

# A Versatile Approach to Ullmann C–N Couplings at Room Temperature: New Families of Nucleophiles and Electrophiles for Photoinduced, Copper-Catalyzed Processes

Daniel T. Ziegler, Junwon Choi, José María Muñoz-Molina, Alex C. Bissember, Jonas C. Peters,\*  
and Gregory C. Fu\*

*Division of Chemistry and Chemical Engineering, California Institute of Technology,  
Pasadena, California 91125*

## Supporting Information

### Table of Contents

I. General Information	S–1
II. Photoinduced, Copper-Catalyzed N-Arylations (Tables 1–5)	S–2
III. Nucleophile Competition Experiments (Table 6)	S–14
IV. Electrophile Competition Experiments (eq 2)	S–14
V. Functional-Group Tolerance Experiments (Table 7)	S–14
VI. Photoinduced, Copper-Catalyzed N-Alkenylations/Alkynylations (Table 8)	S–15
VII. <sup>1</sup> H NMR Spectra	S–18

### I. General Information

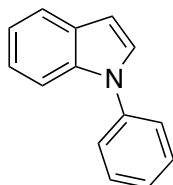
The following reagents were purchased and used as received unless otherwise specified: indole (Aldrich), 6-methoxyindole (AstaTech), 3-methylindole (Aldrich), 2-methylindole (Alfa Aesar), 7-methylindole (Aldrich), benzimidazole (Alfa Aesar), 5-methoxybenzimidazole (Aldrich), 2-methylbenzimidazole (Aldrich), imidazole (Alfa Aesar), 2-methylimidazole (Aldrich), carbazole (Aldrich; recrystallized), 3-methoxycarbazole (Matrix Scientific), iodobenzene (Aldrich), 4-iodotoluene (Aldrich), 2-iodotoluene (Aldrich), 4-iodoanisole (Aldrich), 4-iodobenzonitrile (Aldrich), 3-iodopyridine (Aldrich), bromobenzene (Avocado), 4-chlorobenzonitrile (Avocado), 1-ethyl-4-iodobenzene (Avocado), 4-bromotoluene (Aldrich), 4-chlorotoluene (Aldrich), methyl octanoate (Acros), 1-methyl-2-piperidone (Aldrich), benzylacetone (Aldrich), dicyclohexylamine (Aldrich), cyclohexylamine (Aldrich), chlorobenzene (Aldrich), *trans*-5-decene (Aldrich), *cis*-5-decene (TCI), 5-decyne (Lancaster), dibenzyl ether (Alfa Aesar), bromomethylenecyclohexane (Aldrich), CuI (Aldrich), LiO*t*-Bu (Alfa Aesar), and *t*-BuOH (Aldrich; anhydrous). CH<sub>3</sub>CN was deoxygenated and dried by sparging with nitrogen followed by passage through an activated alumina column (S. G. Water) prior to use.

All coupling reactions were carried out using a Luzchem LZC–4V photoreactor at 254 nm (UVC). <sup>1</sup>H NMR data and <sup>13</sup>C NMR data were collected on a VARIAN 500 MHz spectrometer at ambient temperature. GC analyses were carried out on an Agilent 6890 series system with a DB-1 column (length 30 m, I.D. 0.25 mm) or an HP-5 column (length 30 m, I.D. 0.25 mm) or on an Agilent 6850 series system with a BETA DEX 120 column (length 30 m, I.D. 0.25 mm).

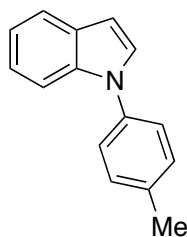
## II. Photoinduced, Copper-Catalyzed N-Arylations (Tables 1–5)

**General Procedure.** The nitrogen heterocycle (1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), and CuI (19.0 mg, 0.10 mmol) were added to an oven-dried 10-mL quartz test tube that contained a stir bar. The test tube was fitted with a rubber septum, the joint was wrapped with electrical tape, and the test tube was evacuated and backfilled with nitrogen (three cycles). Then, CH<sub>3</sub>CN (4.0 mL) and the aryl iodide (1.40 mmol; if the aryl iodide is a solid, then it was added immediately after the addition of CuI) were added in turn via syringe. The test tube was detached from the nitrogen manifold, and the puncture holes in the septum were covered with vacuum grease. The resulting mixture was stirred for 5 min, and then the test tube was transferred to a Luzchem LZC-4V photoreactor, where it was irradiated at 254 nm for 24 h (adequate stirring is important). Next, the mixture was passed through a long plug of silica gel (monitored by TLC), the solvent was removed, and the residue was purified by column chromatography.

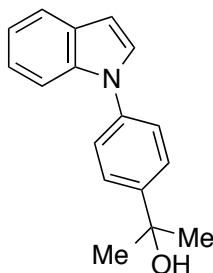
Notes: (a) A Honeywell ultraviolet air treatment system (model #RUVLAMP1), available for ~\$110 from retail outlets such as Amazon or The Home Depot, furnishes a comparable result: indole and iodobenzene couple in 63% yield (calibrated GC analysis) after 48 h. (b) Use of a borosilicate, rather than a quartz, test tube leads to a low yield of the C–N coupling product.



**1-Phenyl-1H-indole (Table 1, entry 1) [16096-33-6].** The title compound was synthesized according to the General Procedure from indole (117 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography (hexanes). Pale-yellow oil. First run: 142 mg (73% yield). Second run: 148 mg (77% yield).



**1-(p-Tolyl)-1H-indole (Table 1, entry 2) [167283-32-1].** The title compound was synthesized according to the General Procedure from indole (117 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-iodotoluene (305 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). Colorless oil. First run: 140 mg (68%). Second run: 143 mg (69%).



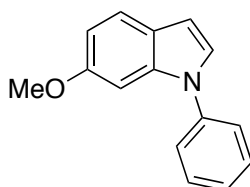
**2-(4-(1*H*-Indol-1-yl)phenyl)propan-2-ol (Table 1, Entry 3).** The title compound was synthesized according to the General Procedure from indole (117 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 2-(4-iodophenyl)propan-2-ol (367 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (20% EtOAc/hexanes) and purified by column chromatography on silica gel (7.5% EtOAc/hexanes→15% EtOAc/hexanes). Pale-orange solid. First run: 144 mg (57%). Second run: 143 mg (57%).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.71–7.69 (m, 1H), 7.67–7.62 (m, 2H), 7.60–7.56 (m, 1H), 7.51–7.46 (m, 2H), 7.34 (d, 1H, *J* = 3.0 Hz), 7.23 (ddd, 1H, *J* = 8.0, 7.0, 1.0 Hz), 7.18 (ddd, 1H, *J* = 8.0, 7.0, 1.0 Hz), 6.69 (dd, 1H, *J* = 3.0, 1.0 Hz), 1.81 (br s, 1H), 1.66 (s, 6H).

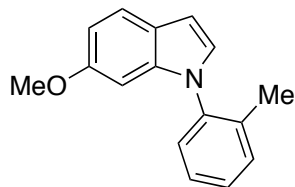
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 147.5, 138.5, 136.0, 129.4, 128.1, 125.9, 124.2, 122.4, 121.2, 120.5, 110.7, 103.6, 72.6, 32.0.

FT-IR (neat) 3541, 3399, 3103, 3049, 2974, 2927, 2868, 1606, 1582, 1570, 1519, 1475, 1457, 1412, 1363, 1347, 1334, 1317, 1298, 1281, 1256, 1234, 1213, 1170, 1137, 1114, 1094, 1066, 1015, 955, 909, 883, 862, 840, 770, 762, 742, 720 cm<sup>-1</sup>.

MS (ESI) *m/z* (M<sup>+</sup>+H) calcd for C<sub>17</sub>H<sub>18</sub>NO: 252, found: 252.



**6-Methoxy-1-phenyl-1*H*-indole (Table 1, entry 4) [487058-34-4].** The title compound was synthesized according to the General Procedure from 6-methoxyindole (147 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by normal-phase column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes) followed by reverse-phase column chromatography on C-18 silica gel (10%→100% CH<sub>3</sub>CN/water). White solid. First run: 147 mg (66%). Second run: 150 mg (67%).



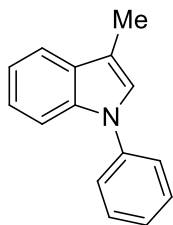
**6-Methoxy-1-(*o*-tolyl)-1*H*-indole (Table 1, entry 5).** The title compound was synthesized according to the General Procedure from 6-methoxyindole (147 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 2-iodotoluene (305 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by normal-phase column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes) followed by reverse-phase column chromatography on C-18 silica gel (10%→100% CH<sub>3</sub>CN/water). Yellow oil. First run: 154 mg (65% yield). Second run: 165 mg (70% yield).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.56 (d, 1H, *J* = 8.5 Hz), 7.40–7.36 (m, 2H), 7.35–7.31 (m, 2H), 7.06 (d, 1H, *J* = 3.2 Hz), 6.82 (dd, 1H, *J* = 8.5, 2.2 Hz), 6.59 (d, 1H, *J* = 3.2 Hz), 6.50 (s, 1H), 3.76 (s, 3H), 2.09 (s, 3H).

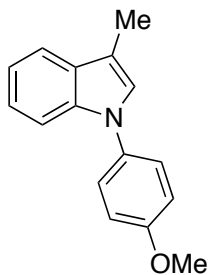
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 156.8, 138.5, 137.8, 136.0, 131.4, 128.3, 128.2, 127.8, 127.0, 122.6, 121.5, 110.1, 102.5, 94.0, 55.8, 17.8.

FT-IR (neat) 3102, 3026, 2994, 2952, 2831, 1621, 1603, 1573, 1513, 1487, 1459, 1380, 1340, 1324, 1292, 1279, 1225, 1205, 1177, 1121, 1095, 1031, 927, 806, 769, 746, 720 cm<sup>-1</sup>.

MS (ESI) *m/z* (M<sup>+</sup>+H) calcd for C<sub>16</sub>H<sub>16</sub>NO: 238, found: 238.

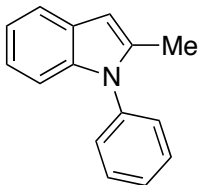


**3-Methyl-1-phenyl-1*H*-indole (Table 1, entry 6) [112817-88-6].** The title compound was synthesized according to the General Procedure from 3-methylindole (131 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). Colorless oil. First run: 152 mg (73%). Second run: 145 mg (70%).

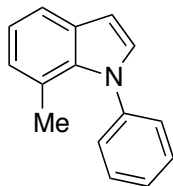


**1-(4-Methoxyphenyl)-3-methyl-1*H*-indole (Table 1, entry 7) [876337-56-3].** The title compound was synthesized according to the General Procedure from 3-methylindole (131 mg, 1.00

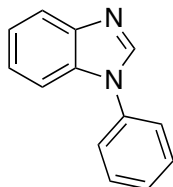
mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-iodoanisole (328 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→2% Et<sub>2</sub>O/hexanes). Colorless oil. First run: 138 mg (58%). Second run: 138 mg (58%).



**2-Methyl-1-phenyl-1H-indole (Table 1, entry 8) [16176-77-5].** The title compound was synthesized according to the General Procedure from 2-methylindole (131 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). Colorless oil. First run: 122 mg (59%). Second run: 124 mg (60%).



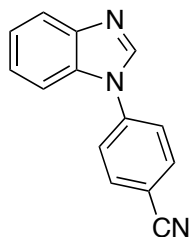
**7-Methyl-1-phenyl-1H-indole (Table 1, entry 9) [473918-43-3].** The title compound was synthesized according to the General Procedure from 7-methylindole (131 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). White solid. First run: 139 mg (67%). Second run: 133 mg (64%).



**1-Phenyl-1H-benzimidazole (Table 2, entry 1) [2622-60-8].** The title compound was synthesized according to the General Procedure from benzimidazole (118 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol), except that a mixture of *t*-BuOH (1.0 mL) and CH<sub>3</sub>CN (3.0 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. The reaction mixture was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by

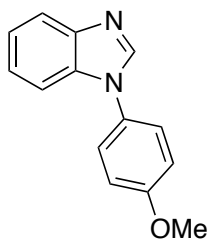
column chromatography on silica gel (0.75% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 15%→25% EtOAc/hexanes). Yellow oil. First run: 158 mg (81%). Second run: 165 mg (85%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.



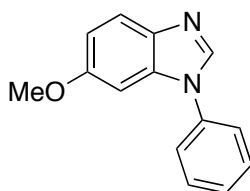
**4-(1H-Benzo[d]imidazol-1-yl)benzonitrile (Table 2, entry 2) [25699-95-0].** The title compound was synthesized according to the General Procedure from benzimidazole (118 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-iodobenzonitrile (321 mg, 1.40 mmol), except that a mixture of *t*-BuOH (1.0 mL) and CH<sub>3</sub>CN (3.0 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. The product was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography on silica gel (1% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 40%→55% EtOAc/hexanes). Yellow solid. First run: 185 mg (84%). Second run: 180 mg (82%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.

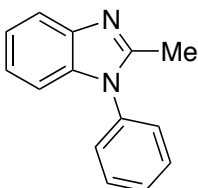


**1-(4-Methoxyphenyl)-1H-benzo[d]imidazole (Table 2, entry 3) [2622-61-9].** The title compound was synthesized according to the General Procedure from benzimidazole (118 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-iodoanisole (328 mg, 1.40 mmol), except that a mixture of *t*-BuOH (1.0 mL) and CH<sub>3</sub>CN (3.0 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. The reaction mixture was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography on silica gel (1% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 30%→50% EtOAc/hexanes). Yellow solid. First run: 177 mg (79%). Second run: 164 mg (73%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.

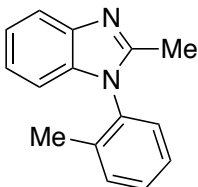


**6-Methoxy-1-phenyl-1H-benzo[d]imidazole (Table 2, entry 4) [69445-55-2].** The title compound was synthesized according to the General Procedure from 5-methoxybenzimidazole (148 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography (1%→5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 20%→35% EtOAc/hexanes). Yellow solid. First run: 182 mg (81%, 6-methoxy-1-phenyl-1H-benzo[d]imidazole / 5-methoxy-1-phenyl-1H-benzo[d]imidazole = 1.0:1). Second run: 190 mg (85%, 6-methoxy-1-phenyl-1H-benzo[d]imidazole / 5-methoxy-1-phenyl-1H-benzo[d]imidazole = 1.1:1).



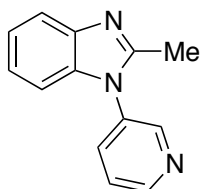
**2-Methyl-1-phenyl-1H-benzo[d]imidazole (Table 2, entry 5) [1484-39-5].** The title compound was synthesized according to the General Procedure from 2-methylbenzimidazole (132 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography on silica gel (1%→5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 20%→35% ethyl acetate/hexanes). Yellow solid. First run: 169 mg (81%). Second run: 175 mg (84%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.



**2-Methyl-1-(o-tolyl)-1H-benzo[d]imidazole (Table 2, entry 6) [68874-09-9].** The title compound was synthesized according to the General Procedure from 2-methylbenzimidazole (132 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 2-iodotoluene (305 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica (10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography (1% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 20% EtOAc/hexanes). Yellow solid. First run: 170 mg (76%). Second run: 166 mg (75%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.



**2-Methyl-1-(pyridin-3-yl)-1H-benzodimidazole (Table 2, entry 7).** The title compound was synthesized according to the General Procedure from 2-methylbenzimidazole (132 mg, 1.00 mmol), LiO*t*-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 3-iodopyridine (287 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica (10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography on silica gel (2% MeOH/CH<sub>2</sub>Cl<sub>2</sub>). Yellow solid. First run: 139 mg (66%). Second run: 140 mg (67%).

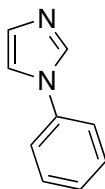
Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.78 (d, 1H, *J* = 3.2 Hz), 8.70 (s, 1H), 7.45 (apparent t, 2H, *J* = 3.2 Hz), 7.55 (dd, 1H, *J* = 7.9, 5.1 Hz), 7.28 (t, 1H, *J* = 7.8 Hz), 7.21 (t, 1H, *J* = 7.8 Hz), 7.10 (d, 1H, *J* = 8.0 Hz), 2.52 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 151.4, 150.1, 148.3, 142.8, 136.3, 134.6, 133.0, 124.5, 123.2, 123.0, 119.4, 109.6, 14.6.

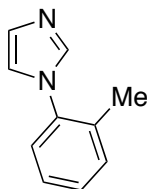
FT-IR (neat) 3391, 3053, 2927, 2851, 1615, 1587, 1575, 1524, 1486, 1456, 1427, 1393, 1372, 1314, 1287, 1249, 1187, 1149, 1105, 1050, 1029, 1015, 999, 929, 878, 810, 765, 745, 712 cm<sup>-1</sup>.

MS (ESI) *m/z* (M<sup>+</sup>+H) calcd for C<sub>13</sub>H<sub>12</sub>N<sub>3</sub>: 210, found: 210.

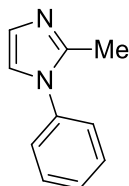


**1-Phenyl-1H-imidazole (Table 3, entry 1) [7164-98-9].** The title compound was synthesized according to the General Procedure from imidazole (102 mg, 1.50 mmol), LiO*t*-Bu (168 mg, 2.10 mmol), CuI (28.6 mg, 0.15 mmol), and iodobenzene (428 mg, 2.10 mmol), except that a mixture of *t*-BuOH (1.5 mL) and CH<sub>3</sub>CN (4.5 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. The reaction mixture was filtered through a plug of silica gel (1% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography (1% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 40%→50% EtOAc/hexanes). Pale-yellow oil. First run: 150 mg (69%). Second run: 148 mg (68%).

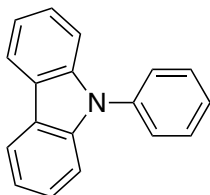




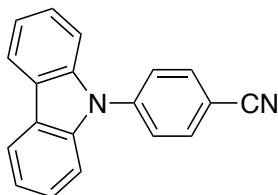
**1-(*o*-Tolyl)-1*H*-imidazole (Table 3, entry 2) [25371-93-1].** The title compound was synthesized according to the General Procedure from imidazole (102 mg, 1.50 mmol), Li*O**t*-Bu (168 mg, 2.10 mmol), CuI (28.6 mg, 0.15 mmol), and 2-iodotoluene (458 mg, 2.10 mmol), except that a mixture of *t*-BuOH (1.5 mL) and CH<sub>3</sub>CN (4.5 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. The reaction mixture was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography (1%→3% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 30%→50% EtOAc/hexanes). Yellow oil. First run: 154 mg (65%). Second run: 161 mg (68%).



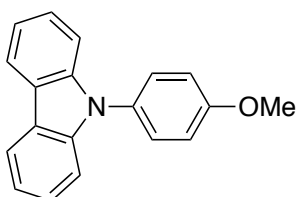
**2-Methyl-1-phenyl-1*H*-imidazole (Table 3, entry 3) [60053-07-8].** The title compound was synthesized according to the General Procedure from 2-methylimidazole (123 mg, 1.50 mmol), Li*O**t*-Bu (168 mg, 2.10 mmol), CuI (28.6 mg, 0.15 mmol), and iodobenzene (428 mg, 2.10 mmol). The reaction mixture was filtered through a plug of silica gel (10% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography (2% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 40%→50% EtOAc/hexanes). Yellow oil. First run: 106 mg (45%). Second run: 109 mg (46%).



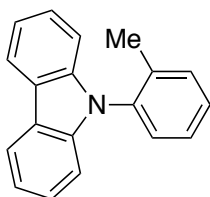
**9-Phenyl-9*H*-carbazole (Table 4, entry 1) [1150-62-5].** The title compound was synthesized according to the General Procedure from carbazole (167 mg, 1.00 mmol), Li*O**t*-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and iodobenzene (286 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). White solid. First run: 212 mg (87%). Second run: 206 mg (85%).



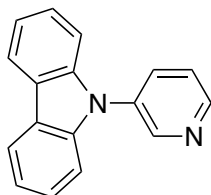
**4-(9H-Carbazol-9-yl)benzonitrile (Table 4, entry 2) [57103-17-0].** The title compound was synthesized according to the General Procedure from carbazole (167 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-iodobenzonitrile (321 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→2% Et<sub>2</sub>O/hexanes). Yellow solid. First run: 203 mg (76%). Second run: 209 mg (78%).



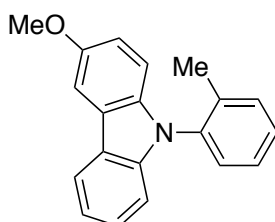
**9-(4-Methoxyphenyl)-9H-carbazole (Table 4, entry 3) [19264-74-5].** The title compound was synthesized according to the General Procedure from carbazole (167 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-iodoanisole (328 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). White solid. First run: 215 mg (79%). Second run: 196 mg (72%).



**9-(o-Tolyl)-9H-carbazole (Table 4, entry 4) [19155-50-1].** The title compound was synthesized according to the General Procedure from carbazole (167 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 2-iodotoluene (305 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). White solid. First run: 213 mg (83%). Second run: 204 mg (79%).



**9-(Pyridin-3-yl)-9H-carbazole (Table 4, entry 5) [168127-56-8].** The title compound was synthesized according to the General Procedure from carbazole (167 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 3-iodopyridine (287 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (50% EtOAc/hexanes) and purified by column chromatography on silica gel (10% EtOAc/hexanes). White solid. First run: 154 mg (63%). Second run: 166 mg (68%).



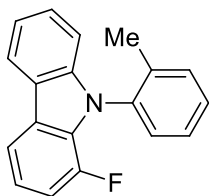
**3-Methoxy-9-(o-tolyl)-9H-carbazole (Table 4, entry 6).** The title compound was synthesized according to the General Procedure from 3-methoxycarbazole (100 mg, 0.51 mmol), LiOt-Bu (56.8 mg, 0.71 mmol), CuI (9.7 mg, 0.051 mmol), and 2-iodotoluene (155 mg, 0.71 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). Colorless oil. First run: 110 mg (76%). Second run: 110 mg (76%).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.12 (d, 1H, *J* = 7.5 Hz), 7.65 (d, 1H, *J* = 2.0 Hz), 7.50–7.33 (m, 5H), 7.27–7.23 (m, 1H), 7.06–7.01 (m, 2H), 6.96 (d, 1H, *J* = 9.0 Hz), 3.96 (s, 3H), 1.97 (s, 3H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 154.2, 141.8, 137.5, 136.4, 136.3, 131.6, 129.4, 128.8, 127.4, 126.0, 123.5, 123.0, 120.4, 119.2, 115.1, 110.7, 110.0, 103.4, 56.3, 17.7.

FT-IR (neat) 3050, 2993, 2932, 2830, 1627, 1600, 1580, 1498, 1485, 1462, 1438, 1381, 1359, 1329, 1285, 1254, 1236, 1206, 1179, 1167, 1149, 1119, 1098, 1035, 943, 912, 860, 847, 806, 764, 746, 720 cm<sup>-1</sup>.

MS (ESI) *m/z* (M<sup>+</sup>) calcd for C<sub>20</sub>H<sub>17</sub>NO: 287, found: 287.



**1-Fluoro-9-(o-tolyl)-9H-carbazole (Table 4, entry 7).** The title compound was synthesized according to the General Procedure from 1-fluorocarbazole (100 mg, 0.54 mmol), LiOt-Bu (60.5 mg, 0.76 mmol), CuI (10.3 mg, 0.054 mmol), and 2-iodotoluene (165 mg, 0.76 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column

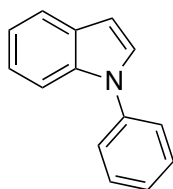
chromatography on silica gel (hexanes→1% Et<sub>2</sub>O/hexanes). Pale-yellow oil. First run: 111 mg (75%). Second run: 110 mg (74%).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.14 (d, 1H, *J* = 8.0 Hz), 7.93 (d, 1H, *J* = 7.5 Hz), 7.47–7.34 (m, 5H), 7.30 (t, 1H, *J* = 7.5 Hz), 7.18 (td, 1H, *J* = 8.0, 4.0 Hz), 7.10 (dd, 1H, *J* = 12.0, 7.5 Hz), 7.01 (d, 1H, *J* = 8.5 Hz), 2.01 (s, 3H).

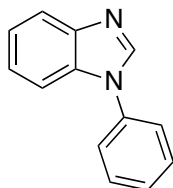
<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 149.6 (d, *J*<sub>CF</sub> = 245.8 Hz), 141.9, 137.4, 137.3, 131.1, 129.0, 128.9, 128.7 (d, *J*<sub>CF</sub> = 8.7 Hz), 127.0 (d, *J*<sub>CF</sub> = 4.8 Hz), 126.9, 126.7, 123.0 (d, *J*<sub>CF</sub> = 2.9 Hz), 120.5, 120.2, 119.8 (d, *J*<sub>CF</sub> = 6.8 Hz), 116.2 (d, *J*<sub>CF</sub> = 3.9 Hz), 112.1 (d, *J*<sub>CF</sub> = 17.3 Hz), 110.3, 17.5.

FT-IR (neat) 3058, 2955, 2924, 1635, 1602, 1577, 1498, 1455, 1435, 1381, 1354, 1339, 1316, 1290, 1248, 1226, 1184, 1154, 1116, 1081, 1053, 1014, 951, 925, 884, 787, 745, 733, 722 cm<sup>-1</sup>.

MS (EI) *m/z* (*M*<sup>+</sup>) calcd for C<sub>19</sub>H<sub>14</sub>FN: 275, found: 275.

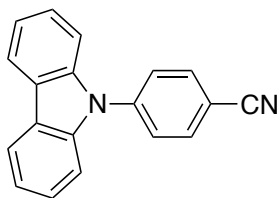


**1-Phenyl-1H-indole (Table 5, entry 1) [16096-33-6].** The title compound was synthesized according to the General Procedure from indole (117 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and bromobenzene (220 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (10% EtOAc/hexanes) and purified by column chromatography (hexanes). Pale-yellow oil. First run: 115 mg (60% yield). Second run: 122 mg (63% yield).

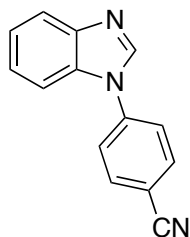


**1-Phenyl-1H-benzimidazole (Table 5, entry 2) [2622-60-8].** The title compound was synthesized according to the General Procedure from benzimidazole (118 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and bromobenzene (220 mg, 1.40 mmol), except that a mixture of *t*-BuOH (1.0 mL) and CH<sub>3</sub>CN (3.0 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. Reaction time: 48 h. The reaction mixture was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by normal-phase column chromatography on silica gel (0.75% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) followed by reverse-phase column chromatography on C-18 silica gel (10%→100% CH<sub>3</sub>CN/water). Yellow oil. First run: 118 mg (61%). Second run: 125 mg (64%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.



**4-(9H-Carbazol-9-yl)benzonitrile (Table 5, entry 3) [57103-17-0].** The title compound was synthesized according to the General Procedure from carbazole (167 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-chlorobenzonitrile (193 mg, 1.40 mmol). The reaction mixture was filtered through a plug of silica gel (20% EtOAc/hexanes) and purified by normal-phase column chromatography on silica gel (hexanes→2% Et<sub>2</sub>O/hexanes) followed by reverse-phase column chromatography on C-18 silica gel (10%→100% CH<sub>3</sub>CN/water). Yellow solid. First run: 192 mg (72%). Second run: 194 mg (72%).



**4-(1H-Benzo[d]imidazol-1-yl)benzonitrile (Table 5, entry 4) [25699-95-0].** The title compound was synthesized according to the General Procedure from benzimidazole (118 mg, 1.00 mmol), LiOt-Bu (112 mg, 1.40 mmol), CuI (19.0 mg, 0.10 mmol), and 4-chlorobenzonitrile (193 mg, 1.40 mmol), except that a mixture of *t*-BuOH (1.0 mL) and CH<sub>3</sub>CN (3.0 mL) was used as the solvent (*t*-BuOH and CH<sub>3</sub>CN were added in turn via syringe), due to the poor solubility of the heterocycle in neat CH<sub>3</sub>CN. Reaction time: 48 h. The product was filtered through a plug of silica gel (5% MeOH/CH<sub>2</sub>Cl<sub>2</sub>) and purified by column chromatography on silica gel (1% MeOH/CH<sub>2</sub>Cl<sub>2</sub>, then 40%→55% EtOAc/hexanes). Yellow solid. First run: 131 mg (60%). Second run: 135 mg (62%).

Note: The reaction mixture was stirred until it became homogeneous, and then it was immediately transferred to the photoreactor before it turned to a white heterogeneous mixture. The reaction proceeded in poor yield when the white precipitate formed.

### III. Nucleophile Competition Experiments (Table 6)

**Procedure.** Both of the nitrogen heterocycles (0.40 mmol each) and LiOt-Bu (32.0 mg, 0.40 mmol) were added to an oven-dried 10-mL quartz test tube that contained a stir bar. Next, the quartz tube was transferred to a glovebox, where *t*-BuOH (0.40 mL) and CH<sub>3</sub>CN (0.40 mL) were added. The reaction mixture was stirred for 3 min, and then a solution of CuI in CH<sub>3</sub>CN (0.80 mL, 0.050 M) was added, followed by iodobenzene (114 mg, 0.56 mmol) and dibenzyl ether (79.3 mg, 0.40 mmol; internal standard). The quartz test tube was capped with a rubber septum and transferred to a Luzchem LZC-4V photoreactor, where it was irradiated at 254 nm (adequate stirring is important). The ratio of products was determined by GC analysis after 2 h.

Note: Reactions with benzimidazole were quickly transferred to the photoreactor before they became heterogeneous.

### IV. Electrophile Competition Experiments (eq 2)

**Procedure.** Indole (46.9 mg, 0.40 mmol) and LiOt-Bu (44.8 mg, 0.56 mmol) were added to an oven-dried 10-mL quartz test tube that contained a stir bar. Next, the quartz tube was transferred to a glovebox, where CH<sub>3</sub>CN (0.80 mL), 1-ethyl-4-iodobenzene (130 mg, 0.56 mmol), and the aryl bromide or chloride (0.56 mmol) were added in turn. The reaction mixture was stirred for 3 min, and then a solution of CuI in CH<sub>3</sub>CN (0.80 mL, 0.050 M) was added, followed by dibenzyl ether (79.3 mg, 0.40 mmol; internal standard). The quartz test tube was capped with a rubber septum and transferred to a Luzchem LZC-4V photoreactor, where it was irradiated at 254 nm (adequate stirring is important). The ratio of products was determined by GC analysis after 1 h.

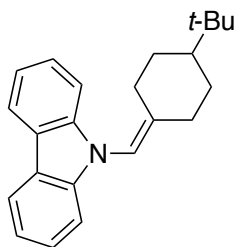
### V. Functional-Group Tolerance Experiments (Table 7)

**Procedure.** Indole (46.9 mg, 0.40 mmol) and LiOt-Bu (44.8 mg, 0.56 mmol) were added to an oven-dried 10-mL quartz test tube that contained a stir bar. Next, the quartz test tube was transferred to a glovebox, where CH<sub>3</sub>CN (0.80 mL) and the additive (0.40 mmol) were added in turn. The reaction mixture was stirred for 3 min, and then a solution of CuI in CH<sub>3</sub>CN (0.80 mL, 0.050 M) was added, followed by iodobenzene (114 mg, 0.56 mmol) and dibenzyl ether (79.3 mg, 0.40 mmol; internal standard). The reaction mixture was stirred for 3 min, and then an aliquot was taken for a *t* = 0 time point. Next, the quartz test tube was capped with a rubber septum, the joint was wrapped with electrical tape, and the quartz tube was transferred to a Luzchem LZC-4V photoreactor, where it was irradiated at 254 nm for 24 h (adequate stirring is important). The yield of product and the percent recovery of the additive were determined by GC analysis.

## VI. Photoinduced, Copper-Catalyzed N-Alkenylations/Alkynylations (Table 8)

**General Procedure.** The nitrogen heterocycle (0.50 mmol) and LiOt-Bu (83.0 mg, 1.04 mmol) were added to an oven-dried 10-mL quartz test tube that contained a stir bar. The quartz tube was fitted with a rubber septum, the joint was wrapped with electrical tape, and the quartz tube was evacuated and backfilled with nitrogen (3 cycles). Then, CH<sub>3</sub>CN (4.0 mL) was added, and the mixture was stirred for 10 min. Next, a solution of CuI in CH<sub>3</sub>CN (500 μL, 0.10 M) was added via syringe, and the mixture was stirred for 10 min. A 4-mL oven-dried vial was charged with the alkenyl iodide (0.85 mmol), closed with a septum cap, and evacuated and backfilled with nitrogen (3 cycles). The alkenyl iodide was transferred to the quartz tube via syringe. The vial was rinsed with CH<sub>3</sub>CN (0.50 mL), and the washing was transferred to the quartz tube. The test tube was detached from the nitrogen manifold, and the puncture holes in the septum were covered with vacuum grease. The mixture was stirred for 10 min, and then the test tube was transferred to a Luzchem LZC-4V photoreactor, where it was irradiated at 254 nm for 12 h (adequate stirring is important). Next, the reaction mixture was passed through a plug of silica gel (10% EtOAc/hexanes; monitored by TLC), the solvent was removed, and the residue was purified by column chromatography.

Note: For the N-alkynylation process (Table 8, entry 5), the same procedure was employed, except that the reaction mixture was irradiated for 24 h.



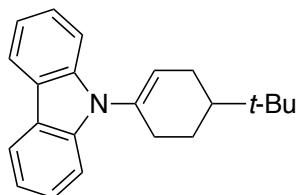
**9-((4-(*tert*-Butyl)cyclohexylidene)methyl)-9H-carbazole (Table 8, entry 1).** The title compound was synthesized according to the General Procedure from carbazole (84 mg, 0.50 mmol) and 1-(iodomethylidene)-4-*tert*-butyl-cyclohexane (237 mg, 0.85 mmol). The product was purified by column chromatography (hexanes). White solid. First run: 138 mg (87%). Second run: 131 mg (83%).

<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.11 (d, 2H, *J* = 7.7 Hz), 7.46 (t, 2H, *J* = 7.7 Hz), 7.40–7.21 (m, 4H), 6.45 (s, 1H), 2.75–2.62 (m, 1H), 2.36–2.25 (m, 2H), 2.11–2.03 (m, 1H), 1.89–1.70 (m, 2H), 1.35–1.20 (m, 2H), 1.08–0.96 (m, 1H), 0.89 (s, 9H).

<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 144.6, 140.9, 125.6, 122.9, 120.1, 119.3, 114.1, 110.0, 48.1, 33.2, 32.5, 29.2, 28.9, 28.1, 27.6.

FT-IR (neat) 3054, 2986, 2305, 1479, 1457, 1422, 1265, 896 cm<sup>-1</sup>.

MS (EI) *m/z* (M<sup>+</sup>) calcd for C<sub>23</sub>H<sub>27</sub>N: 317, found: 317.



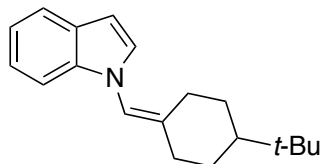
**9-(4-(*tert*-Butyl)cyclohex-1-en-1-yl)-9*H*-carbazole (Table 8, entry 2).** The title compound was synthesized according to the General Procedure from carbazole (84 mg, 0.50 mmol) and 4-*tert*-butyl-1-iodo-1-cyclohexene (225 mg, 0.85 mmol). The product was purified by column chromatography (hexanes). White solid. First run: 114 mg (75%). Second run: 114 mg (75%).

$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.11 (d, 2H,  $J = 7.7$  Hz), 7.46–7.36 (m, 4H), 7.28–7.19 (m, 2H), 6.10–6.06 (m, 1H), 2.50–2.36 (m, 3H), 2.24–2.12 (m, 1H), 2.10–2.00 (m, 1H), 1.65–1.50 (m, 2H), 1.00 (s, 9H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.4, 134.3, 128.3, 125.6, 122.9, 120.2, 119.1, 109.8, 44.0, 32.4, 28.5, 27.3, 26.6, 24.4.

FT-IR (neat) 3054, 2987, 2305, 1452, 1422, 1265, 896  $\text{cm}^{-1}$ .

MS (EI)  $m/z$  ( $\text{M}^+$ ) calcd for  $\text{C}_{22}\text{H}_{25}\text{N}$ : 303, found: 303.



**1-((4-(*tert*-Butyl)cyclohexylidene)methyl)-1*H*-indole (Table 8, entry 3).** The title compound was synthesized according to the General Procedure from indole (59 mg, 0.50 mmol) and 1-(iodomethylidene)-4-*tert*-butyl-cyclohexane (237 mg, 0.85 mmol). The product was purified by column chromatography (hexanes). White solid. First run: 100 mg (75%). Second run: 99 mg (74%).

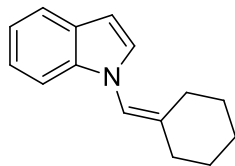
$^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d, 1H,  $J = 7.8$  Hz), 7.32 (d, 1H,  $J = 8.3$  Hz), 7.27–7.21 (m, 1H), 7.18–7.12 (m, 1H), 7.11–7.08 (m, 1H), 6.58–6.53 (m, 2H), 2.66–2.48 (m, 2H), 2.26–2.15 (m, 1H), 2.05–1.97 (m, 1H), 1.91–1.79 (m, 2H), 1.31–1.15 (m, 2H), 1.10–0.97 (m, 1H), 0.89 (s, 9H).

$^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  139.7, 136.6, 128.5, 128.1, 121.7, 120.7, 119.7, 116.4, 110.3, 101.8, 48.1, 33.4, 32.5, 29.0, 28.4, 28.2, 27.6.

FT-IR (neat) 3054, 2986, 2305, 1422, 1265, 896  $\text{cm}^{-1}$ .

MS (EI)  $m/z$  ( $\text{M}^+$ ) calcd for  $\text{C}_{19}\text{H}_{25}\text{N}$ : 267, found: 267.





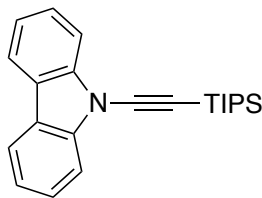
**1-(Cyclohexylidene)methyl-1H-indole (Table 8, entry 4).** The title compound was synthesized according to the General Procedure from indole (59 mg, 0.50 mmol) and bromomethylenecyclohexane (155 mg, 0.85 mmol). Reaction time: 48 h. The product was purified by column chromatography (hexanes). Colorless oil. First run: 58 mg (55%). Second run: 60 mg (57%).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 (d, 1H,  $J = 7.8$  Hz), 7.34 (d, 1H,  $J = 8.2$  Hz), 7.29–7.23 (m, 1H), 7.20–7.15 (m, 1H), 7.13–7.09 (m, 1H), 6.61–6.57 (m, 2H), 2.38–2.32 (m, 2H), 2.24–2.18 (m, 2H), 1.78–1.70 (m, 2H), 1.70–1.63 (m, 2H), 1.60–1.52 (m, 2H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.1, 136.6, 128.5, 128.1, 121.7, 120.7, 119.7, 116.8, 110.3, 101.8, 33.4, 28.5, 28.3, 27.4, 26.5.

FT-IR (neat) 2934, 2956, 2253, 1674, 1511, 1475, 1462, 1377, 1319, 1234, 1088, 907  $\text{cm}^{-1}$ .

MS (EI)  $m/z$  ( $\text{M}^+$ ) calcd for  $\text{C}_{15}\text{H}_{17}\text{N}$ : 211, found: 211.



**9-((Triisopropylsilyl)ethynyl)-9H-carbazole (Table 8, entry 5).** The title compound was synthesized according to the General Procedure from carbazole (84 mg, 0.50 mmol) and 2-bromo-1-triisopropylsilyl acetylene (222 mg, 0.85 mmol). The product was purified by column chromatography (hexanes). White solid. First run: 109 mg (63%). Second run: 107 mg (62%).

$^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d, 2H,  $J = 7.7$  Hz), 7.64 (d, 2H,  $J = 8.1$  Hz), 7.53 (t, 2H,  $J = 7.7$  Hz), 7.34 (t, 2H,  $J = 7.7$  Hz), 1.31–1.14 (m, 21H).

$^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  140.5, 126.7, 123.3, 122.0, 120.3, 111.4, 92.6, 72.8, 18.8, 11.4.

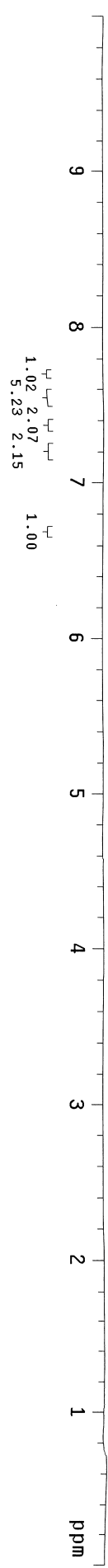
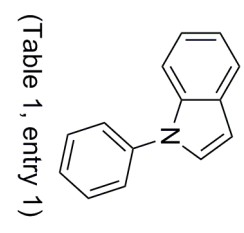
FT-IR (neat) 3054, 2986, 2305, 2178, 1422, 1265, 896  $\text{cm}^{-1}$ .

MS (EI)  $m/z$  ( $\text{M}^+$ ) calcd for  $\text{C}_{23}\text{H}_{29}\text{NSi}$ : 347, found: 347.

# VII. 1H NMR Spectra

JC92658 CDC13  
 exp20 PROTON

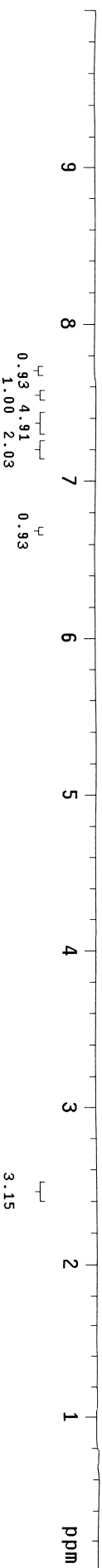
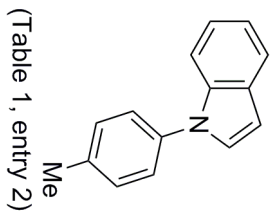
SAMPLE PRESATURATION  
 date Mar 6 2013 satmode n  
 solvent cdc13 wet SPECIAL n  
 file /indy/jwcho1/~ temp not used  
 ynmr/sys/data/JC926~ gain 30  
 5B\_1H\_13C\_CDC13/PR~ OTON01.fid spin 20  
 ACQUISITION hst 0.008  
 8000.0 pw90 9.700  
 3.000 alfa 10.000  
 48000  
 np not used  
 fb not used  
 bs 32 in n  
 d1 2.000 dp y  
 nt 16 hs nm  
 ct 16  
 TRANSMITTER H1  
 tn 1b fn 0.20  
 sfra 499.708 not used  
 tof 499.7 sp DISPLAY  
 tpwr 61 wp -0.1  
 pw 4.850 rfp 4996.8  
 DECOUPLER C13 rfp 4630.1  
 dn 0 tp 3627.9  
 dof 0 1p 25.2  
 dm mnm PLOT -59.8  
 decave g WC 250  
 dpwr 35 SC D  
 dmf 32258 VS 80  
 ai cdc ph 6



DZ-04-292-1-Purified

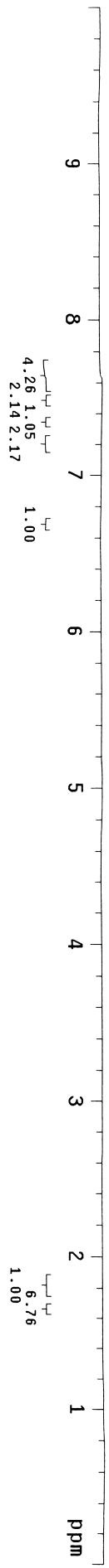
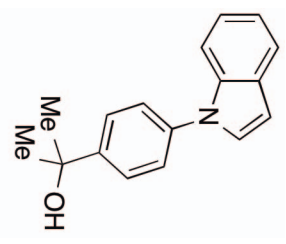
exp20 PROTON

SAMPLE PRESATURATION  
date Apr 2 2013 satmode n  
solvent cdc13 wet n  
file /indy/dz1egle~ SPECIAL  
r/vnmrSYS/data/DZ-~ temp not used  
04-292-1-Purified/~ gain 32  
PROTON01.fid hst 20  
ACQUISITION spm 20  
SW 8000.0 pw90 9.900  
at 3.000 alfa 10.000  
np 48000  
fb not used i1 n  
bs 32 in n  
d1 1.000 dp nm  
nt 16 hs y  
ct 16  
TRANSMITTER 1b 0.20  
tn H1 fn not used  
sfreq 499.708 DISPLAY  
tof 499.7 SP -0.1  
tpwr 61 wp 4996.8  
pw 4.950 rffl 4630.1  
DECOUPLER rfp 3627.9  
dn C13 rp 20.6  
dm 0 1p -73.7  
dm nm PLOT  
decwave W40\_autox7~ WC 250  
991 SC 0  
41 VS 40  
32258 th 25  
ai cdc ph



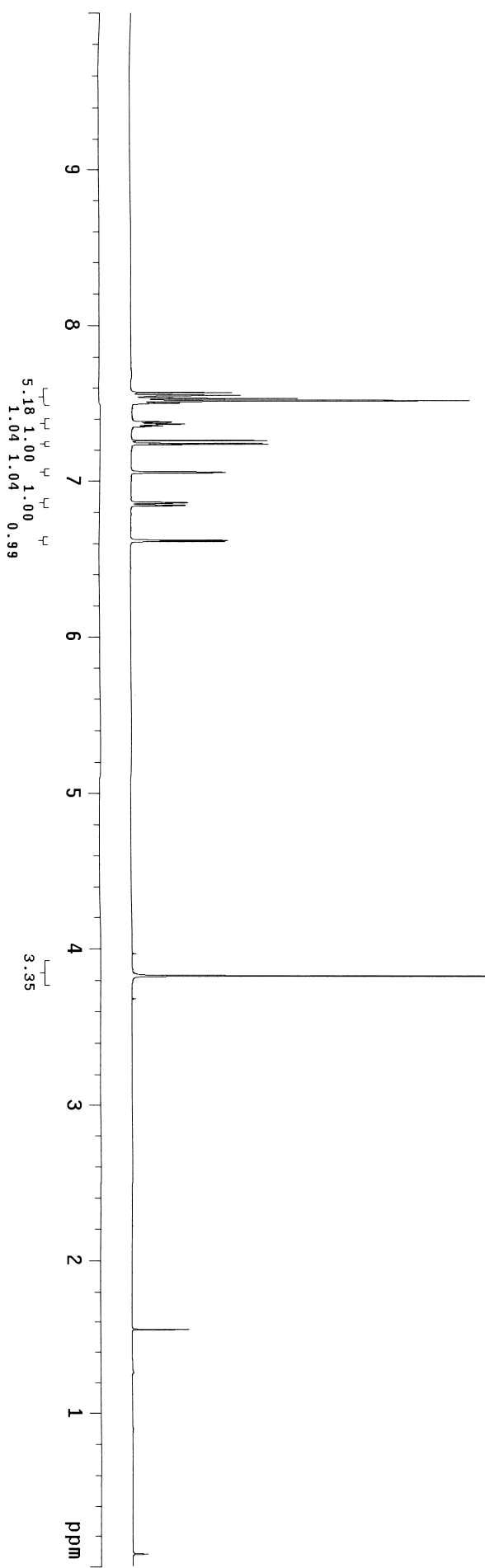
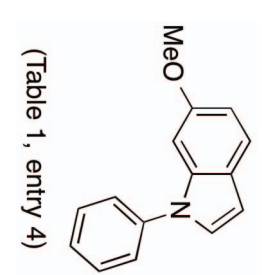
02-05-082-1-Purified  
 exp25 PROTON

SAMPLE PRESATURATION  
 date Jun 26 2013 satmode n  
 solvent cdc13 wet n  
 file /indy/dzlegle~ SPECIAL  
 r/vnmr/sys/data/DZ-~ not used  
 05-082-1-Purified/~ gain 32  
 PROTON01.fid spin 20  
 ACQUISITION hst 0.008  
 SW 8000.0 pw90 9.900  
 at 3.000 alfa 10.000  
 np 48000 FLAGS  
 fb not used i1 n  
 bs 32 in n  
 d1 1.000 dp y  
 nt 32 hs n  
 ct 32  
 TRANSMITTER 1b 1b  
 tn H1 fn 0.20  
 srfq 499.698 not used  
 tof 499.7 DISPLAY  
 tpwr 61 wp 4996.8  
 pw 4.950 rffl 4632.1  
 DECOUPLER rfp 3627.8  
 dn C13 fp -81.8  
 dof 0 lp -72.7  
 dm nnn PLOT  
 dm decwawe w40\_autox7~ 250  
 991 SC 0  
 dpwr 41 VS 11  
 dmf 32258 th 16  
 ai cdc ph



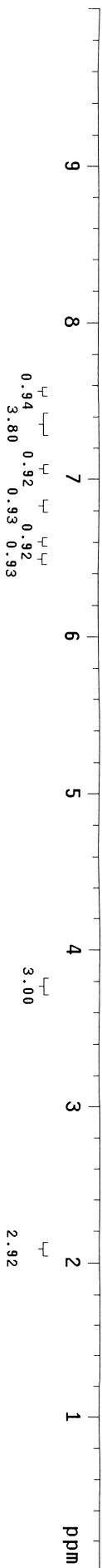
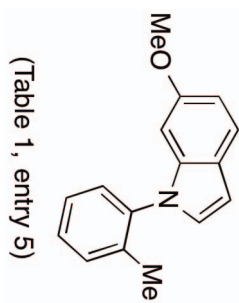
DZ-04-200-2-Purified  
 exp20 PROTON

date	Mar 21 2013	SAMPLE	PRESATURATION	n
solvent	cdcl3	wet	satmode	n
file	/indy/dziegle~	SPECIAL		
r/vmrsys/data/DZ-~		not used		
04-200-2-Purified/~		temp	30	
PROTON02.fid		gain	20	
ACQUISITION		spin	9.700	
8000.0		hst	0.008	
3.000		pw90	10.000	
at	48000	alpha		
np	not used	FLAGS		
fb	1.000	il	n	
bs	1.000	in	n	
dl	16	dp	y	
nt	16	hs	nn	
ct	16	PROCESSING		
tn	H1	tb	0.20	
sfreq	499.708	fn	not used	
tof	499.7	DISPLAY		
tpwr	61	sp	-0.1	
pw	4.850	wp	4996.8	
DECOUPLER	C13	rfl	4630.1	
dn	0	rffp	3627.9	
dof	0	rp	42.6	
dm	nmh	tp	-72.6	
decwawe	g	PLOT		
dpwr	35	WC	250	
dmf	32258	SC	0	
		VS	48	
		th	13	
		ai	cdc	
			ph	



JC9267 1H CDC13  
exp20 PROTON

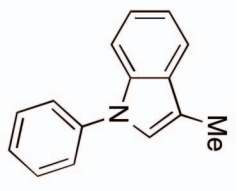
SAMPLE PRESATURATION  
date Mar 10 2013 satmode n  
solvent cdcl3 wet SPECIAL n  
file /indy/jwcho1/~ not used  
vnmrSYS/data/JC926~ gain 30  
7\_1H\_CDC13/PROTON0~ spin 20  
1.fid hst 0.008  
ACQUISITION pw90 9.700  
SW 8000.0 aifa 10.000  
at 3.000  
np 48000  
fb not used  
bs 32 in  
d1 2.000 dp  
nt 16 hs  
ct 16  
TRANSMITTER 1b  
tn H1 0.20  
sfra 499.708 fn not used  
tof 499.7 SP DISPLAY  
tpwr 61 wp 4996.8  
pw 4.850 rfp 4630.1  
DECOUPLER C13 rfp 3627.9  
dn 0 1p 42.0  
dm mn -73.7  
dof 0  
decave g WC 250  
dpwr 35 SC 0  
dmf 32258 VS 63  
ai cdc ph 50



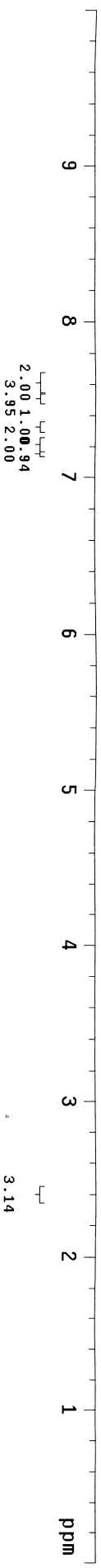
DZ-04-244-1-Purified

exp21 PROTON

date	Mar 16 2013	satmode	
solvent	cdcl3	wet	
file	/Indy/dz1egle-	SPECIAL	
r/vmrsys/data/DZ-		temp	not used
04-244-1-Purified/~		gain	30
PROTON01.fid		spin	20
ACQUISITION		hst	0.008
sw	8000.0	pw90	9.700
at	3.000	alfa	10.000
np	48000	FLAGS	
fb	not used	l1	n
bs	32	in	n
d1	1.000	dp	h
nt	16	hs	Y
ct	16	hs	nm
tn	TRANSMITTER	1b	0.20
sfrq	H1	fn	not used
tof	499.708	DISPLAY	
tpwr	499.7	SP	-0.1
pw	61	WD	4996.8
	4.850	rfl	4630.1
		rffp	3627.9
		fp	42.9
		1p	-72.2
dn	C13		
dof	0		
dm	nmn		
decwave	g	WC	250
dpwr	35	VS	0
dmf	32258	th	61
		at	41
			cdc ph



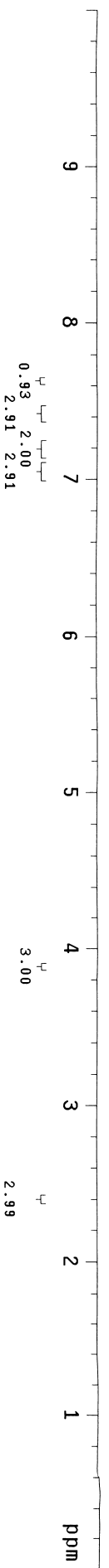
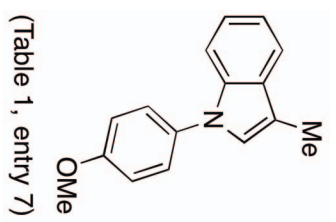
(Table 1, entry 6)



JC9299 CDC13

exp20 PROTON

SAMPLE PRESATURATION  
 date Mar 21 2013 satmode n  
 solvent cdc13 wet n  
 file /Indy/jwchoi/~ SPECIAL  
 ynmr-sys/data/JC929~ temp not used  
 9\_1H\_13C CDC13/PRO~ gain 30  
 TON01.fid hst 20  
 ACQUISITION spin 0.008  
 SW 8000.0 pw90 9.700  
 at 3.000 alfa 10.000  
 np 48000 FLAGS  
 fb not used i1 n  
 bs 32 in n  
 d1 2.000 dp n  
 nt 16 hs y  
 ct 16  
 TRANSMITTER 1b 0.20  
 tn H1 fn not used  
 sfrq 499.708 DISPLAY  
 tof 499.7 SP -0.1  
 tpwr 61 WP 4996.8  
 pw 4.850 rfp 4630.1  
 DECOUPLER C13 rfp 3627.9  
 dn 0 tp 42.3  
 dof 0 1p -72.5  
 dm nm PLOT  
 decwve g WC 250  
 dpwr 35 SC 0  
 dmf 32258 VS 22  
 th 50  
 ai cdc ph

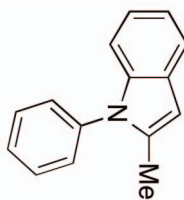




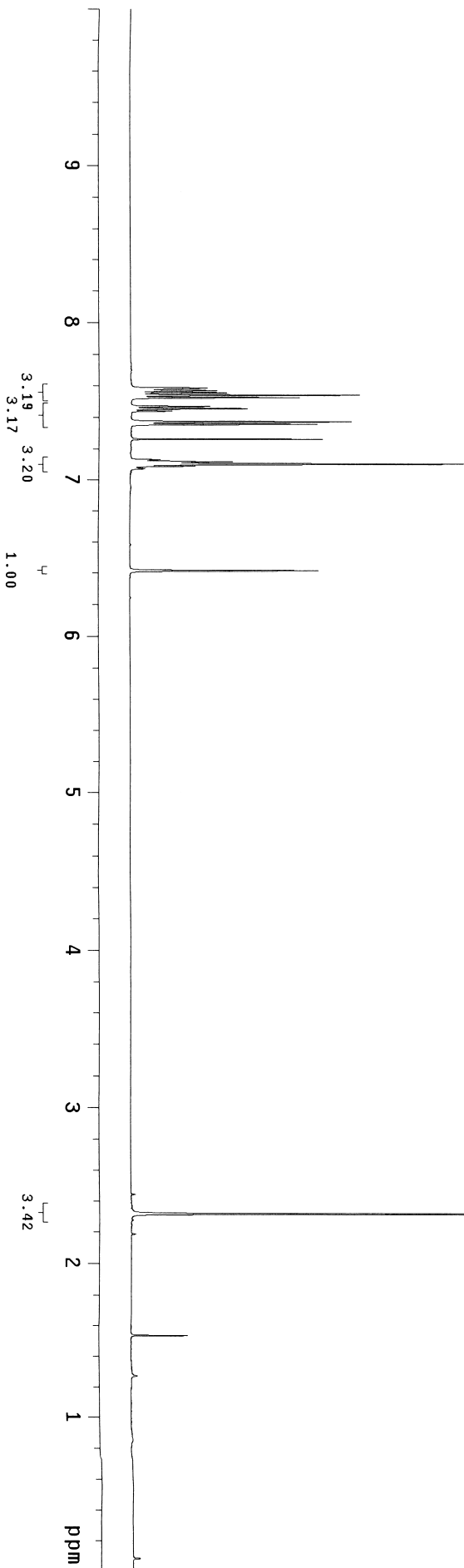
DZ-04-266-2-Purified

exp20 PROTON

SAMPLE PRESATURATION  
date Mar 20 2013 satmode n  
solvent cdcl3 wet  
file /indy/dz1egle~ SPECIAL  
r/vmrsys/data/DZ-~ temp not used  
04-266-2-Purified/~ gain 30  
PROTON01.fid spin 20  
ACQUISITION hst 0.008  
8000.0 pw90 9.700  
3.000 aifa 10.000  
48000 not used  
fb not used  
bs 32 in  
d1 1.000 dp  
nt 16 hs  
ct 16  
TRANSMITTER  
tn H1 1b  
sfra 499.708 fn not used  
tof 499.7 sp DISPLAY  
tpwr 61 wp 4996.8  
pw 4.850 rfp 4630.1  
DECOUPLER C13 rp 3627.9  
dn 0 1p -73.7  
dm nm  
dof 0  
decwave g WC 250  
dpwr 35 SC 0  
dmf 32258 VS 79  
ai cdc ph 25



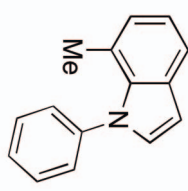
(Table 1, entry 8)



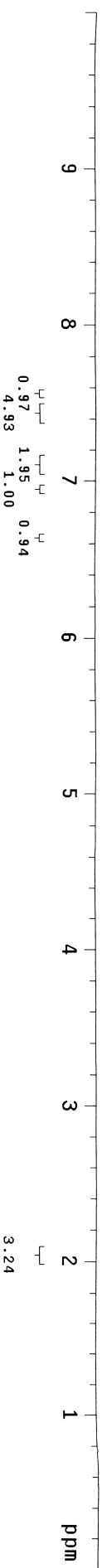
DZ-04-246-1-Purified

exp20 PROTON

SAMPLE		PRESATURATION	
date	Mar 16 2013	satmode	n
solvent	cdcl3	wet	n
file	/indy/dz1egle~	SPECIAL	
r/vmr	sys/data/DZ-~	temp	not used
04-246-1-Purified/~		gain	30
PROTON01.fid		spn	20
ACQUISITION		hst	0.008
sw	8000.0	pw90	9.700
at	3.000	alpha	10.000
np	48000	FLAGS	
fb	not used	i1	n
bs	32	in	n
d1	1.000	dp	y
nt	16	hs	nm
ct	16	hs	nm
TRANSMITTER		PROCESSING	
tn	H1	tb	0.20
sfrq	499.708	fn	not used
tof	499.7	SP	DISPLAY
tpwr	61	wp	-0.1
pw	4.850	rf1	4996.8
DECOUPLER		rfp	4630.1
dn	C13	fp	3627.9
dof	0	lp	41.9
dm	nm	lp	-70.9
decwave	9	PLOT	
dpwr	35	wc	250
dmf	32258	sc	0
		vs	85
		th	25
		ai	cdc ph

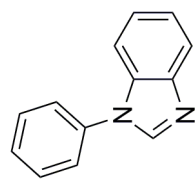


(Table 1, entry 9)

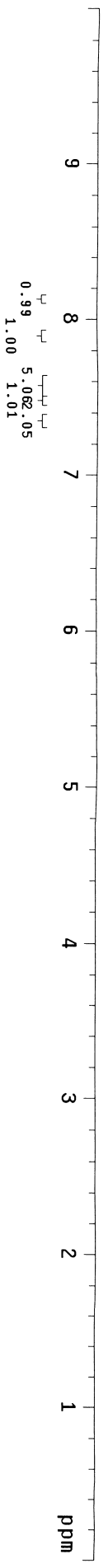


DZ-04-196-1-Purified  
 exp20 PROTON

SAMPLE PRESATURATION  
 date Mar 23 2013 satmode n  
 solvent cdc13 wet  
 file /indy/dz1egle- SPECIAL  
 r/vmr/sys/data/DZ-~ not used  
 04-196-1-Purified/~ gain 30  
 PROTON02.fid spin 20  
 ACQUISITION hst 0.008  
 SW 8000.0 pw90 9.700  
 at 3.000 alfa 10.000  
 np 48000  
 fb not used i1 n  
 bs 32 in n  
 d1 1.000 dp n  
 nt 16 hs y  
 ct 16  
 TRANSMITTER 1b 0.20  
 tn H1 fn not used  
 sfra 499.708 DISPLAY  
 tof 499.7 SP -0.1  
 tpwr 61 WP 4996.8  
 pw 4.850 rfp 4630.1  
 DECOUPLER C13 rfp 3627.9  
 dn 0 rfp 44.9  
 dof 0 1p -72.3  
 dm nm  
 decouple g WC 250  
 dpwr 35 SC 0  
 dmf 32258 VS 65  
 th 33  
 al cdc ph

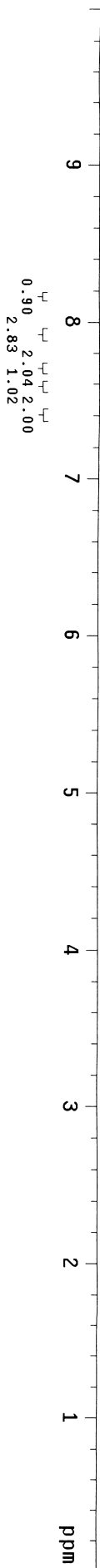
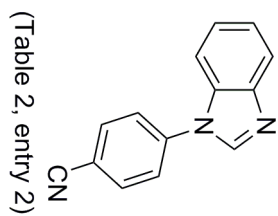


(Table 2, entry 1)



DZ-04-270-1-Purified  
 exp20 PROTON

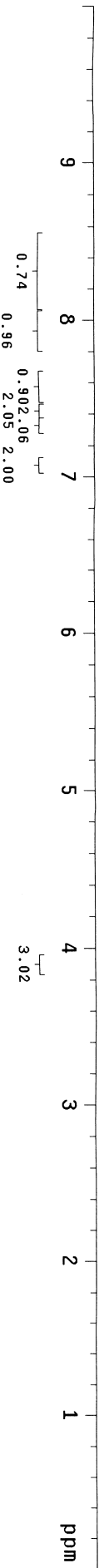
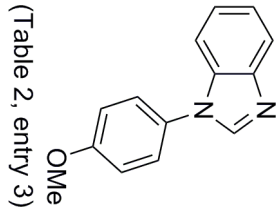
SAMPLE PRESATURATION  
 date Mar 26 2013 satmode n  
 solvent cdc13 wet n  
 file /indy/dzleg1e~ SPECIAL  
 r/vnmr/sys/data/DZ-~ temp not used  
 04-270-1-Purified/~ gain 32  
 PROTON01.fid spin 0  
 ACQUISITION hst 0.008  
 SW 8000.0 pw90 9.900  
 at 3.000 alfa 10.000  
 np 48000  
 fb not used  
 bs 32 in n  
 d1 1.000 dp n  
 nt 16 hs Y  
 ct 16 mm  
 PROCESSING  
 tn 1b 0.20  
 TRANSMITTER H1 fn not used  
 sfreq 499.708 DISPLAY  
 tof 499.7 SP -0.1  
 tpwr 61 WP 4996.8  
 pw 4.950 rfp 4629.9  
 DECOUPLER C13 rfp 3627.9  
 dn 0 tp 24.7  
 dof 0  
 dm mnh PLOT -72.1  
 decouple w40\_autocx7~ WC 250  
 991 SC 0  
 dpwr 41 VS 135  
 dmf 32258 th 4  
 ai cdc ph



DZ-04-268-3-Purified

exp20 PROTON

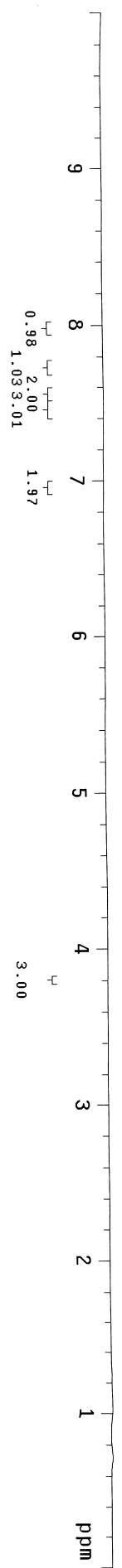
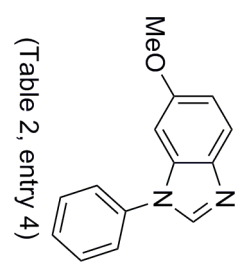
SAMPLE 2 2013  
date Apr 2 2013  
solvent cdc13  
file /Indy/dz1egle~  
r/vmrsys/data/DZ-~  
04-268-3-Purified/~  
PROTON02.fid  
ACQUISITION  
SW 8000.0  
at 3.000  
np 48000  
fb not used  
bs 32  
d1 1.000  
nt 16  
ct 16  
TRANSMITTER H1  
tn 499.708  
sfreq 499.7  
tof 61  
tpwr 4.950  
pw DECOUPLER C13  
dn 0  
dof 0  
dm nm  
decwave W40\_autox7~  
dpwr 991  
dmf 41  
32258  
satmode wet  
PRESATURATION n  
temp gain  
SPECIAL not used  
sp in  
hst 0.008  
pw90 9.900  
alpha 10.000  
FLAGS  
i1 n  
in n  
dp n  
hs y  
nm  
PROCESSING 0.20  
1b not used  
fn DISPLAY  
H1 -0.1  
sp 4996.8  
wp 4630.1  
rfp 3627.9  
f1 21.6  
p1 -79.4  
PLOT 250  
wc 0  
sc 25  
vs 13  
th  
ai cdc ph



JG9273A 1H 13C CDC13

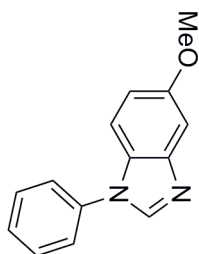
exp20 PROTON

SAMPLE	Mat 14 2013	PRESATURATION	n
date	Mat 14 2013	satmode	n
solvent	cdcl3	wet	n
file	/indy/jwcho1/~	SPECIAL	not used
vmrSYS/data/JG927~		temp	18
3A_1H_13C CDC13/PR~		gain	20
OTON02.fid		spin	0.008
ACQUISITION	8000.0	hst	9.700
pw90	10.000	alfta	10.000
at	3.000	alfta	10.000
mp	48000	FLAGS	n
fb	not used	il	n
bs	not used	in	n
d1	2.000	dp	Y
nt	16	hs	nn
ct	16	hs	nn
TRANSMITTER	H1	1b	0.20
tn	499.708	fn	not used
strq	499.7	sp	-0.1
tof	61	wp	4996.8
tpwr	4.850	rf1	4630.1
pw	DECOUPLER	rfp	3627.9
dn	C13	rp	47.4
dof	0	1p	-81.6
dm	nmn	PLOT	250
decwawe	9	wc	0
dpwr	35	sc	17
dmf	32258	vs	25
ai	cdc	ph	

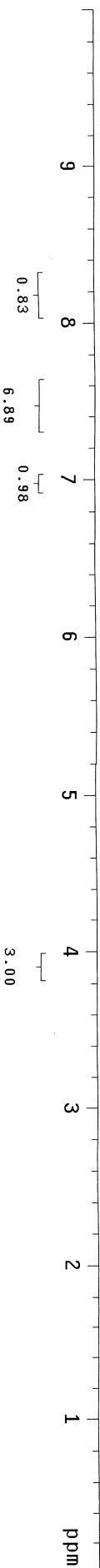


JC9273 Lower-spot 1H CDCl3  
 exp20 PROTON

SAMPLE	Mar 10 2013	PRESATURATION	n
date	Mar 10 2013	satmode	n
solvent	CdCl3	wet	n
file	/Indy/jwchoi/~	SPECIAL	not used
nmrsvs	/data/JC9273~	temp	30
3_Lower-spot_1H_CD~		gain	20
C13/PROTON02.fid		spin	28
ACQUISITION	8000.0	hst	9.700
sw	3.000	alpha	10.000
at	48000	flags	
np	not used		
fb	32		
bs	2.000		
d1	16		
nt	16		
ct			
TRANSMITTER	H1	PROCESSING	0.20
tn	499.708	fb	not used
sfrq	499.7	fn	DISPLAY
tof	61	wp	-0.1
tpwr	4.850	rfl	4996.8
pw		rfl	4630.1
DECOUPLER	C13	rffp	3627.9
dn	0	rp	38.0
dof	0	lp	-68.1
dm	nmn		
decwvave	9	WC	250
dpwr	35	SC	0
dmf	32258	VS	51
		th	13
		at	cdc ph

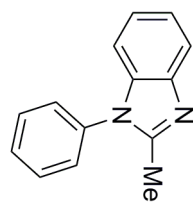


(Table 2, entry 4)

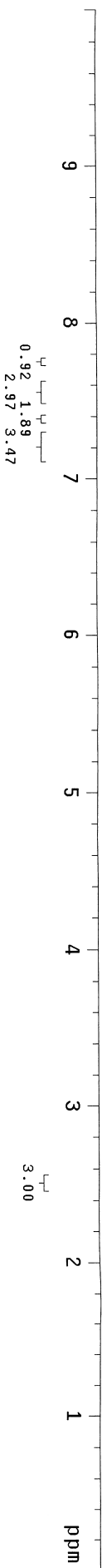


JC9291 1H CDC13  
exp20 PROTON

date	Mar 21 2013	PRESATURATION	satmode	n
solvent	cdcl3	WET	wet	n
file	/indy/jwchoi/~	SPECIAL		
ymmsys	/data/JC929~	temp	not used	30
1_1H_CDC13/PROTON0~	2.fid	gain	20	
ACQUISITION	8000.0	spin	20	
at	3.000	hst	0.008	
np	48000	pw90	9.700	
fb	not used	alfa	10.000	
bs	32	FLAGS		
d1	2.000	in	n	
nt	16	dp	n	
ct	16	hs	Y	
TRANSMITTER	H1	PROCESSING	0.20	
stfq	499.708	fn	not used	
tof	499.7	DISPLAY		
tpwr	61	sp	-0.1	
pw	4.850	wp	4996.8	
DECOUPLER	C13	rfl	4630.1	
dn	0	rffp	3627.9	
dof	0	fp	41.9	
dm	nmn	lp	-68.8	
decwave	9	WC	250	
dpwr	35	SC	0	
dmf	32258	VS	43	
		th	6	
		at	cdc	ph



(Table 2, entry 5)



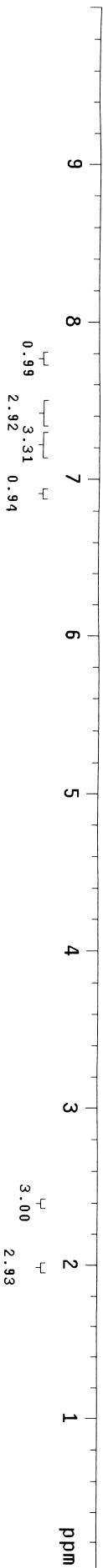
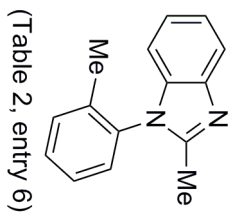


JC9303B CDC13  
 exp20 PROTON

SAMPLE Mar 25 2013  
 date Mar 25 2013  
 solvent cdc13  
 file /indy/jwchoi/~  
 vnmr/sv/data/JC9303/PR-  
 3B\_IH\_13C\_CDC13/PR-  
 OTON01.fid  
 ACQUISITION  
 SW 8000.0  
 AT 3.000  
 NP 48000  
 FB not used  
 BS 32  
 D1 2.000  
 NT 16  
 CT 16  
 TRANSMITTER H1  
 TN 499.708  
 SFRQ 499.7  
 TOF 61  
 TPWR 4.950  
 PW 4.950  
 DECOUPLER C13  
 DN 0  
 DOF 0  
 DM nnp  
 DECWAVE W40\_autox7~  
 DPWR 41  
 DMF 32258  
 AI cdc ph

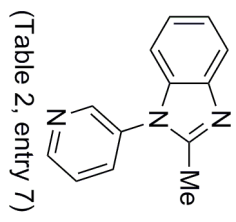
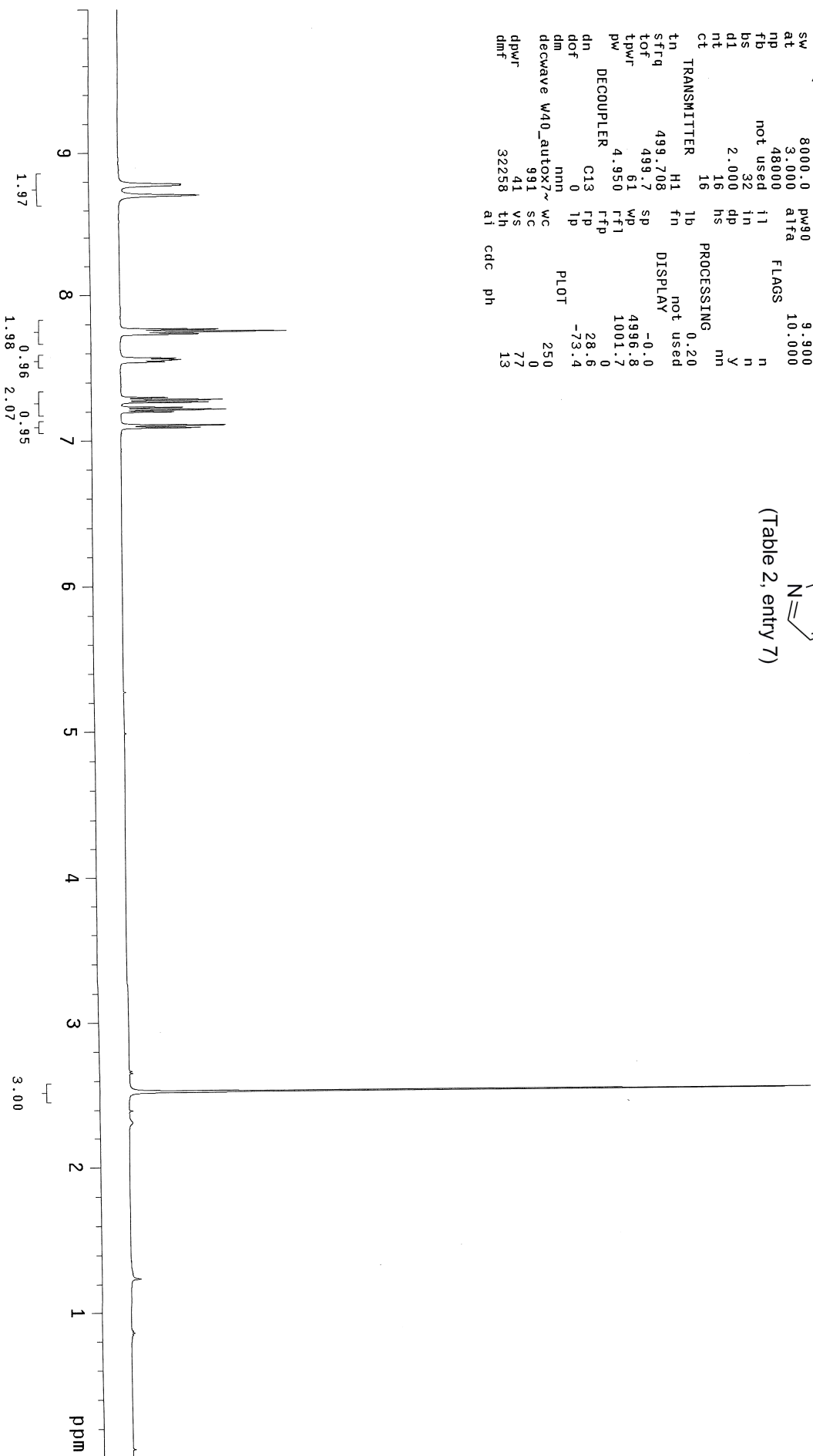
PRESATURATION satmode n  
 SPECIAL not used  
 gain 32  
 spin 0  
 hst 0.008  
 pw90 9.900  
 atfa 10.000  
 FLAGS  
 I1 n  
 IN n  
 DP n  
 HS y  
 nm

PROCESSING 0.20  
 1b not used  
 FN DISPLAY  
 SD -0.1  
 WP 4996.8  
 FT1 4629.9  
 RFP 3627.9  
 TP 28.6  
 LP -75.3  
 PLOT  
 WC 250  
 SC 0  
 VS 39  
 TH 50



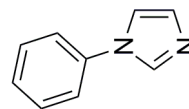
JC10009 CDC13  
exp20 PROTON

SAMPLE	date	Mar 29 2013	PRESATURATION	satmode	n
SOIvent	cdcl3		wet		n
file	/Indy/jwchoi/~		SPECIAL		not used
nmrsvs	/data/JC100~		temp	gain	20
09_1H_13C	CDC13/PR~		OTON01.fid	SPIN	0
ACQUISITION	8000.0		HST		0.008
at	3.000		PW90		9.900
np	48000		ALFA		10.000
fb	not used		FLAGS		n
bs	not used				n
dl	32				Y
nt	2.000				nn
ct	16		PROCESSING		0.20
tn	TRANSMITTER	H1	fb		not used
stfq	499.708		fn		DISPLAY
tof	499.7		sp		-0.0
tpwr	61		wp		4996.8
pw	4.950		FFI		1001.7
DECOUPLER	C13		FFP		0
dn	0		FP		28.6
dof	0		TP		-73.4
dm	nmn		PLOT		250
decwave	W40_autox7~		WC		0
dpwr	991		SC		0
dmf	41		VS		77
	th		th		13
	ai		cdc		ph

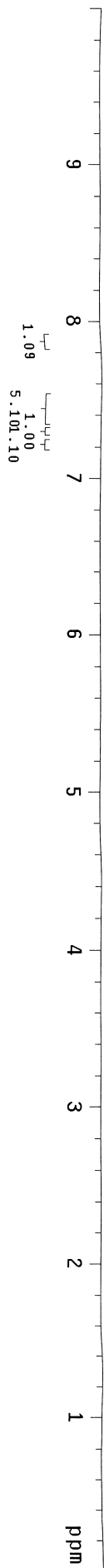


DZ-Ph-imidazole 1H CDC13  
 exp10 PROTON

date	Mar 29 2013	satmode	n
solvent	cdc13	wet	n
file	/indy/jwcho1/~	SPECIAL	
nmr/sys/data/DZ-Ph~		temp	not used
-imidazole 1H CDC13		gain	32
3/PROTON01.fid		spin	0
ACQUISITION		hst	0.008
SW	8000.0	pw90	9.900
at	3.000	alfa	10.000
np	48000	FLAGS	
fb	not used	i1	n
bs	32	in	n
d1	2.000	dp	y
nt	16	hs	nm
ct	16	PROCESSING	
tn	TRANSMITTER	tb	0.20
sf	499.708	fn	not used
tof	499.7	DISPLAY	
tpwr	61	wp	-0.1
pw	4.950	fft1	4996.8
DECOUPLER		rfp	4629.9
dn	C13	rp	3627.9
dof	0	tp	27.6
dm	nmn	PL0T	-76.2
decwave	w40_autok7~	WC	250
dpwr	991	SC	0
dmf	41	VS	100
	32258	th	49
ai	cdc	ph	



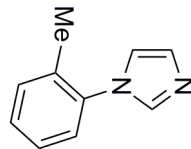
(Table 3, entry 1)



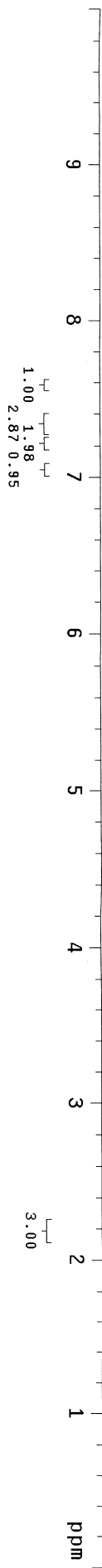
JC10013 CDC13

exp20 PROTON

SAMPLE	date	Mar 31 2013	satmode	n
solvent	cdcl3	wet	n	SPECIAL
file	/indy/jwcho1/~	temp	not used	32
nmrSYS/data/JC10013	13_1H_13C_CDC13/PR~	gain	0	0
OTON01.fid	ACQUISITION	hst	0.008	0
8000.0	pw90	alpha	9.900	10.000
3.000	alpha	10.000	10.000	10.000
48000	not used	il	n	n
not used	32	dp	n	n
2.000	16	hs	Y	Y
16	16	hs	nm	nm
TRANSMITTER	tb	PROCESSING	0.20	0.20
fn	fn	not used	not used	not used
499.708	sp	DISPLAY	-0.1	-0.1
499.7	wp	4996.8	4629.9	3627.9
61	rf1	4629.9	3627.9	22.8
4.950	rfp	22.8	-73.0	250
DECOUPLER	C13	rp	-73.0	250
0	tp	250	0	0
0	WC	0	39	25
decwave w40_autox7~	SC	0	39	25
991	VS	0	39	25
41	TH	0	39	25
32258	AI	cdc	ph	25
dmf				



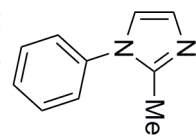
(Table 3, entry 2)



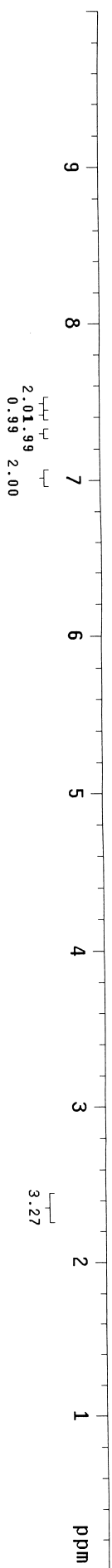
D2-04-254-1-Purified

exp10 PROTON

SAMPLE	date	Mar 16 2013	PRESATURATION	satmode	n
solvent	cdcl3		wet		n
file	/indy/dz1eg1e~		SPECIAL		not used
r/vnmr	sys/data/D2-~		temp		30
04-254-1-Purified/~			gain		20
PROTON01.fid			spin		0.008
ACQUISITION	8000.0	pw90	hst		9.700
at	3.000	alfa			10.000
np	48000		FLAGS		
fb	not used		il		n
bs	not used		in		n
dl	1.000	dp	dp		y
nt	16	hs	hs		nm
ct	16		PROCESSING		0.20
TRANSMITTER	H1	tb	fn		not used
tn	499.708	sp	DISPLAY		-0.1
sfreq	499.7	wp			4996.8
tof	61	rfl			4630.1
tpwr	4.850	rffl			3627.9
pw	DECOUPLER	rfp			42.5
dn	C13	tp			-73.7
dof	0	1p			
dm	nmn		PLOT		250
decwave	9	wc			0
dpwr	35	sc			23
dmf	32258	vs			13
		th			
		ai	cdc	ph	



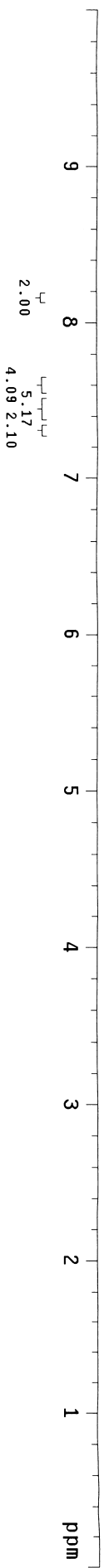
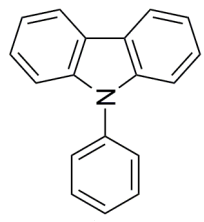
(Table 3, entry 3)



DZ-04-230-3-Purified

exp20 PROTON

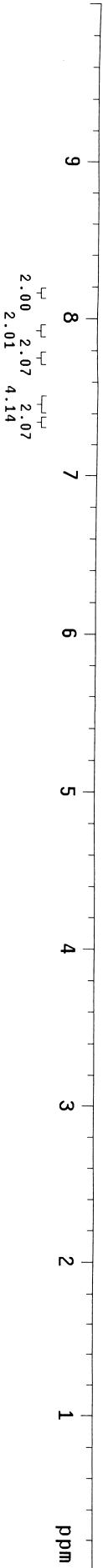
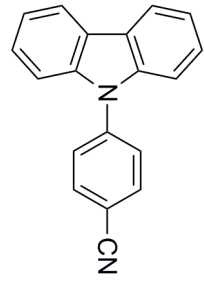
date	Mar 18 2013	SAMPLE	PRESATURATION	
solvent	cdcl3	file	cdcl3	wet
r/vmrsys	/indy/dzlegle~	temp	not used	SPECIAL
04-230-3-Purified/~		gain	30	
PROTON01.fid		spn	20	
ACQUISITION		hst	0.008	
SW	8000.0	pw90	9.700	
at	3.000	alfa	10.000	FLAGS
np	48000			
fb	not used			
bs	32			
d1	1.000			
nt	16			
ct	16			
TRANSMITTER	H1	1b	0.20	PROCESSING
tn	499.708	fn	not used	
sfrq	499.7	sp	-0.1	DISPLAY
tpwr	61	wp	4996.8	
pw	4.850	rf1	4630.1	
DECOUPLER	C13	rfp	3627.9	
dn	0	tp	40.0	
dof	0	1p	-71.1	PLOT
dm	nmn			
decwave	g	WC	250	
dpwr	35	SC	0	
dmf	32258	VS	50	
		th	38	
		ai	cdc	ph



JG9283 1H CDC13  
 exp20 PROTON

SAMPLE Mar 17 2013  
 solvent cdcl3  
 file /indy/jwchoi/~  
 vnmr/sys/data/JG9283~  
 3\_1H\_CDC13/PROTON0~  
 1.fid  
 ACQUISITION  
 SW 8000.0  
 at 3.000  
 np 48000  
 fb not used  
 bs 32  
 d1 2.000  
 nt 16  
 ct 16  
 TRANSMITTER  
 tn H1  
 sfreq 499.708  
 tof 499.7  
 tpwr 61  
 pw 4.850  
 DECOUPLER C13  
 dn 0  
 dof 0  
 dm nmn  
 decwve g  
 dpwr 35  
 dmf 32258  
 ai cdc ph

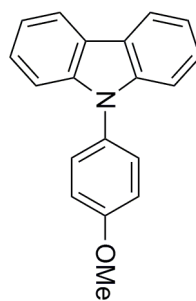
PRESATURATION  
 satmode n  
 wet n  
 SPECIAL  
 temp not used  
 gain 30  
 spin 20  
 hst 0.008  
 pw90 9.700  
 alfa 10.000  
 FLAGS  
 i1 n  
 in n  
 dp y  
 hs nn  
 PROCESSING  
 lb 0.20  
 fn not used  
 DISPLAY  
 sp -0.1  
 wp 4996.8  
 rffl 4630.1  
 rfp 3627.9  
 rp 43.3  
 lp -75.9  
 PLOT  
 wc 250  
 sc 0  
 vs 49  
 th 37



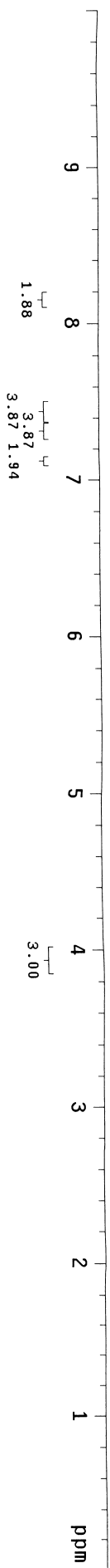
JG9281 CDC13

exp20 PROTON

SAMPLE	date	Mar 16 2013	PRESATURATION	satmode	n
solvent	file	/indy/jwcho1/~	wet		n
nmrSYS	data	/JG928~	SPECIAL		
1_1H_13C	CDC13/PRO~		temp	not used	
TON01.fid			gain	30	
ACQUISITION			spin	20	
			hst	0.008	
sw	8000.0		pw90	9.700	
at	3.000		alpha	10.000	
np	48000		FLAGS		
fb	not used		i1	n	
bs	not used		in	n	
di	2.000		dp	y	
nt	16		hs	nm	
ct	16		PROCESSING	0.20	
tn	TRANSMITTER	H1	1b	fn	not used
sfrq	499.708		fn		
tof	499.7		sp		DISPLAY
tpwr	61		wp		-0.1
pw	4.850		rfl		4996.8
DECOUPLER	C13		rffl		4630.1
dn	0		rfp		3627.9
dof	0		1p		41.5
dm	nmn		1p		-74.5
decwave	9		WC		250
dpwr	35		SC		0
dmf	32258		VS		42
			th		25
			ai		cdc ph



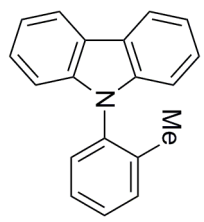
(Table 4, entry 3)



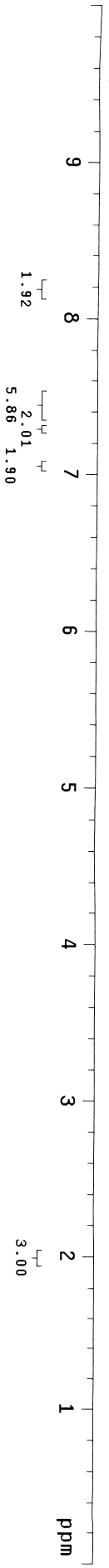


JC9275B CDC13  
 exp20 PROTON

SAMPLE PRESATURATION  
 date Mar 13 2013 satmode n  
 solvent cdc13 wet n  
 file //indy/jwchoi/~ SPECIAL not used  
 vnmr/svs/data/JC9275-5B\_CDC13/PROTON01.~ temp 30  
 gain 20  
 fid hst 0.008  
 spn 20  
 pw90 9.700  
 a1fa 10.000  
 ACQUISITION  
 SW 8000.0  
 at 3.000  
 np 48000  
 fb not used  
 bs 32  
 d1 2.000  
 nt 16  
 ct 16  
 TRANSMITTER  
 tn H1  
 sfreq 499.708  
 tof 499.7  
 tpwr 4.850  
 pw 4.850  
 DECOUPLER C13  
 dn 0  
 dof 0  
 dm nm  
 decwve 9  
 dpwr 35  
 vs 141  
 dmf 32258  
 ai cdc ph  
 PLOT  
 WC 250  
 SC 0  
 VS 141  
 TH 25

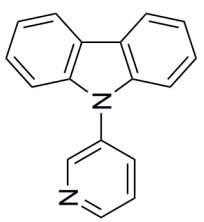


(Table 4, entry 4)

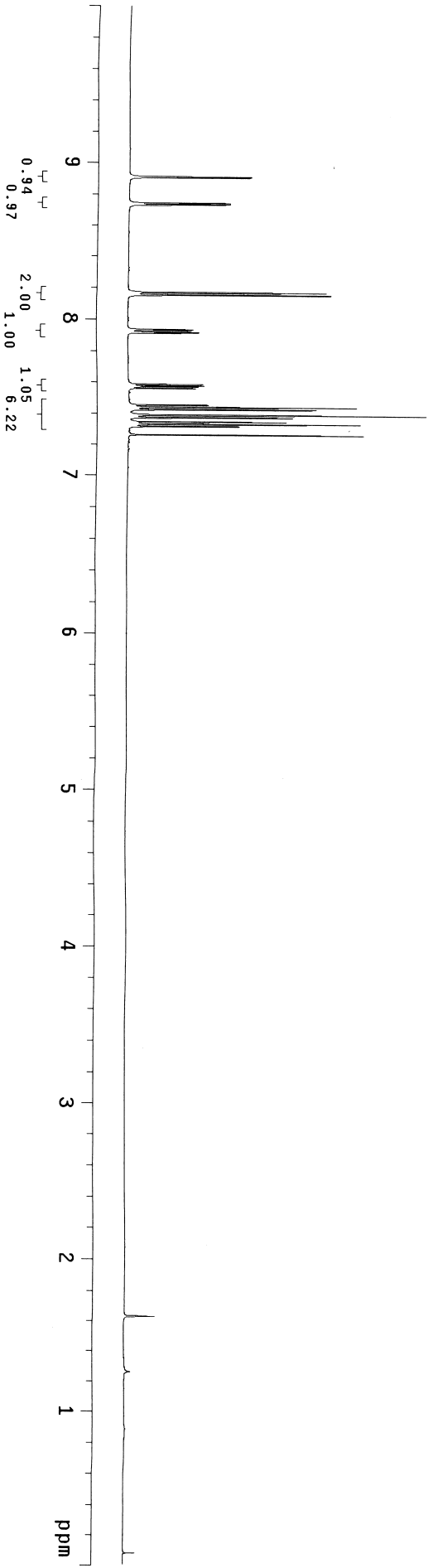


DZ-04-252-1-Purified  
 exp20 PROTON

SAMPLE	Mar 18 2013	PRESATURATION	
date	Mar 18 2013	satmode	n
solvent	cdcl3	wet	n
file	/indy/dziegle	SPECIAL	
r/vnmr	sys/data/DZ-~	temp	not used
04-252-1-Purified/~		gain	30
PROTON01.fid		spin	20
ACQUISITION		hst	0.008
sw	8000.0	pw90	9.700
at	3.000	alfta	10.000
np	48000	FLAGS	
fb	not used	i1	n
bs	32	in	n
d1	1.000	dp	y
nt	16	hs	nm
ct	16	hs	nm
TRANSMITTER	H1	1b	0.20
tn	499.708	fn	not used
sfrq	499.7	DISPLAY	
tof	61	wp	-0.1
tpwr	4.850	rf1	4996.8
pw		rf2	4630.1
DECOUPLER	C13	rfp	3627.9
dn	0	tp	41.0
dof	nmn	1p	-73.0
dm	g	WC	250
decwvave	35	SC	0
dpwr	35	VS	75
dmt	32258	th	40
ai	cdc	ph	

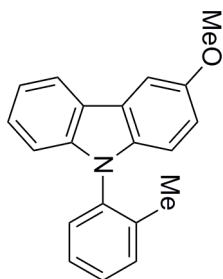


(Table 4, entry 5)

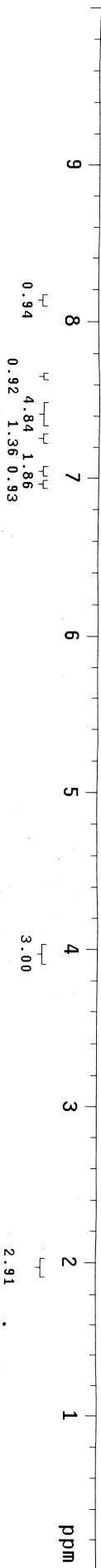


DZ-04-262-1-Purified  
 exp20 PROTON

SAMPLE PRESATURATION  
 date Mar 18 2013 satmode n  
 solvent Mar cdc13 wet n  
 file /indy/dz1egle~ SPECIAL not used  
 r/vnmrSYS/data/DZ-~ gain 30  
 04-262-1-Purified/~ spin 20  
 PROTON02.fid hst 0.008  
 ACQUISITION pw90 9.700  
 SW 8000.0 atfa 10.000  
 at 3.000  
 np 48000  
 fb not used  
 bs 32 in n  
 d1 1.000 dp n  
 nt 18 hs y  
 ct 18  
 TRANSMITTER  
 tn H1 1b 0.20  
 sfreq 499.708 fn not used  
 tof 499.7 DISPLAY  
 tpwr 61 WP -0.1  
 pw 4.850 rfp 4996.8  
 DECOUPLER C13 rfp 4630.1  
 dn 0 rfp 3627.9  
 dof 0 tp 40.0  
 dm mn -70.0  
 decouple g WC 250  
 dpwr 35 SC 0  
 dmf 32258 VS 44  
 th 50  
 ai cdc ph



(Table 4, entry 6)

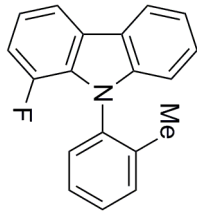


D2-04-264-1-Purified

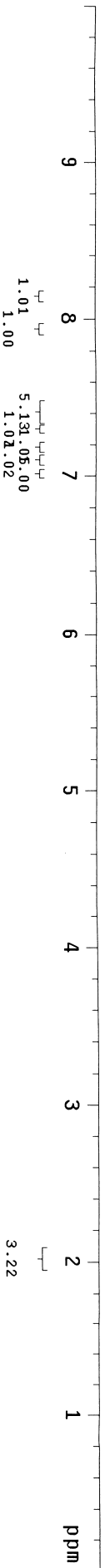
exp20 PROTON

```
SAMPLE          PRESATURATION
date Mar 18 2013 satmode
solvent Mar 18 2013 cdc13 wet
file /indy/dz1eg1e~ SPECIAL
r/vnmrSYS/data/D2~ temp not used
04-264-1-Purified/~ gain 30
PROTON02.fid sp in 20
ACQUISITION hst 0.008
SW 8000.0 pw90 9.700
at 3.000 alfa 10.000
np 48000
fb not used i1 n
bs 1.000 dp in n
d1 1.000 dp Y
nt 16 hs nn
ct 16 hs nn

TRANSMITTER H1 fb 1b PROCESSING 0.20
fn not used
sfrq 499.708 fn DISPLAY
tof 499.7 wp sp -0.1
tpwr 61 rfp 4996.8
pw 4.850 rff1 4630.1
DECOUPLER C13 fp rffp 3627.9
dn C13 fp 42.2
dof 0 tp -74.1
dm nmn PLOT 250
dm decave 9 wc 0
dpwr 35 sc 0
dmf 32258 vs 97
ai th 25
cdc ph
```

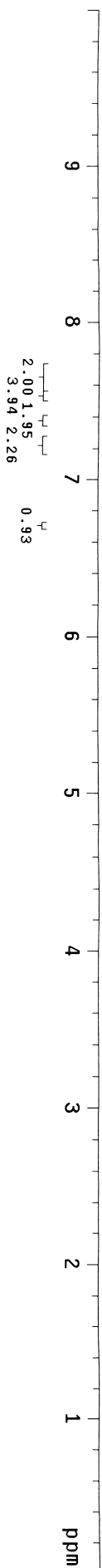
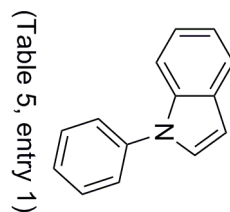


(Table 4, entry 7)



DZ-05-030-1-Purified  
 exp10 PROTON

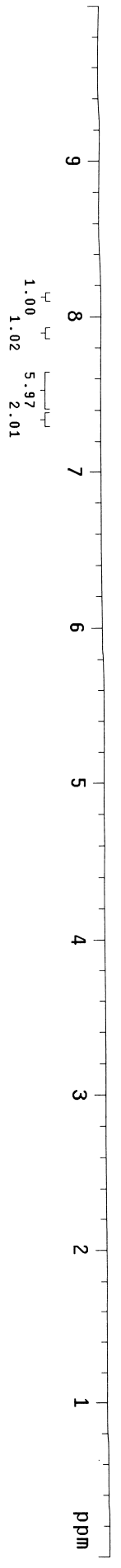
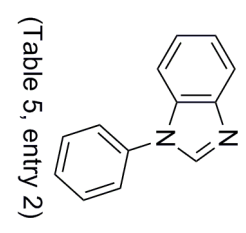
SAMPLE PRESATURATION  
 date Apr 19 2013 satmode h  
 solvent cdcl3 wet h  
 file /indy/dzlegle~ SPECIAL  
 r/vnmrSYS/data/DZ-~ temp not used  
 05-030-1-Purified/~ gain 32  
 PROTON01.fid spin 20  
 ACQUISITION hst 0.008  
 SW 8000.0 pw90 9.900  
 at 3.000 alfa 10.000  
 np 48000  
 fb not used  
 bs 32 in  
 d1 1.000 dp  
 nt 16 hs  
 ct 16  
 TRANSMITTER  
 tn H1 lb  
 sfrq 499.708 fn  
 tof 499.7 SP DISPLAY  
 tpwr 61 wp 4996.8  
 pw 4.950 rfp 4630.1  
 DECOUPLER C13 rfp 3627.9  
 dn 0 tp 13.3  
 dof 0 lp -67.9  
 dm nm  
 decwawe w40\_autox7~ WC PLOT 250  
 991 SC  
 41 VS  
 dmf 32258 th  
 ai cdc ph



JMM1250

exp10 PROTON

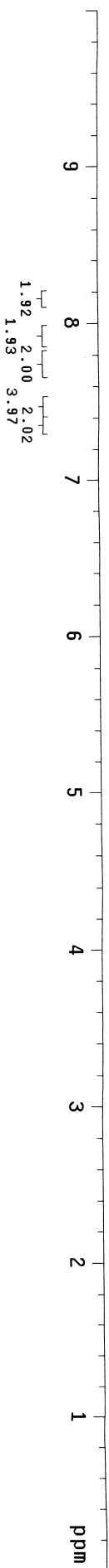
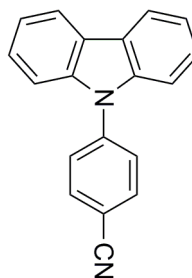
SAMPLE	date	Apr 27 2013	satmode	n
solvent	CdCl3		wet	n
file	/indy/jmm011n~		SPECIAL	not used
a/vmrsys/data/jmm~	1250/PROTON01.fid		temp	20
ACQUISITION			gain	20
SW	8000.0		spin	0.008
at	3.000		pw90	9.900
np	48000		alpha	10.000
fb	not used		FLAGS	
bs	32		11	n
d1	2.000		in	n
nt	16		dp	y
ct	16		hs	nn
TRANSMITTER	H1	1b	PROCESSING	0.20
tn	499.708	fn	not used	
sfrq	499.7		DISPLAY	-0.0
tof	61	sp		4996.8
tpwr	4.950	wp	rff1	1001.7
pw		rff1		0
DECOUPLER	C13	rff1		24.3
dn	0	rfp		-68.3
dof	0	tp		
dm	nmh			
decwave	W40_autox7~			
	931	WC	PLOT	250
dpwr	41	SC		0
dmf	32258	VS		101
		th		31
		ai	cdc	ph



JC10065 1H CDC13

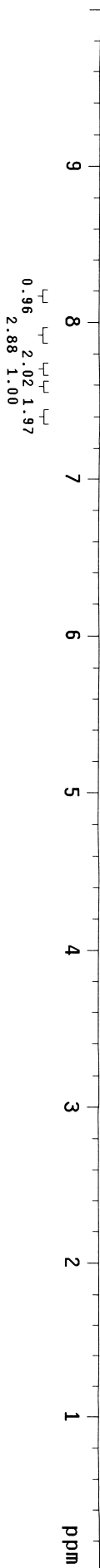
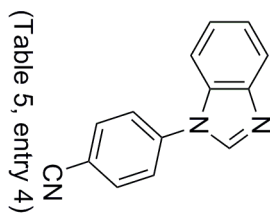
exp10 PROTON

SAMPLE	date	Apr 27 2013	PRESATURATIION	satmode	n
solvent	file	/indy/jwcho1/~	wet	cdcl3	n
nmrSYS/data/JC100~	65_1H_CDC13/PROTON~	02.fid	temp	gain	32
ACQUISITION	8000.0	pw90	hst	spin	20
at	3.000	alpha	10.000	alpha	9.900
np	48000	not used	11	11	n
fb	not used	32	in	dp	n
bs	2.000	16	hs	nt	y
d1	16	16	hs	nt	nn
ct	TRANSMITTER	H1	1b	fn	0.20
tn	499.708	sp	not used	DISPLAY	not used
stf	499.7	wp	-0.1	4996.8	
tof	61	rf1	4630.1	3627.9	
tpwr	4.950	rfp	17.3	-64.2	
pw	DECOUPLER	C13	rfp	1p	
dn	0	nmn	250	0	
dof	0	sc	97	33	
dm	decwave	w40_autox7~	91	41	
decwave	991	vs	th	32258	
dpwr	41	th	33	cdc	ph
dmf	32258	at			



DZ-05-034-1-Purified  
 exp10 PROTON

SAMPLE	Apr 19 2013	PRESATURATION	n
date	Apr 19 2013	satmode	n
solvent	cdcl3	wet	n
file	/indy/dzlegle/~	SPECIAL	not used
r/vnmr	sys/data/DZ-~	temp	32
05-034-1-Purified/~	gain	spin	20
PROTON01.fid	hst	pw90	0.008
ACQUISITION	atfa	atfa	9.900
80000.0	atfa	atfa	10.000
3.000	atfa	atfa	10.000
48000	atfa	atfa	10.000
not used	atfa	atfa	10.000
32	atfa	atfa	10.000
1.000	atfa	atfa	10.000
16	atfa	atfa	10.000
16	atfa	atfa	10.000
16	atfa	atfa	10.000
16	atfa	atfa	10.000
TRANSMITTER	1b	PROCESSING	0.20
tn	fn	DISPLAY	not used
sfrq	499.708	DISPLAY	not used
tof	499.7	SP	-0.1
tpwr	61	WP	4996.8
pw	4.950	rfl	4630.1
DECOUPLER	C13	rfl	3627.9
dn	0	rp	15.6
dof	0	lp	-72.5
dm	nmr	PLOT	250
decwave	w40_autox7~	WC	0
991	sc	VS	0
41	vs	th	100
32258	th	ai	4
dmf	ai	cdc	ph

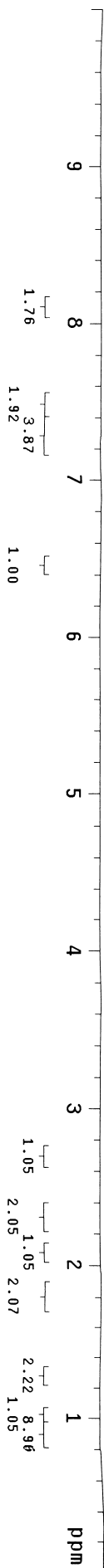
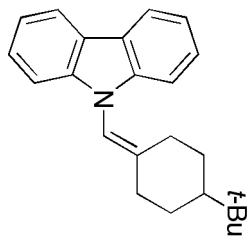




JMM1180

exp10 PROTON

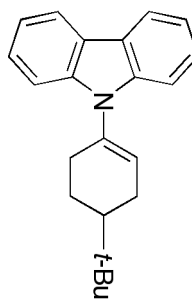
SAMPLE		PRESATURATION	
date	Mar 29 2013	satmode	n
solvent	cdcl3	wet	n
file	/indy/jmm011h~	SPECIAL	not used
a/vnmr/sys/data/JMM~	1180/PROTON03.fid	temp	20
ACQUISITION		gain	0
SW	8000.0	spin	0.008
at	3.000	pw90	9.900
np	48000	atfa	10.000
fb	not used	FLAGS	
bs	32	l1	n
d1	1.000	in	n
nt	16	dp	y
ct	16	hs	nm
TRANSMITTER		PROCESSING	
tn	H1	lb	0.20
sfrq	499.708	fn	not used
tof	499.7	DISPLAY	
tpwr	61	SP	-0.0
pw	4.950	WP	4996.8
DECOUPLER		rf1	1001.7
dn	C13	rfp	0
dof	0	tp	30.8
dm	nmr	lp	-73.2
decwawe	w40_autox7~	PLOT	
dpwr	991	WC	250
dmf	41	SC	0
	32258	VS	81
		th	6
ai	cdc	ph	



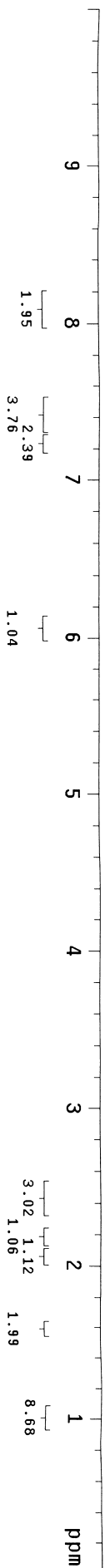
JMM1200

exp10 PROTON

date	Mar 29 2013	PRESATURATION	satmode	n
solvent	cdcl3	wet		n
file	/indy/jmm011r	SPECIAL		
a/vnmr/sys/data/JMM~		temp	not used	
1200/PROTON03.f1d		gain	32	
ACQUISITION		spin	0	
SW	8000.0	hst	0.008	
at	3.000	pw90	9.900	
np	48000	alfa	10.000	
fb	not used	FLAGS		
bs	32	i1	n	
d1	1.000	in	n	
nt	32	dp	y	
ct	32	hs	nm	
TRANSMITTER	H1	PROCESSING	0.20	
tn	499.708	fb	not used	
sfrq	499.7	fn		
tof	61	SP	-0.0	
tpwr	4.950	WP	4996.8	
pw		rf1	1001.7	
DECOUPLER	C13	rfp	0	
dn	0	lp	29.1	
dof	nnn	PLT	-72.6	
dm	nnn	WC	250	
decouple w40_autox7~	991	SC	0	
dpwr	41	VS	40	
dmf	32258	th	13	
ai	cdc	ph		



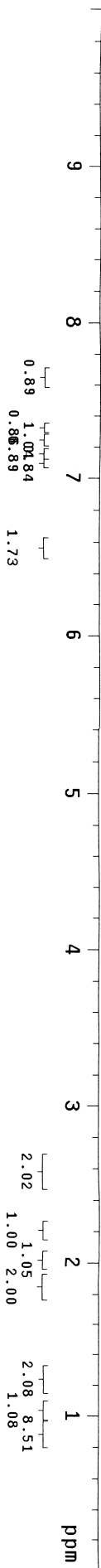
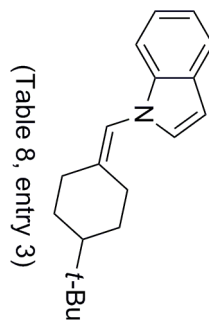
(Table 8, entry 2)



JMM1193

exp10 PROTON

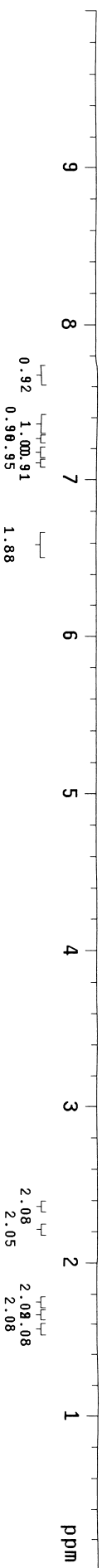
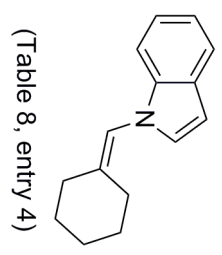
SAMPLE PRESATURATION  
date Mar 26 2013 satmode n  
solvent cdc13 wet n  
file /indy/jmm011h~ SPECIAL  
a/vnmr/sys/data/JMM~ temp not used  
1193/PROTON01.fid gain 20  
ACQUISITION hst 0  
8000.0 spin 0.008  
3.000 pw90 9.900  
48000 alfa 10.000  
not used  
32 i1 n  
1.000 in n  
16 dp y  
16 hs nm  
ct TRANSMITTER H1 1b  
tn 499.708 fn 0.20  
sfrq 499.7 DISPLAY not used  
tof 61 SP -0.0  
tpwr 4.950 WP 4996.8  
DECOUPLER C13 rffl 1001.7  
dn 0 ffp 0  
dof 0 rp 26.0  
dm nnn lp -70.8  
decwave W40\_autox7~ PLOT 250  
931 WC 0  
41 SC 45  
32258 VS 13  
dmf th ai cdc ph



JMM1234

exp10 PROTON

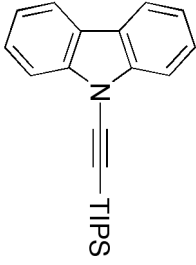
SAMPLE PRESATURATION  
date Apr 26 2013 satmode n  
solvent cdc13 wet n  
f1file /indy/jmm011r~ SPECIAL  
a/vmrsys/data/JMM~ temp not used  
1234/PROTON01.fid gain 20  
ACQUISITION hst 20  
sw 8000.0 pwr90 9.900  
at 3.000 alfa 10.000  
np 48000 not used  
fb not used  
bs 32 i1 n  
d1 1.000 in n  
nt 16 dp y  
ct 16 hs nm  
TRANSMITTER PROCESSING  
tn H1 1b 0.20  
sfrq 499.708 fn not used  
tof 499.7 DISPLAY  
tpwr 4.950 SP -0.0  
pw 4.950 WP 4996.8  
DECOUPLER C13 rffl 1001.7  
dn 0 rfp 0  
dof 0 rfp 28.4  
dm nnn 1p -81.8  
decwave W40\_autox7~ PLOT 250  
dpwr 931 WC 0  
dmf 41 SC 0  
32258 th VS 113  
at cdc ph 50



JMM1202

exp10 PROTON

date	Mar 26 2013	PRESATURATION	n
solvent	cdcl3	satmode	n
file	/indy/jmmoljrn~	temp	not used
a/vnmr/sys/data/JMM~	1202/PROTON01.f1d	gain	20
ACQUISITION		spin	0
sw	8000.0	hst	0.008
at	3.000	pw90	9.900
np	48000	alfa	10.000
fb	not used	FLAGS	
bs	32	i1	n
d1	1.000	in	n
nt	16	dp	y
ct	16	hs	nm
tn	TRANSMITTER	PROCESSING	0.20
strq	499.708	fn	not used
tof	499.7	DISPLAY	
tbwr	61	sp	-0.0
pw	4.950	wp	4996.8
dn	DECOUPLER	rfl	1001.7
dn	C13	rflp	0
dof	0	rp	25.5
dmm	nmn	lp	-71.9
decwave	W40_autox7~	PL0T	250
dpwr	991	wc	0
dmf	41	sc	0
	32258	vs	47
		th	3
		ai	cdc
			ph



(Table 8, entry 5)

