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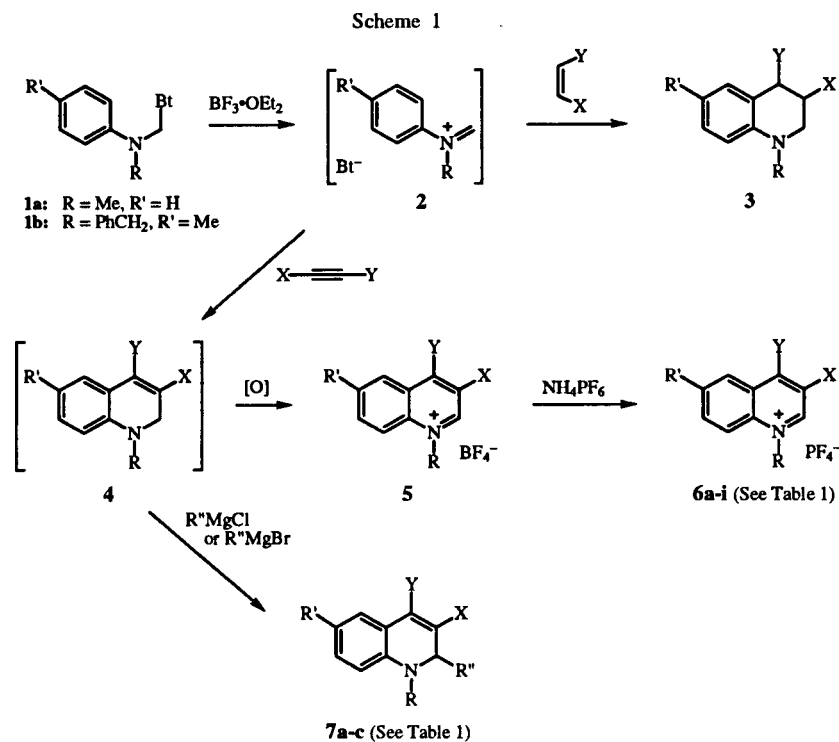
A novel method for the synthesis of 1,4- and 1,3,4-substituted quinolinium salts and 1,2,4- and 1,2,3,4-substituted-1,2-dihydroquinolines from *N*-substituted-*N*-(benzotriazol-1-ylmethyl)aniline derivatives is reported.

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Introduction.

We recently reported that *N*-substituted-*N*-(benzotriazol-1-ylmethyl)aniline derivatives of type 1, under catalysis by Lewis acids, reacted with both activated [1] and non-activated alkenes [2] to form tetrahydroquinolines 3, via the Mannich type intermediate 2 (Scheme 1).

dye from phosphoryl chloride and *N*-methylacetanilide by postulating a quinolinium salt intermediate. This reaction was later developed into a general synthesis of quinolinium salts by Meth-Cohn and Taylor [5]; however, only electron-rich alkenes were used in this reverse Vilsmeier approach. Minkin *et al.* reported that *N*-arylquinolinium



In an effort to expand the scope of this methodology, we reacted intermediate 2 with alkynes, but obtained quinolinium salts instead of the expected 1,2-dihydroquinolines 4. We now report that such reactions offer a new route for the preparation of quinolinium salts from *N*-substituted-*N*-(benzotriazol-1-ylmethyl)aniline derivatives.

Quinolinium salts have attracted much attention. They are generally prepared by the quaternization of quinolines and some syntheses by ring formation are known [3]. Fischer, Muller and Vilsmeier [4] rationalized the formation of a red

salts were prepared by the condensation of diarylamines with aldehydes [6,7].

Results and Discussion.

N-Substituted-*N*-(benzotriazol-1-ylmethyl)aniline derivatives 1a,b were prepared from the corresponding *N*-alkylanilines, benzotriazole and formaldehyde [8]. Treatment of *N*-substituted-*N*-(benzotriazol-1-ylmethyl) anilines 1a,b and alkynes with boron trifluoride etherate at 0° followed by stirring in air for several hours at room temperature

gave the corresponding quinolinium salts **5a-i**. Anion exchange with ammonium hexafluorophosphate lead to the sparingly soluble quinolinium salts **6a-i**. The results are summarized in Scheme 1 and Table 1.

Table 1

Quinolinium Hexafluorophosphates **6** and 1,2-Dihydroquinolines **7**

Compound	R'	R	X	Y	R''	Yield (%)	Mp (°C)
6a	H	Me	H	Ph	H	79	213-215
6b	H	Me	Et	Et	H	72	145-147
6c	H	Me	H	<i>n</i> -Bu	H	70	122-124
6d	H	Me	Ph	Ph	H	58	223-225
6e	H	Me	Me	Ph	H	56	210-212
6f	Me	Bn	H	Ph	H	75	173-175
6g	Me	Bn	Et	Et	H	68	187-189
6h	Me	Bn	Me	Ph	H	71	114-116
6i	Me	Bn	H	<i>n</i> -Bu	H	62	143-145
7a	H	Me	H	Ph	Ph	74	128-130
7b	H	Me	Me	Ph	Ph	54	152-154
7c	H	Me	H	Ph	<i>n</i> -Bu	71	oil

As previously discussed [9], the iminium salt intermediates **2** are formed from **1** in the presence of Lewis acid. Intermediates **2** were quenched by alkynes to generate the 1,2-dihydroquinolines **4** which were easily oxidized by air in an acidic medium to form **5**. Similar oxidations (dehydrogenations) have been reported by Minkin and Ardashev [6] and Reid [10]. Quinolinium salts **6a-i** were isolated as hexafluorophosphate salts in yields of 56-79%. The structures of **6a-i** were characterized by ^1H and ^{13}C nmr spectra and elemental (C, H, N) analyses.

In initial experiments, the reaction of **1a** gave *N*-methylaniline as a byproduct in about 20% yield along with the expected quinolinium salt, indicating some hydrolysis of **1a** under the reaction conditions. Therefore, in subsequent reactions, excess *N*-substituted-*N*-(benzotriazol-1-ylmethyl)-aniline derivatives were used. Direct reaction of the crude mixture, **5a,e**, with Grignard reagents at 0° gave the expected 1,2-dihydroquinolines **7a-c** in good yields. Products **7a-c** were characterized by ^1H and ^{13}C nmr spectra and elemental (C, H, N) analyses or high resolution mass spectrometry.

In conclusion, the present method provides a general method to synthesize 1,4- and 1,3,4-substituted quinolinium salts and 1,2,4- and 1,2,3,4-substituted-1,2-dihydroquinolines by using readily available, crystalline *N*-substituted-*N*-(benzotriazol-1-ylmethyl)aniline derivatives as starting materials.

EXPERIMENTAL

Melting points were determined using a Kofler hot stage apparatus, and are uncorrected. The ^1H nmr and ^{13}C nmr spectra were recorded on a Gemini 300 nmr spectrometer (300 MHz and 75 MHz respectively) in chloroform-*d* or dimethyl sulfoxide-*d*₆

(with tetramethylsilane for ^1H and chloroform-*d* or dimethyl sulfoxide-*d*₆ for ^{13}C as the internal reference). Microanalyses were performed on a Carlo Erba 1106 elemental analyzer.

Compound **1a** was prepared by following the procedure of the reference [8].

N-(Benzotriazol-1-ylmethyl)-*N*-methylaniline (**1a**).

This compound was obtained as a pale yellow solid (77%, 19.25 g), mp 77° (lit [8]) mp $76-78^\circ$; ^1H nmr (chloroform-*d*): δ 8.01 (d, $J = 7.1$ Hz, 1H), 7.30-7.26 (m, 4H), 7.19 (d, $J = 7.2$ Hz, 1H), 7.00 (d, $J = 8.0$ Hz, 2H), 6.90 (t, $J = 7.1$ Hz, 1H), 6.12 (s, 2H), 3.01 (s, 3H); ^{13}C nmr (chloroform-*d*): δ 147.8, 146.1, 132.5, 129.4, 127.3, 123.8, 119.9, 119.7, 115.1, 110.0, 66.7, 37.5.

Preparation of *N*-(Benzotriazol-1-ylmethyl)-*N*-(4-methylphenyl)benzylamine (**1b**).

4-Methylaniline (50 mmoles) and benzaldehyde (50 mmoles) were dissolved in dry benzene (100 ml) and heated under reflux for 2 hours in a flask equipped with a Dean-Stark apparatus and condenser. After cooling, the solvent was removed under reduced pressure. The resulting crude imine was dissolved in methanol (100 ml) and sodium borohydride (40 mmoles) was added. The reaction mixture was further stirred for 1 hour. Water (20 ml) and ethyl acetate (30 ml) were added. The organic layer was washed with water, dried with anhydrous sodium sulfate and filtered. Removal of the solvent under reduced pressure gave an oil which was purified by column chromatography to give *N*-(4-methylphenyl)benzylamine (90%). Benzotriazole (45 mmoles) was added to *N*-(4-methylphenyl)benzylamine (45 mmoles) in ethanol (40 ml). After stirring 30 minutes, an aqueous solution of formaldehyde (37%, 30 mmoles) was added and the reaction mixture stirred at room temperature for 3 hours. The mixture was filtered and the white solid was rinsed with ethanol and dried to give *N*-(benzotriazol-1-ylmethyl)-*N*-(4-methylphenyl)benzylamine (**1b**) (86%, 8.27 g), mp $134-136^\circ$; ^1H nmr (chloroform-*d*): δ 7.99 (d, $J = 8.5$ Hz, 1H), 7.28-7.21 (m, 7H), 7.09 (d, $J = 8.5$ Hz, 1H), 7.02 (d, $J = 8.5$ Hz, 2H), 6.92 (d, $J = 8.5$ Hz, 2H), 6.05 (s, 2H), 4.52 (s, 2H), 2.23 (s, 3H); ^{13}C nmr (chloroform-*d*): δ 145.9, 145.3, 137.3, 132.8, 130.1, 129.9, 128.5, 127.5, 127.2, 123.7, 119.6, 117.1, 109.9, 65.2, 53.6, 20.3.

Anal. Calcd. for $\text{C}_{21}\text{H}_{20}\text{N}_4$: C, 76.80; H, 6.14; N, 17.06. Found: C, 76.92; H, 6.27; N, 17.23.

General Procedure for the Preparation of Quinolinium Hexafluorophosphates **6a-i**.

N-Substituted-*N*-(benzotriazol-1-ylmethyl)aniline **1** (4.5 mmoles) was dissolved in methylene chloride at 0° and alkyne (3 mmoles) was added. After 10 minutes, boron trifluoride etherate (6 mmoles) was added and the mixture was stirred at this temperature for 4 hours. The septum was removed to allow the oxidation and the reaction mixture was stirred at room temperature for 3 hours. The solvent was removed and the residue dissolved in a minimum of ethyl acetate (for compounds **6a-e**) or dimethyl sulfoxide (for compounds **6f-i**). Ammonium hexafluorophosphate (4.5 mmoles) and water were added until no more precipitation was detected. The residue was filtered and washed with water to give the expected quinolinium salts **6a-i** as solid products.

1-Methyl-4-phenylquinolinium Hexafluorophosphate (**6a**).

This compound was obtained as pale yellow crystals (79%, 865 mg), mp $213-215^\circ$; ^1H nmr (dimethyl sulfoxide-*d*₆): δ 9.54 (d, $J = 5.7$ Hz, 1H), 8.60 (d, $J = 8.6$ Hz, 1H), 8.33 (t, $J = 7.9$ Hz,

1H), 8.22 (d, $J = 8.2$ Hz, 1H), 8.16 (d, $J = 5.8$ Hz, 1H), 8.04 (t, $J = 7.5$ Hz, 1H), 7.70 (br s, 5H), 4.70 (s, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 157.7, 149.2, 138.8, 135.1, 134.8, 130.6, 130.2, 129.8, 129.2, 128.1, 127.2, 122.2, 119.5, 45.2.

Anal. Calcd. for $\text{C}_{16}\text{H}_{14}\text{F}_6\text{NP}$: C, 52.61; H, 3.86; N, 3.83. Found: C, 52.86; H, 3.76; N, 3.83.

1-Methyl-3,4-diethylquinolinium Hexafluorophosphate (6b).

This compound was obtained as a pale green solid (72%, 745 mg), mp 145-147°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.38 (br s, 1H), 8.59 (d, $J = 8.5$ Hz, 1H), 8.46 (d, $J = 8.8$ Hz, 1H), 8.21 (t, $J = 7.5$ Hz, 1H), 8.03 (t, $J = 7.9$ Hz, 1H), 4.60 (s, 3H), 3.38 (q, $J = 7.6$ Hz, 2H), 3.00 (q, $J = 7.6$ Hz, 2H), 1.36 (t, $J = 7.4$ Hz, 3H), 1.31 (t, $J = 7.5$ Hz, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 159.4, 150.1, 136.8, 135.3, 133.8, 129.7, 127.7, 126.2, 119.4, 44.9, 23.2, 21.5, 14.9, 14.7.

Anal. Calcd. for $\text{C}_{14}\text{H}_{18}\text{F}_6\text{NP}$: C, 48.70; H, 5.25; N, 4.06. Found: C, 48.57; H, 5.27; N, 4.03.

1-Methyl-4-*n*-butylquinolinium Hexafluorophosphate (6c).

This compound was obtained as a pale yellow solid (70%, 724 mg), mp 122-124°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.36 (d, $J = 6.1$ Hz, 1H), 8.61 (d, $J = 8.3$ Hz, 1H), 8.50 (d, $J = 8.8$ Hz, 1H), 8.27 (t, $J = 7.7$ Hz, 1H), 8.06 (t, $J = 8.2$ Hz, 1H), 8.05 (d, $J = 6.3$ Hz, 1H), 4.60 (s, 3H), 3.38 (t, $J = 7.7$ Hz, 2H), 1.78-1.70 (m, 2H), 1.50-1.42 (m, 2H), 0.96 (t, $J = 7.3$ Hz, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 161.6, 149.0, 138.0, 134.9, 129.7, 127.9, 126.4, 121.6, 119.6, 45.0, 31.9, 31.8, 22.0, 13.6.

Anal. Calcd. for $\text{C}_{14}\text{H}_{18}\text{F}_6\text{NP}$: C, 48.70; H, 5.25; N, 4.06. Found: C, 48.64; H, 5.50; N, 4.07.

1-Methyl-3,4-diphenylquinolinium Hexafluorophosphate (6d).

This compound was obtained as a beige solid (58%, 767 mg), mp 223-225°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.76 (s, 1H), 8.63 (d, $J = 8.6$ Hz, 1H), 8.31 (t, $J = 7.1$ Hz, 1H), 8.01 (t, $J = 6.9$ Hz, 1H), 7.88 (d, $J = 8.2$ Hz, 1H), 7.50 (br s, 3H), 7.36-7.34 (m, 7H), 4.76 (s, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 155.2, 150.7, 137.5, 134.8, 134.4, 134.1, 133.5, 130.4, 130.1, 129.7, 129.3, 128.6, 128.5, 128.4, 119.2, 45.2.

Anal. Calcd. for $\text{C}_{22}\text{H}_{18}\text{F}_6\text{NP}$: C, 59.87; H, 4.11; N, 3.17. Found: C, 59.36; H, 4.06; N, 3.13.

1,3-Dimethyl-4-phenylquinolinium Hexafluorophosphate (6e).

This compound was obtained as white microcrystals (56%, 637 mg), mp 210-212°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.63 (s, 1H), 8.57 (d, $J = 8.8$ Hz, 1H), 8.25 (t, $J = 7.9$ Hz, 1H), 7.95 (t, $J = 7.9$ Hz, 1H), 7.73-7.71 (m, 4H), 7.45 (d, $J = 6.3$ Hz, 2H), 4.71 (s, 3H), 2.39 (s, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 156.3, 151.0, 137.1, 134.0, 133.7, 130.3, 130.0, 129.6, 129.1, 128.5, 128.4, 127.7, 119.1, 45.2, 17.4.

Anal. Calcd. for $\text{C}_{17}\text{H}_{16}\text{F}_6\text{NP}$: C, 53.83; H, 4.25; N, 3.69. Found: C, 54.14; H, 4.40; N, 3.89.

1-Benzyl-4-phenyl-6-methylquinolinium Hexafluorophosphate (6f).

This compound was obtained as a beige solid (75%, 1.02 g), mp 173-175°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.53 (d, $J = 5.8$ Hz, 1H), 8.33 (d, $J = 9.0$ Hz, 1H), 8.04 (d, $J = 5.2$ Hz, 1H), 7.90 (d, $J = 8.8$ Hz, 1H), 7.82 (s, 1H), 7.55 (s, 5H), 7.28-7.23 (m, 5H), 6.25 (s, 2H), 2.34 (s, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 157.9, 148.2, 140.8, 137.2, 136.6, 135.0, 134.0, 130.6, 129.8, 129.1, 128.8, 128.1, 127.3, 127.0, 122.7, 119.5, 59.8, 21.0.

Anal. Calcd. for $\text{C}_{23}\text{H}_{20}\text{F}_6\text{NP}$: C, 60.66; H, 4.43; N, 3.08. Found: C, 60.38; H, 4.43; N, 3.22.

1-Benzyl-3,4-diethyl-6-methylquinolinium Hexafluorophosphate (6g).

This compound was obtained as a white solid (68%, 704 mg), mp 187-189°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.60 (s, 1H), 8.40 (s, 1H), 8.34 (d, $J = 9.1$ Hz, 1H), 7.96 (d, $J = 9.0$ Hz, 1H), 7.39 (s, 5H), 6.31 (s, 2H), 3.40 (q, $J = 7.2$ Hz, 2H), 3.07 (q, $J = 7.4$ Hz, 2H), 2.61 (s, 3H), 1.41 (t, $J = 7.4$ Hz, 3H), 1.35 (t, $J = 7.2$ Hz, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 160.0, 149.0, 140.3, 136.0, 135.8, 134.5, 134.2, 129.1, 128.6, 128.5, 127.0, 125.2, 119.4, 59.7, 23.3, 21.7, 21.0, 14.8.

Anal. Calcd. for $\text{C}_{21}\text{H}_{24}\text{F}_6\text{NP}$: C, 57.93; H, 5.56; N, 3.22. Found: C, 57.87; H, 5.38; N, 3.18.

1-Benzyl-4-phenyl-3,6-dimethylquinolinium Hexafluorophosphate (6h).

This compound was obtained as a pale yellow powder (71%, 1.00 g), mp 114-116°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.80 (s, 1H), 8.43 (d, $J = 8.9$ Hz, 1H), 7.98 (d, $J = 8.8$ Hz, 1H), 7.69-7.71 (m, 3H), 7.38-7.41 (m, 8H), 6.38 (s, 2H), 2.43 (s, 3H), 2.40 (s, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 156.6, 150.1, 140.3, 136.1, 135.0, 134.0, 133.8, 129.6, 129.3, 129.1, 128.9, 128.7, 128.3, 127.1, 126.6, 119.1, 59.9, 21.0, 17.7.

Anal. Calcd. for $\text{C}_{24}\text{H}_{22}\text{F}_6\text{NP}_2\text{H}_2\text{O}$: C, 57.03; H, 5.18; N, 2.77. Found: C, 57.47; H, 5.07; N, 2.62.

1-Benzyl-4-*n*-butyl-6-methylquinolinium Hexafluorophosphate (6i).

This compound was obtained as a yellow solid (62%, 642 mg), mp 143-145°; ^1H nmr (dimethyl sulfoxide- d_6): δ 9.54 (d, $J = 4.9$ Hz, 1H), 8.41 (s, 1H), 8.38 (s, 1H), 8.13 (d, $J = 4.9$ Hz, 1H), 8.03 (d, $J = 8.5$ Hz, 1H), 7.38 (s, 5H), 6.31 (s, 2H), 3.45-3.35 (m, 2H), 2.62 (s, 3H), 1.79-1.77 (m, 2H), 1.40 (q, $J = 6.7$ Hz, 2H), 0.99 (t, $J = 6.7$ Hz, 3H); ^{13}C nmr (dimethyl sulfoxide- d_6): δ 162.2, 148.0, 140.3, 137.0, 135.7, 134.1, 129.1, 128.7, 128.6, 127.1, 125.4, 121.9, 119.5, 59.6, 32.0, 31.6, 22.1, 20.9, 13.7.

Anal. Calcd. for $\text{C}_{21}\text{H}_{24}\text{F}_6\text{NP}$: C, 57.93; H, 5.56; N, 3.22. Found: C, 57.75; H, 5.51; N, 3.18.

Preparation of 1-Methyl-2,4-diphenyl-1,2-dihydroquinoline (7a), 1,3-Dimethyl-2,4-diphenyl-1,2-dihydroquinoline (7b) and 1-Methyl-2-*n*-butyl-4-phenyl-1,2-dihydroquinoline (7c) via *N*-(Benzotriazol-1-ylmethyl)-*N*-methylaniline (1a).

N-(Benzotriazol-1-ylmethyl)-*N*-methylaniline (1a) (4.5 mmoles) was dissolved in methylene chloride at 0° and phenylacetylene (3 mmoles) was added. After 10 minutes, boron trifluoride etherate (6 mmoles) was added and the mixture was stirred at this temperature for 4 hours. The septum was removed to allow the oxidation and the reaction mixture was stirred at room temperature for 3 hours. The solvent was removed and the residue was triturated with ether and put under vacuum overnight. Dry tetrahydrofuran was added (20 ml) and the reaction mixture was cooled to 0° for 20 minutes. Phenyl magnesium bromide (1.5 *M* solution in tetrahydrofuran, 15 ml, 15 mmoles) was added dropwise at 0°. After the addition was complete, the mixture was allowed to warm to room temperature and then stirred for 4 hours. Water (50 ml) was added carefully and the reaction mixture was extracted with ethyl acetate. The organic extracts were successively washed with a 1 *M* solution of sodium hydroxide and a saturated sodium chlo-

ride solution. The reaction mixture was dried with magnesium sulfate and the solvent removed. The residue was purified by column chromatography to give 1,2-dihydro-1-methyl-2,4-phenylquinoline (**7a**) as a pale yellow solid (74%, 659 mg), mp 128-130°; ¹H nmr (chloroform-*d*): δ 7.10-7.25 (m, 10H), 7.11 (t, J = 7.2 Hz, 1H), 6.90 (d, J = 7.2 Hz, 1H), 6.57 (t, J = 7.5 Hz, 1H), 6.50 (d, J = 8.1 Hz, 1H), 5.67 (d, J = 5.4 Hz, 1H), 5.17 (d, J = 5.1 Hz, 1H), 2.75 (s, 3H); ¹³C nmr (chloroform-*d*): δ 145.0, 141.9, 139.5, 136.4, 129.4, 129.0, 128.7, 128.1, 127.8, 127.3, 126.6, 126.1, 124.7, 121.9, 116.3, 110.4, 65.4, 36.6.

Anal. Calcd. for C₂₂H₁₉N C, 88.85; H, 6.44; N, 4.71. Found: C, 88.64; H, 6.62; N, 4.76.

Compound **7b** was obtained using the above procedure (with 1-phenyl-1-propyne as the unsaturated system) as a yellow solid, (81%, 1.27 g), mp 152-154°; ¹H nmr (chloroform-*d*): δ 7.03-7.40 (m, 11H), 6.50-6.53 (m, 2H), 6.40-6.43 (m, 1H), 4.82 (br s, 1H), 2.73 (s, 3H), 1.47 (s, 3H); ¹³C nmr (chloroform-*d*): δ 143.3, 140.5, 138.6, 132.2, 130.2, 130.1, 129.9, 128.5, 128.3, 128.2, 127.9, 126.8, 125.9, 124.1, 116.4, 109.8, 70.5, 36.0, 18.6; hrms: m/z Calcd for C₂₃H₂₁N 311.1674. Found: 311.1675.

Compound **7c** was obtained using the same procedure, with phenylacetylene as the unsaturated system and *n*-butylmagnesium chloride as the Grignard reagent, as a brown oil (71%); ¹H nmr (chloroform-*d*): δ 7.39-7.29 (m, 5H), 7.11 (t, J = 7.2 Hz, 1H), 6.88 (d, J = 7.5 Hz, 1H), 6.56-6.50 (m, 1H); 5.68 (d, J = 5.7 Hz, 1H), 4.08-4.01 (m, 1H); 2.92 (s, 3H), 1.67-1.64 (m, 1H); 1.56-1.49 (m, 1H); 1.33-1.30 (m, 4H); 0.89 (t, J = 6.9 Hz, 3H); ¹³C nmr (chloroform-*d*): δ 145.4, 140.4, 137.4, 129.0,

128.9, 128.0, 127.2, 125.8, 124.3, 122.6, 115.8, 110.5, 60.2, 36.6, 32.5, 26.5, 22.9, 14.0; hrms: m/z Calcd for C₂₀H₂₂N 276.1752. Found 276.1740.

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