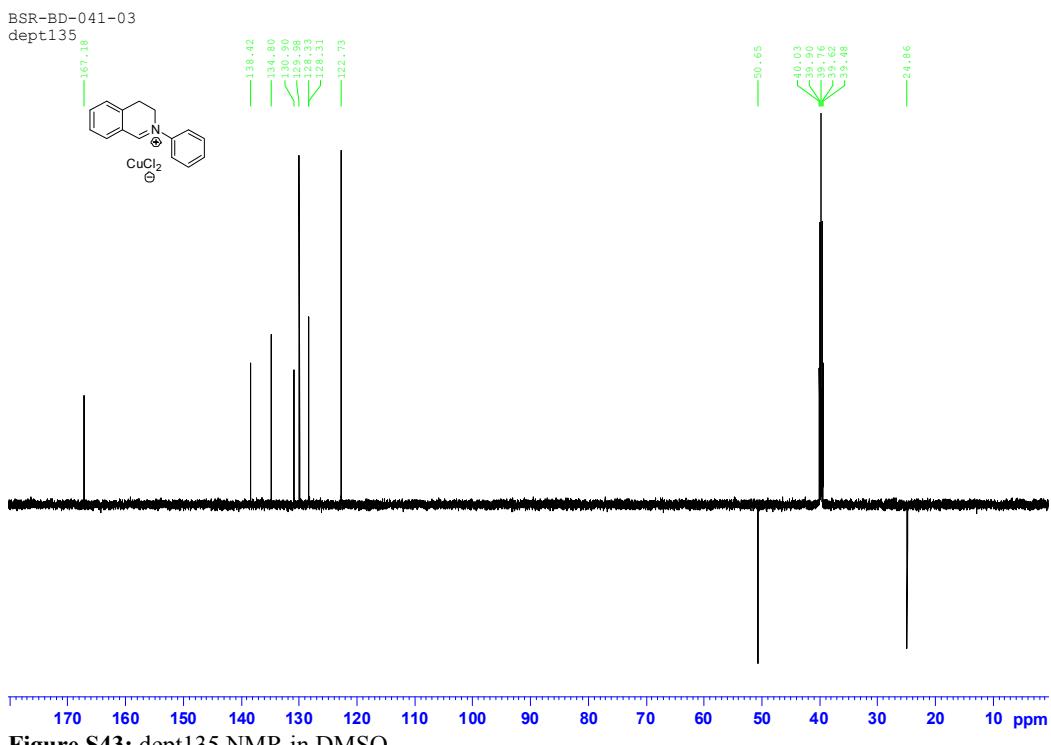
**Figure S40:** dept135 NMR in  $\text{CD}_3\text{OD}$  (crude product)



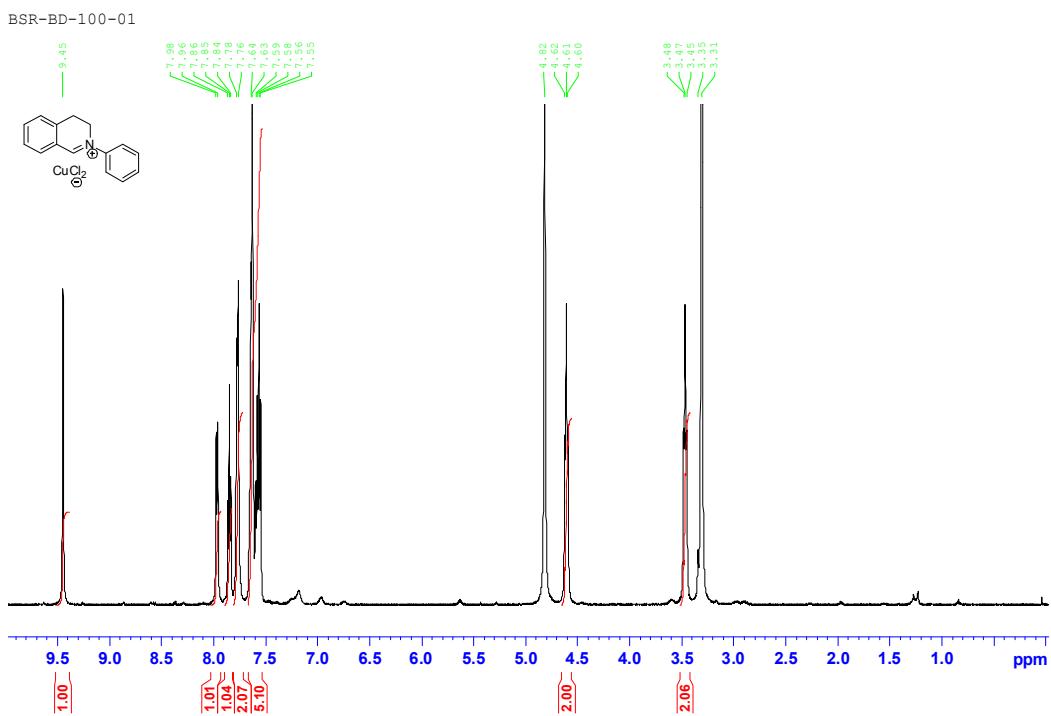
**Figure S41:**  $^1\text{H}$  NMR in DMSO



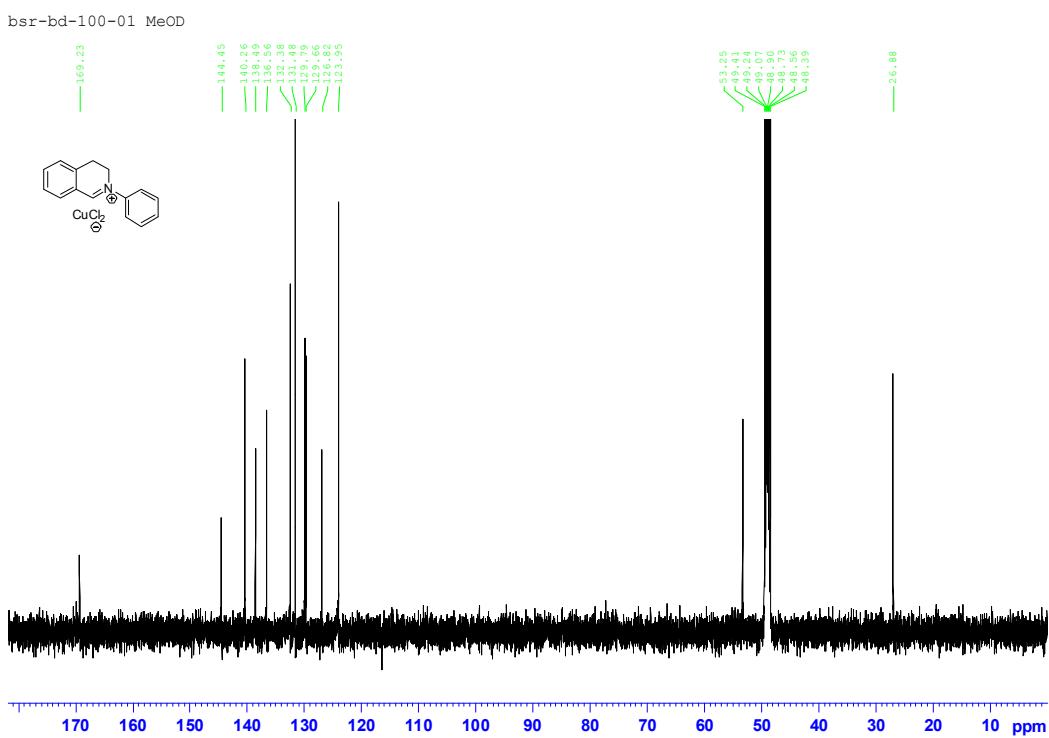
**Figure S42:**  $^{13}\text{C}$  NMR in DMSO



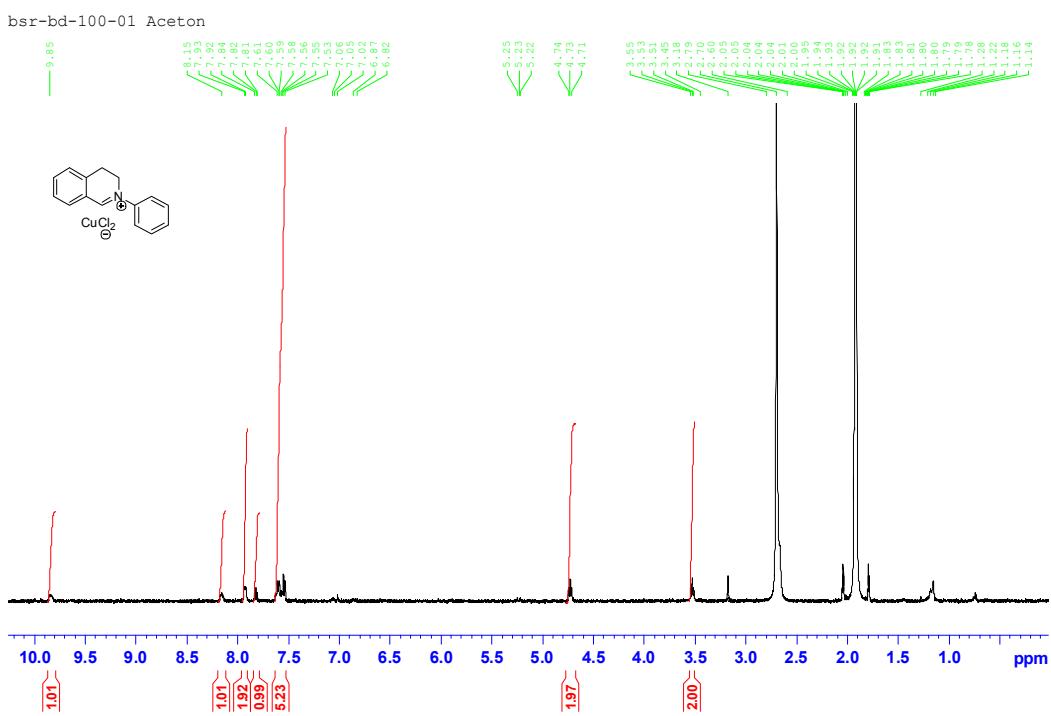
**Figure S43:** dept135 NMR in DMSO



**Figure 44:**  $^1\text{H}$  NMR in MeOH-d<sub>4</sub>



**Figure 45:**  $^{13}\text{C}$  NMR in  $\text{MeOH-d}_4$

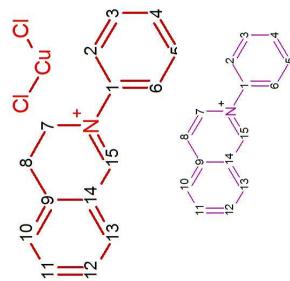
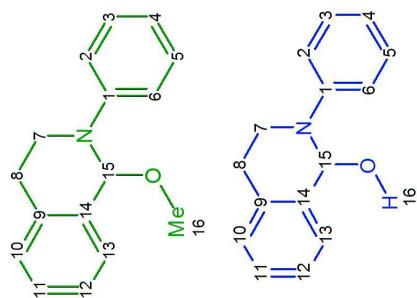
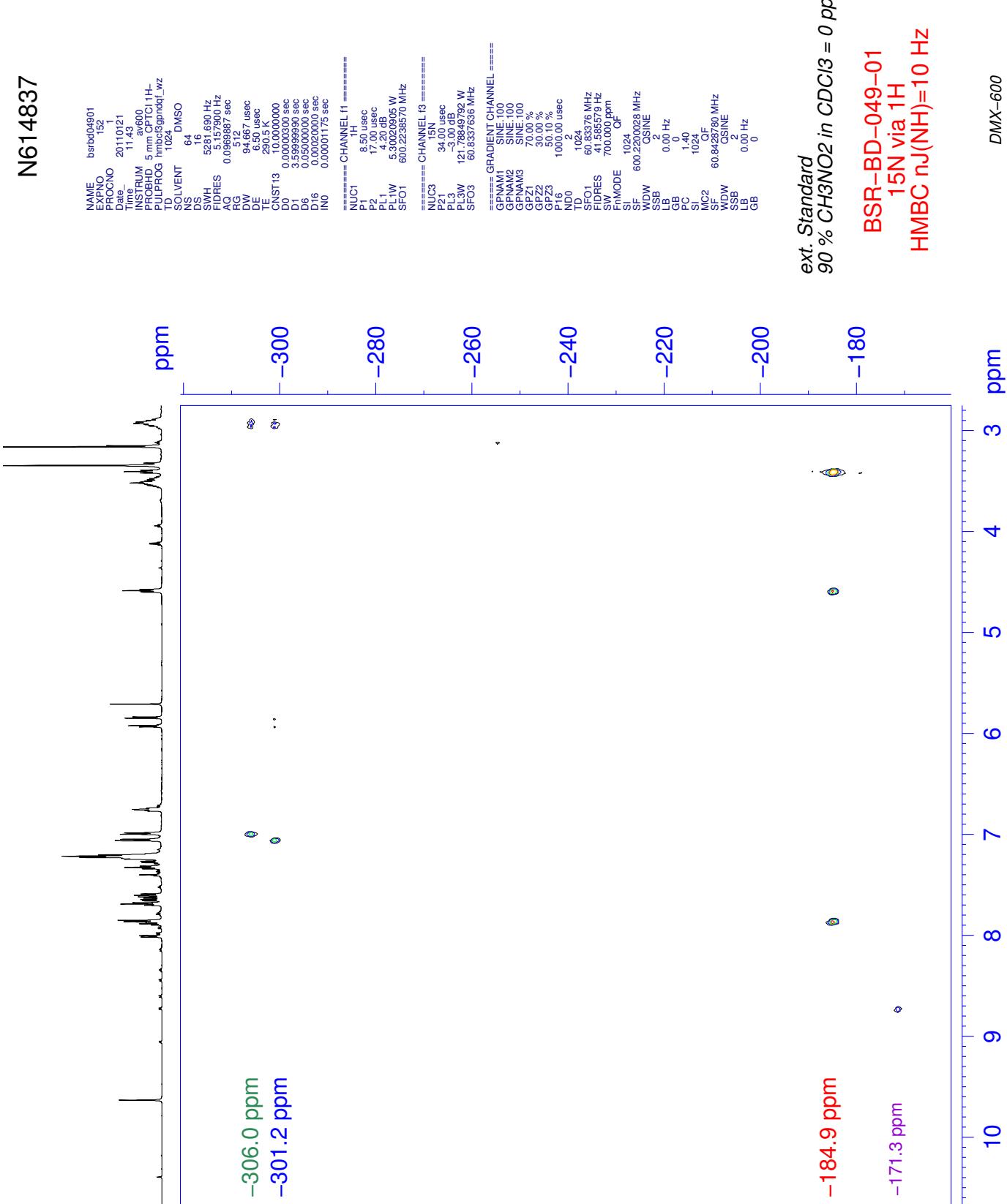


**Figure 46:**  $^1\text{H}$  NMR in  $\text{Aceton-d}_6$  (saturated solution).

### **Appendix: assignment of NMR peaks**

- $^{15}\text{N}$  via  $^1\text{H}$  HMBC of the 3:1 reaction mixture.
- Full assignment of  $^1\text{H}$ - and  $^{13}\text{C}$ -NMR peaks of components **4-7** in the 3:1 reaction mixture.

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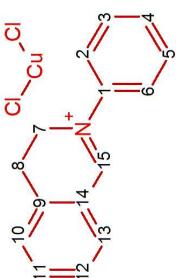
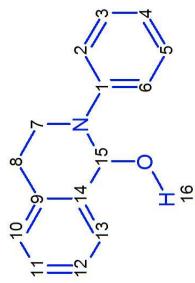
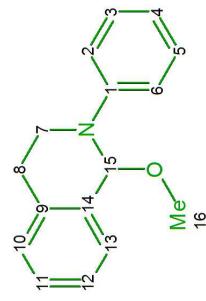
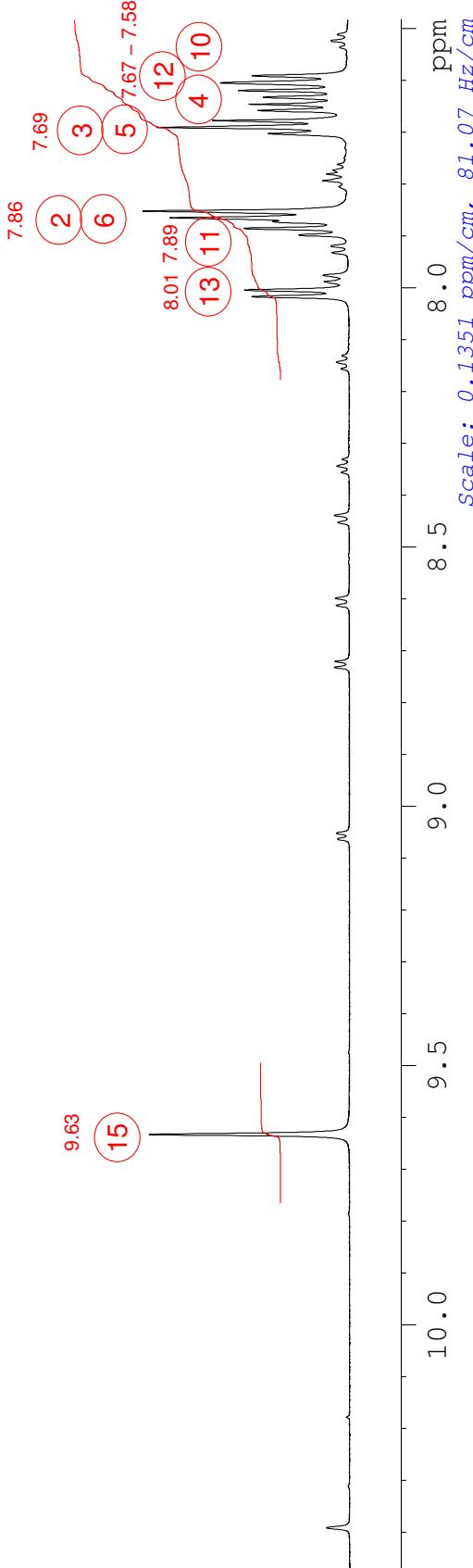
H614831

```

NAME      lsb0d04901
EXPRO    10
PROCN   1
Date_ 2010/17
Time_ 16:20
INSTRUM  av600
PROBD  5 mm CPTC1 1H-
PULPROG zg30
NS       65536
DMSO
SOLVENT 32
DS       2
DS       12019,230 Hz
SMWH
FIDRES 0.183369 Hz
AQ      2.726477 sec
RG      8
DW      41600 usec
DE      290.5 K
TE      1.000000 sec
TD1     1
TDO
===== CHANNEL f1 =====
NUC1      1H
P1        8.50 usec
PL1      4.50 usec
PL1W
SFO1      5.30029105 W
SFO1      60.3234813 MHz
SI        65536
SF      600.220669 MHz
WDW
SSB
LB
GB
PC

```

BSR-BD-049-01

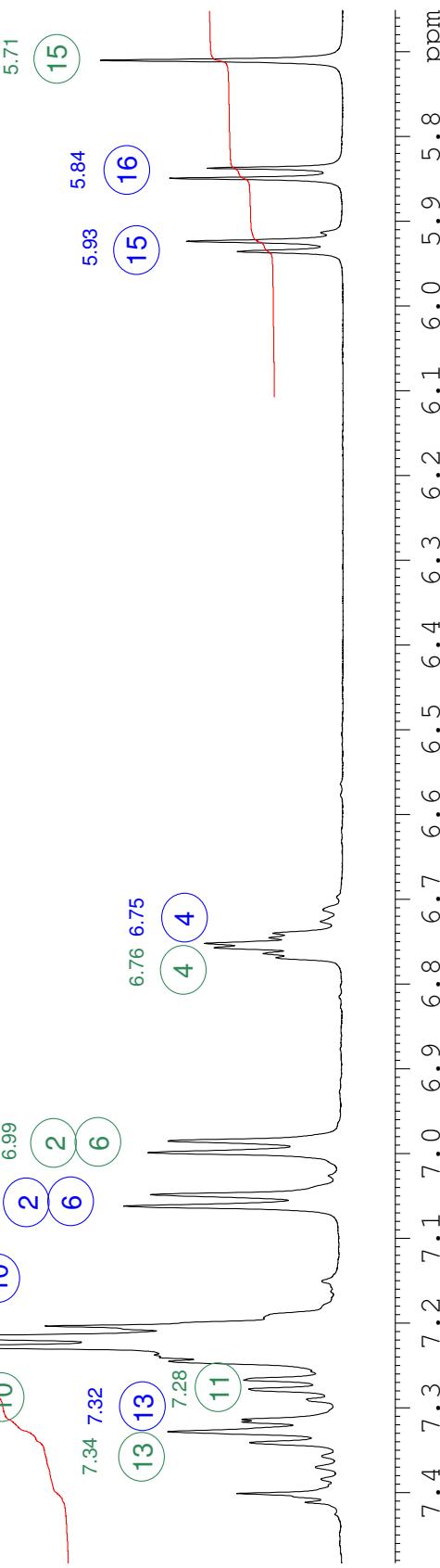


av600

Scale: 0.08273 ppm/cm, 49.66 Hz/cm

S38

BSR-BD-049-01



===== CHANNEL 11 =====  
NAME bsrdd04901  
EXPNO 10  
PROCNO 1  
Date 20110117  
Time 16.20  
INSTRUM av600  
PROBID 5 mm CPTCI 1H-  
PULPROG 65536  
TD 230  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 12019.230 Hz  
FIDRES 0.18399 Hz  
AQ 2.7263477 sec  
RG 8  
DW 41.600 usec  
DE 10.00 usec  
TE 290.5 K  
D1 1.000000 sec  
TDO 1

NAME bsrdd04901

EXPNO 10

PROCNO 1

Date 20110117

Time 16.20

INSTRUM av600

PROBID 5 mm CPTCI 1H-  
PULPROG 65536

TD 230

SOLVENT DMSO

NS 32

DS 2

SWH 12019.230 Hz

FIDRES 0.18399 Hz

AQ 2.7263477 sec

RG 8

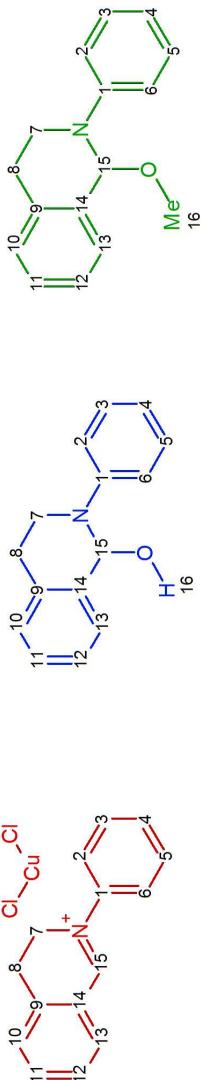
DW 41.600 usec

DE 10.00 usec

TE 290.5 K

D1 1.000000 sec

TDO 1



H614831

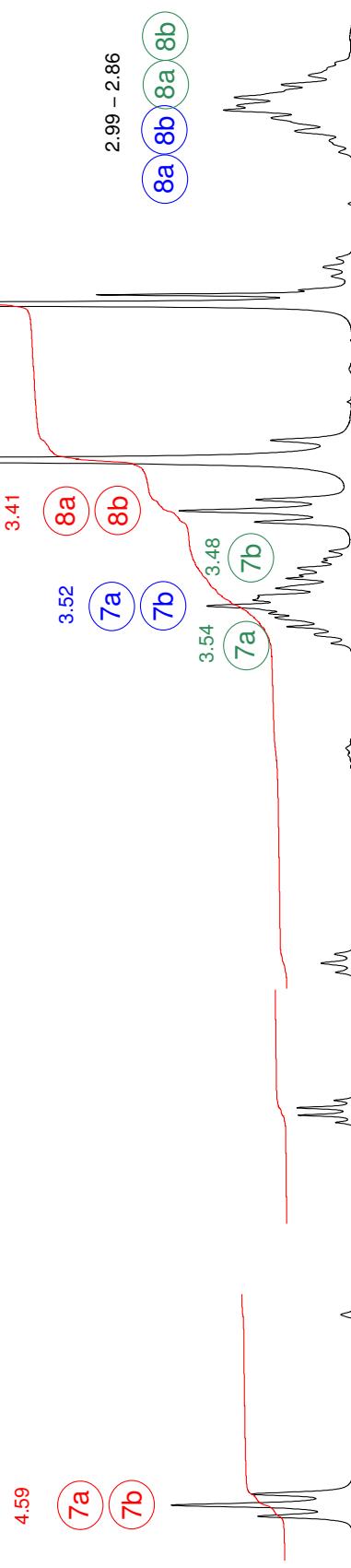
5.710  
5.849  
5.913  
5.923  
5.935  
6.011  
6.122  
6.162  
6.166  
6.169  
6.170  
6.172  
6.175  
6.176  
6.179  
6.182  
6.185  
6.198  
6.206  
6.208  
6.216  
6.226  
6.230  
6.233  
6.237  
6.240  
6.245  
6.257  
6.267  
6.277  
6.290  
6.313  
6.316  
6.328  
6.341  
6.356  
6.370  
6.384  
6.401  
6.415  
6.428  
6.441  
6.455  
6.468  
6.481  
6.494  
6.507  
6.520  
6.533  
6.546  
6.559  
6.572  
6.585  
6.598  
6.611  
6.624  
6.637  
6.650  
6.663  
6.676  
6.689  
6.702  
6.715  
6.728  
6.741  
6.754  
6.767  
6.780  
6.793  
6.806  
6.819  
6.832  
6.845  
6.858  
6.871  
6.884  
6.897  
6.910  
6.923  
6.936  
6.949  
6.962  
6.975  
6.988  
6.998  
7.011  
7.024  
7.037  
7.050  
7.063  
7.076  
7.089  
7.102  
7.115  
7.128  
7.141  
7.154  
7.167  
7.180  
7.193  
7.206  
7.219  
7.232  
7.245  
7.257  
7.270  
7.283  
7.296  
7.309  
7.322  
7.335  
7.348  
7.361  
7.374  
7.387  
7.390  
7.393  
7.396  
7.399  
7.402

av600

Scale: 0.08273 ppm/cm, 49.66 Hz/cm

S39

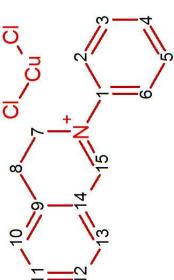
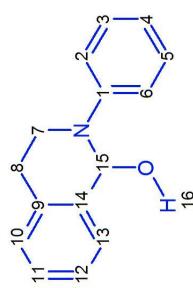
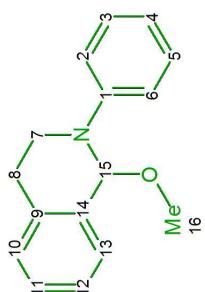
BSR-BD-049-01



===== CHANNEL 11 =====  
NAME bsrdd04901  
EXPNO 10  
PROCNO 1  
Date 20110117  
Time 16.20  
INSTRUM av600  
PROBID 5 mm CPTCI 1H-  
PULPROG 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 12019.230 Hz  
FIDRES 0.183899 Hz  
AQ 2.7253477 sec  
RG 8  
DW 4.1600 usec  
DE 10.00 usec  
TE 290.5 K  
D1 1.0000000 sec  
TD0 1

NAME bsrdd04901  
EXPNO 10  
PROCNO 1  
Date 20110117  
Time 16.20  
INSTRUM av600  
PROBID 5 mm CPTCI 1H-  
PULPROG 65536  
SOLVENT DMSO  
NS 32  
DS 2  
SWH 12019.230 Hz  
FIDRES 0.183899 Hz  
AQ 2.7253477 sec  
RG 8  
DW 4.1600 usec  
DE 10.00 usec  
TE 290.5 K  
D1 1.0000000 sec  
TD0 1

16



3.688  
3.563  
3.552  
3.543  
3.533  
3.527  
3.519  
3.512  
3.509  
3.507  
3.498  
3.486  
3.482  
3.474  
3.466  
3.463  
3.454  
3.446  
3.436  
3.427  
3.419  
3.410  
3.406  
3.393  
3.383  
3.374  
3.363  
3.352  
3.346  
3.340  
3.333  
3.322  
3.316  
3.311  
3.306  
3.276  
3.261  
3.161  
3.127  
3.116  
3.106  
2.980  
2.966  
2.955  
2.930  
2.915  
2.890  
2.874  
2.859

H614831

H614831

7.780  
7.793  
7.804  
7.819  
7.822  
7.835  
7.847  
7.851  
7.864  
7.872  
7.885  
7.897  
7.919  
7.932  
7.945  
7.964  
8.004  
8.016  
8.143  
8.156  
8.343  
8.356  
8.438  
8.452  
8.598  
8.612  
8.720  
8.732  
9.052  
9.063  
9.633

```

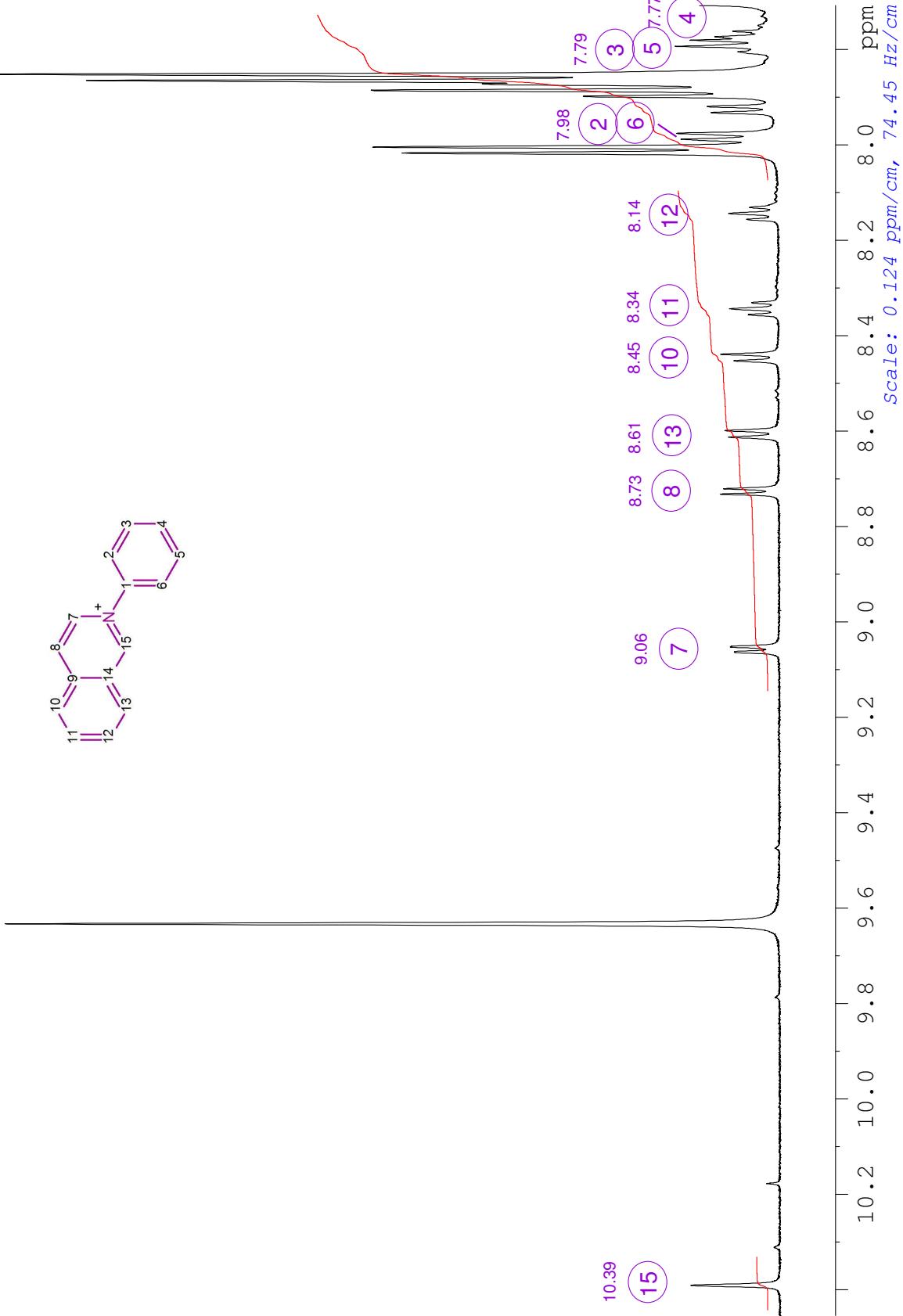
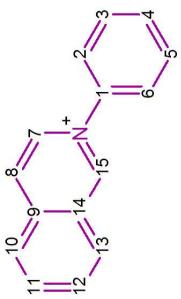
NAME      bsr0d04901
EXPNO     10
PROCNO    1
Date      20110117
Time      16.20
INSTRUM   av600
PROBID    5 mm CPTCI 1H-
PULPROG  65536
TD        2^30
SOLVENT   DMSO
NS        32
DS        2
SWH      12019.230 Hz
FIDRES   0.18399 Hz
AQ        2.7253477 sec
RG        8
DW        41.600 usec
DE        10.00 usec
TE        290.5 K
D1        1.0000000 sec
TDO      1

```

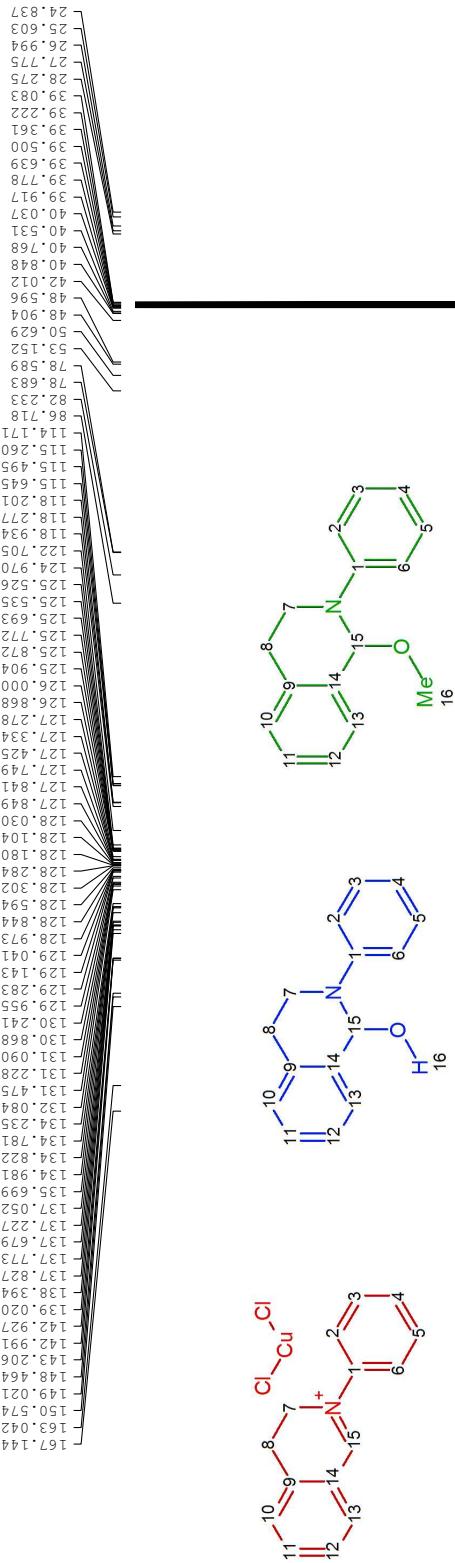
```

===== CHANNEL 11 =====
NUC1      1H
P1        8.50 usec
PL1      4.420 dB
PL1W    5.30020905 W
SFO1    600.2234813 MHz
SF       65536
WDW      no
SSB      0.00 Hz
LB      0.00 Hz
GB      1.00
PC      1.00

```



10.391



```

NAME          bsb0d04901
EXPNO         11
PROCNO        1
Date         20110118
Time         12:12
INSTRUM      av600
PROBHD      5 mm CPTCI 1H-
PULPROG     90904
TD           290dc30
SOLVENT      DMSO
NS            4000
DS            128
SWH         45454.547 Hz
FIDRES     0.999940 sec
AQ            512
RG            1.000 usec
DE            50.78 usec
TE            290.5 K
D1           0.1000000 sec
D11          0.0300000 sec
TDO          1

```

```

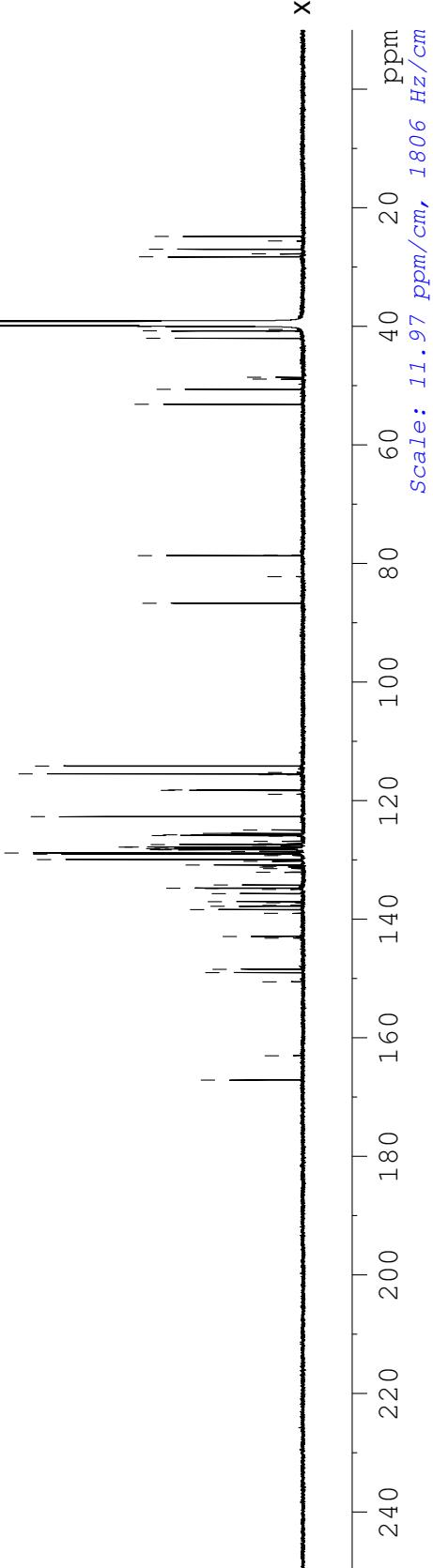
===== CHANNEL 11 =====
NUC1          13C
P1           11.00 usec
PL1          -1.00 dB
PL1W        109.73103333 W
SF01        150.9405316 MHz

```

```

===== CHANNEL 12 =====
CPDPRG2      WALT65
NUC2          1H
PCPD2        70.00 usec
PL2           4.20 dB
PL12         22.51 dB
PL12W       5.30020905 W
PL12W       0.07821552 W
SF02        600.2224098 MHz
SF           131.0722
SF01        150.9255699 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.00

```



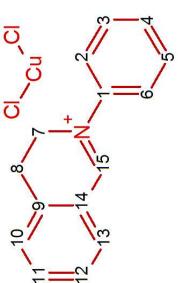
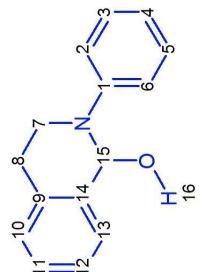
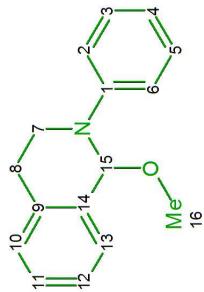
132.084  
131.475  
131.228  
131.090

135.699  
134.981  
134.822  
134.235  
137.052  
137.227  
137.679  
137.773  
138.394  
139.020

143.206  
142.991  
142.927

149.021  
148.464

150.574



163.042

167.144

```

NAME          bstd04901
EXPNO         11
PROCNO        1
Date         20110118
Time         12:12
INSTRUM      av600
PROBHD      5 mm CPTCI 1H-
PULPROG     90904
TD           40000
SOLVENT      DMSO
NS            128
DS            128
SWH         45454.547 Hz
FIDRES     0.500028 Hz
AQ            0.999940 sec
RG            512
DW           1.000 usec
DE            50.78 ussec
TE            290.5 K
D1           0.1000000 sec
T1           0.03000000 sec
TD0           1

```

```

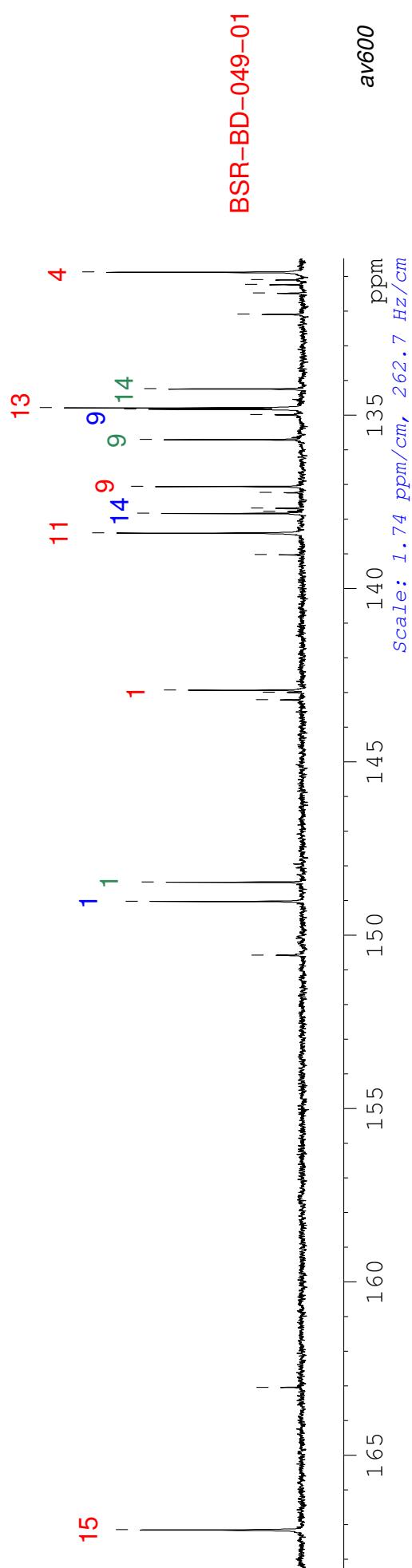
===== CHANNEL 11 =====
NUC1          13C
PL1           11.00 usec
PL1W         109.73103333 W
SF01         150.9405316 MHz

```

```

===== CHANNEL 12 =====
CPDPRG2      WALT65
NUC2          1H
PCPD2        -1.00 usec
PL2           4.20 dB
PL12          22.51 dB
PL12W        5.30020905 W
PL12W        0.07821552 W
SF02         600.2224009 MHz
SF           131.0722
SF02         150.9255099 MHz
WDW           EM
SSB            0
LB            1.00 Hz
GB            0
PC            1.00

```



C614832

124.970

125.526  
125.535  
125.693  
125.772  
125.822  
125.904  
126.000

126.868

127.278  
127.334  
127.425

127.749

127.881

128.030

128.104

128.180

128.284

128.302

128.594

128.844

128.973

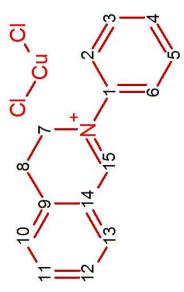
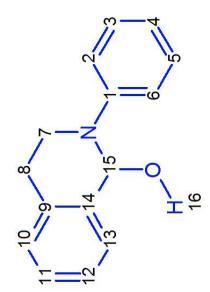
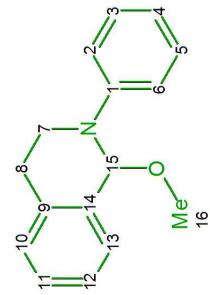
129.041

129.143

129.283

129.555

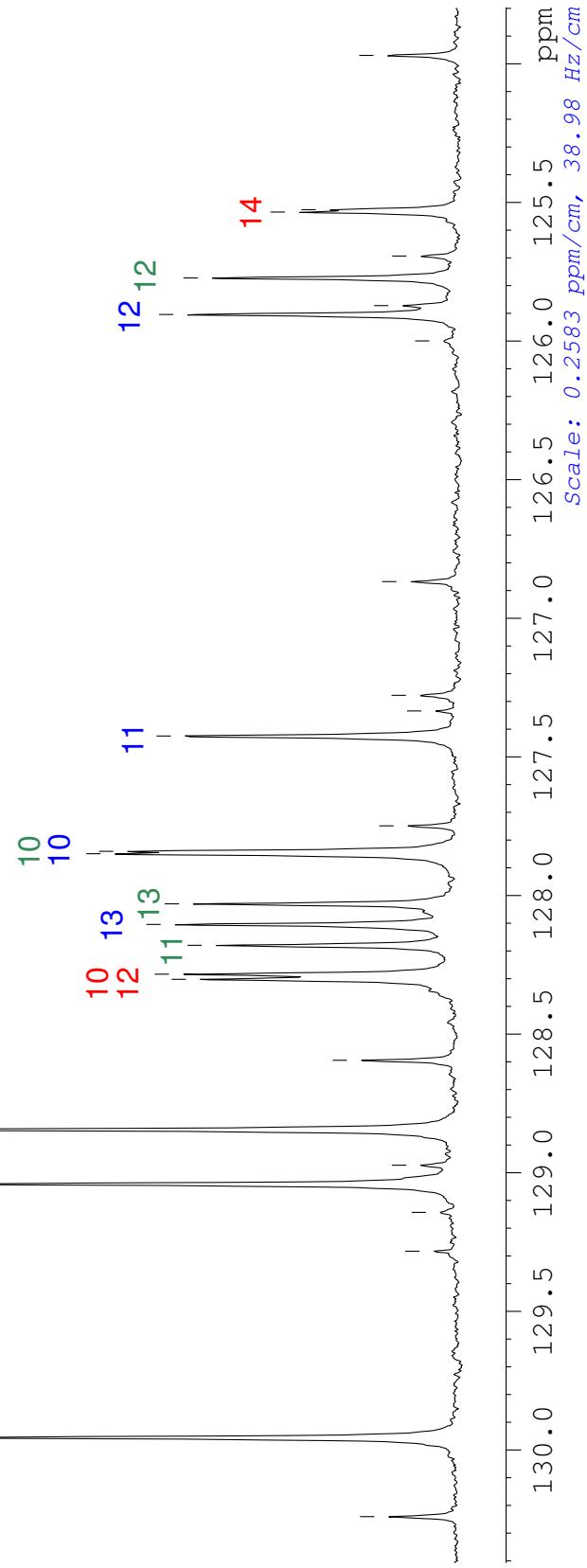
130.241

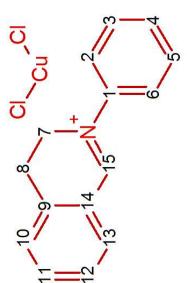
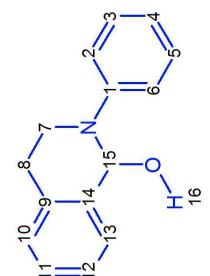
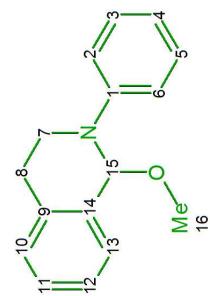


3 5

3 5

3 5





114.171

115.260

115.495

115.645

118.277

118.934

122.705

```

NAME          bstd04901
EXPNO         11
PROCNO        1
Date         20110118
Time         12:12
INSTRUM      av600
PROBHD      5 mm CPTCI 1H-
PULPROG     90/904
TD           40000
SOLVENT      DMSO
NS            128
DS            128
SWH         45454.547 Hz
FIDRES     0.500028 Hz
AQ            0.999940 sec
RG            512
DW           1.000 usec
DE            50.78 usec
TE            290.5 K
D1       0.1000000 sec
T1           0.0300000 sec
TD0          1

```

```

===== CHANNEL 11 =====
NUC1          13C
PL1          11.00 usec
PL1W        109.73103333 W
SF01        150.9405316 MHz

```

```

===== CHANNEL 12 =====
CPDPRG2      Wait65
NUC2          1H
PCPD2        70.00 usec
PL2          4.20 dB
PL12         22.51 dB
PL12W       5.30020905 W
PL12W       0.07821552 W
SF02        600.2224098 MHz
SF          131.0722
SF        150.9255699 MHz
WDW          EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.00

```

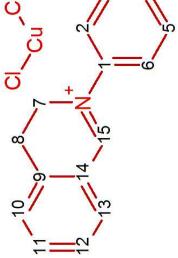
**2 6****2 6****4 4****2 6****BSR-BD-049-01**

av600

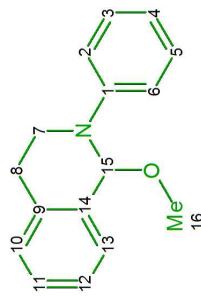
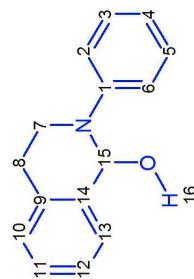
Scale: 0.4639 ppm/cm, 70.01 Hz/cm

24.837  
25.603  
26.994  
27.775  
28.275

39.083  
39.222  
39.361  
39.500  
39.639  
39.778  
39.917  
40.037  
40.531  
40.768  
40.848  
42.012  
48.596  
48.904  
50.629  
53.152

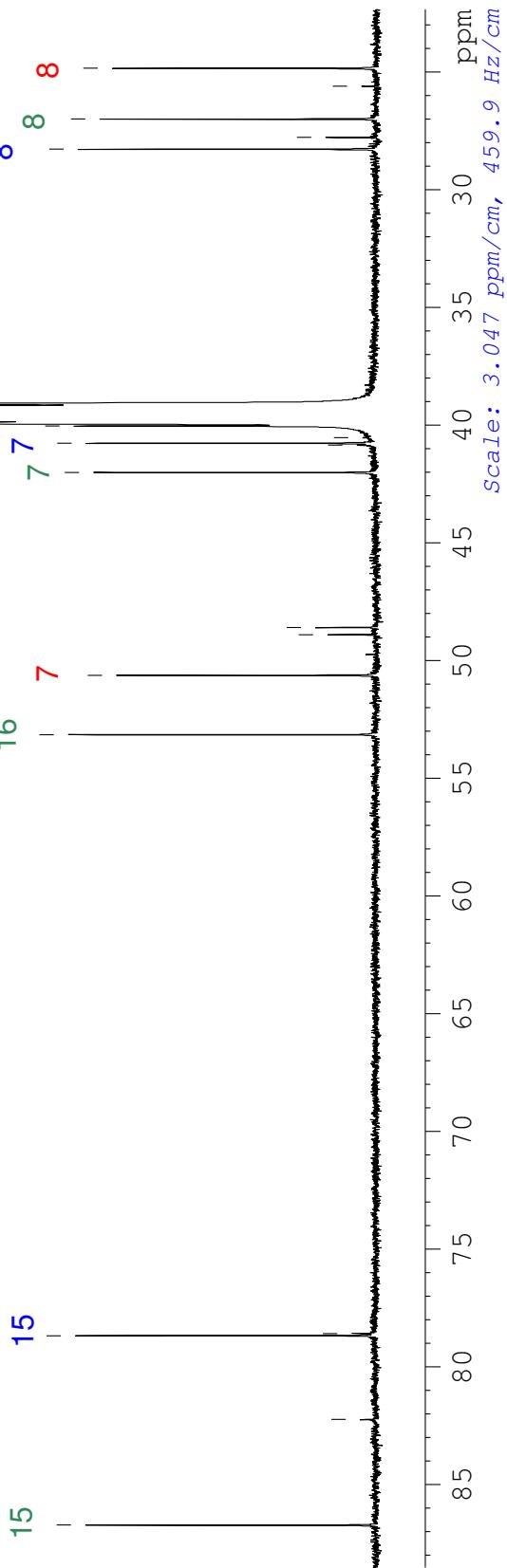


78.589  
82.233  
86.718

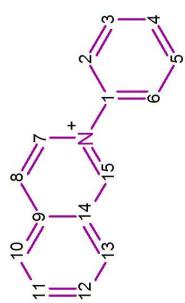


```

=====
NAME      bstd04901
EXPNO    11
PROCNO   1
Date     20110118
Time     12:12
INSTRUM  av600
PROBHD  5 mm CPTCI 1H-
PULPROG  90904
TD       40000
SOLVENT  DMSO
NS       128
SWH     45454.547 Hz
FIDRES  0.500028 Hz
AQ      0.999940 sec
RG      512
DW      1.000 usec
DE      50.78 ussec
TE      290.5 K
D1      0.1000000 sec
T1      0.0300000 sec
TD0      1
===== CHANNEL 11 =====
NUC1    13C
P1      11.00 usec
PL1    -1.00 dB
PL1W   109.73103333 W
SF01   150.9405316 MHz
===== CHANNEL 12 =====
CPDPRG2  Wait65
NUC2    1H
PCPD2   70.00 usec
PL2      4.20 dB
PL12    22.51 dB
PL12W   5.30020905 W
PL12W   0.07821552 W
SF02   600.2224098 MHz
SF    131.0722 MHz
WDW    EM
SSB    0
LB      1.00 Hz
GB      0
PC      1.00
=====
```



134.235  
134.781  
134.822  
134.981  
135.699  
137.052  
137.227  
137.679  
137.773  
137.827  
138.394  
139.020  
142.927  
142.991  
143.206  
148.464  
149.021  
150.574



```

=====
NAME      bs1bd04901
EXPNO    11
PROCNO   1
Date     20110118
Time     12:12
INSTRUM  av600
PROBHD  5 mm CPTCI 1H-
PULPROG  29dc30
TD       90904
SOLVENT  DMSO
NS       4000
DS       128
SWH     45454.547 Hz
FIDRES  0.00028 Hz
AQ      0.999940 sec
RG      512
DW      1.000 usec
DE      50.78 usec
TE      290.5 K
D1      0.1000000 sec
T1      0.0300000 sec
TD0      1
===== CHANNEL 11 =====
NUC1    13C
P1      11.00 usec
PL1    -1.00 dB
PL1W   109.73103333 W
SF01   150.9405316 MHz
===== CHANNEL 12 =====
CPDPRG2  WALT65
NUC2    1H
PCPD2   70.00 usec
PL2     4.20 dB
PL12    22.51 dB
PL12W   5.30020905 W
PL12W   0.07821552 W
SF02   600.2224098 MHz
SI      131072
SF     150.9255699 MHz
WDW    EM
SSB     0
LB      1.00 Hz
GB      0
PC      1.00
=====
```

BSR-BD-049-01

av600

Scale: 0.8174 ppm/cm, 123.4 Hz/cm

S46

124.970

125.526  
125.535  
125.693  
125.772  
125.872  
125.904  
126.000

126.868

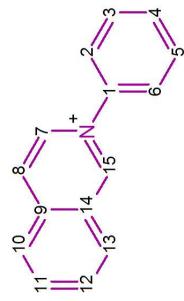
127.425  
127.334  
127.278  
127.849  
127.841  
128.030  
128.104  
128.180  
128.284  
128.302  
128.341  
128.594

128.844  
128.973  
128.143  
129.143  
129.283

129.955

130.241

130.868  
131.090  
131.228  
131.475  
132.084



```

=====
NAME      bsb0d4901
EXPNO    11
PROCNO   1
Date     20110118
Time     12.12
INSTRUM  av600
PROBHD  5 mm CPTCI 1H
PULPROG  90904
TD       4000
SOLVENT  DMSO
NS        128
SWH     45454.547 Hz
FIDRES  0.500028 Hz
AQ      0.999940 sec
RG       512
DW       1.000 usec
DE       50.78 ussec
TE       290.5 K
D1      0.1000000 sec
T1D0
===== CHANNEL 11 =====
NUC1      13C
P1      11.00 usec
PL1     -1.00 dB
PL1W    109.73103333 W
SF01    150.9405316 MHz
===== CHANNEL 12 =====
CPDPRG2  WALT65
NUC2      1H
PCPD2    4.00 usec
PL2      4.20 dB
PL12     22.51 dB
PL12W   5.30020905 W
PL12W   0.0782152 W
SF02    600.2224009 MHz
SF      131.072
SF     150.9255699 MHz
WDW     EM
SSB      0
LB      1.00 Hz
GB      0
PC      1.00
=====
```

```

=====
NAME      bsb0d4901
EXPNO    11
PROCNO   1
Date     20110118
Time     12.12
INSTRUM  av600
PROBHD  5 mm CPTCI 1H
PULPROG  90904
TD       4000
SOLVENT  DMSO
NS        128
SWH     45454.547 Hz
FIDRES  0.500028 Hz
AQ      0.999940 sec
RG       512
DW       1.000 usec
DE       50.78 ussec
TE       290.5 K
D1      0.1000000 sec
T1D0
===== CHANNEL 11 =====
NUC1      13C
P1      11.00 usec
PL1     -1.00 dB
PL1W    109.73103333 W
SF01    150.9405316 MHz
===== CHANNEL 12 =====
CPDPRG2  WALT65
NUC2      1H
PCPD2    4.00 usec
PL2      4.20 dB
PL12     22.51 dB
PL12W   5.30020905 W
PL12W   0.0782152 W
SF02    600.2224009 MHz
SF      131.072
SF     150.9255699 MHz
WDW     EM
SSB      0
LB      1.00 Hz
GB      0
PC      1.00
=====
```

