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# Ring Fragmentation of Benzotriazol-1-yl Carbanions

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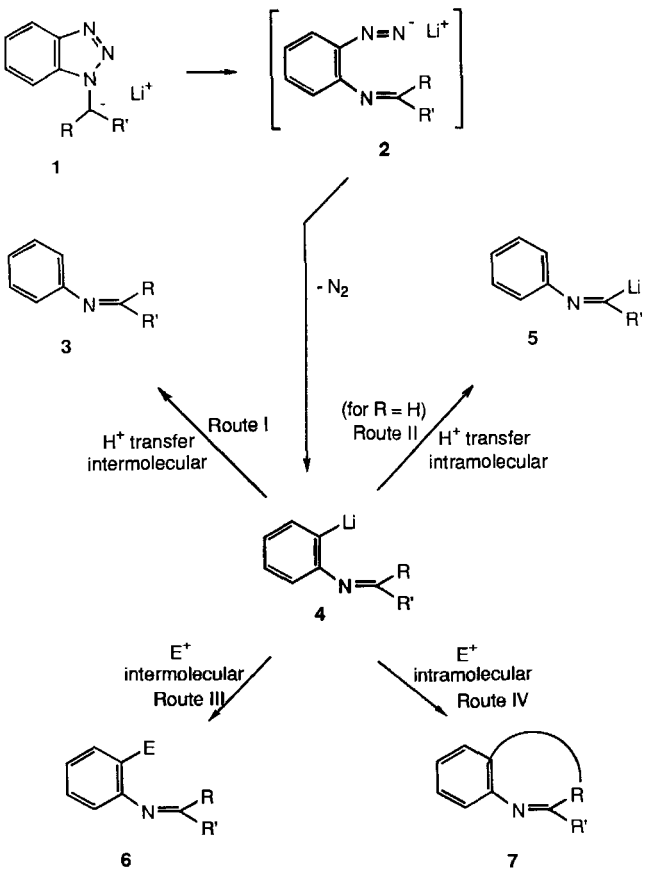
The title carbanions **1** are shown to lose nitrogen to afford *ortho*-carbiminophenyl anions **4** which can be protonated by inter- or intramolecular proton transfer or react inter- or intramolecularly with other types of electrophilic center.

The benzotriazole ring is highly stable to acids and bases, to oxidation and reduction, and to heat. Ring opening by pyrolysis requires forcing conditions (200 °C) and 1-alkylbenzotriazoles afford complex rearranged products in low yields after elimination of nitrogen[1]. Flash-vacuum pyrolyses (500–700 °C) of *N*-vinylbenzotriazoles yield *N*-phenylketene imines with loss of nitrogen[2]. Photolyses of 1-substituted benzotriazoles extrude nitrogen to form diradicals[3], which then cyclize: thus 1-phenylbenzotriazole on irradiation gave carbazole in nearly quantitative yield[4].

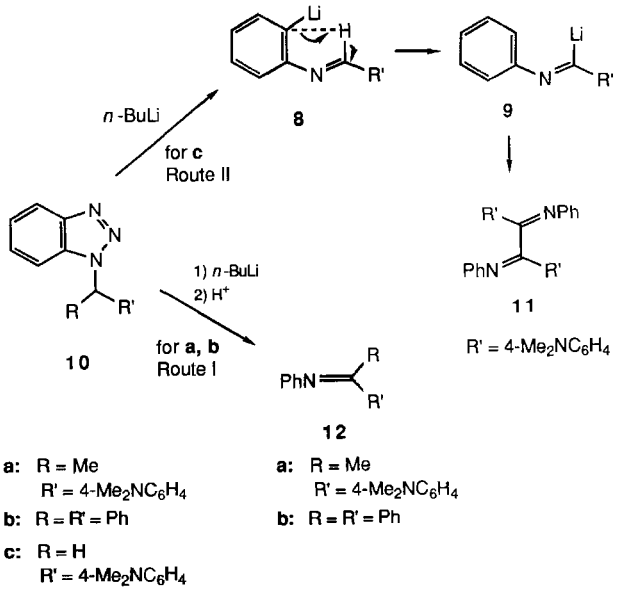
In the course of our work utilizing benzotriazoles as a synthetic auxiliary, we have only rarely observed cleavage of the benzotriazole ring. Minor amounts of phenylenediamines were observed[5] during the reaction of Grignard reagents with 1-(α-alkoxyalkyl)- and 1-[α-(aryloxy)alkyl]benzotriazoles. Reactions of 1-imidoylbenzotriazole with Grignard reagents[6] afforded a variety of products in which the triazole ring opened or was modified. In both these cases, nucleophilic attack of the Grignard reagents at the benzotriazolyl nitrogen atoms was involved.

We now report on a different type of opening of the benzotriazole ring in which benzotriazol-1-yl carbanions **1** open via **2** to lose nitrogen giving intermediates of type **4** which can pick up a proton inter- (to give **3**) or intramolecularly (to give **5**) or be trapped by various electrophilic centers inter- (to form **6**) or intramolecularly (to give **7**) (see Scheme 1).

Scheme 1




Scheme 2




Examples of routes I and II are given in Scheme 2. 4-[1-(Benzotriazol-1-yl)ethyl]-*N*,*N*-dimethylaniline (**10a**) and 1-(diphenylmethyl)benzotriazole (**10b**) on treatment with *n*-butyllithium (*n*BuLi) gave the corresponding imines **12a–b** (route I). On the other hand, 4-(benzotriazol-1-ylmethyl)-*N*,*N*-dimethylaniline (**10c**) gave the coupled product **11** in a

yield of 27%. Evidently, the aryllithium **8** deprotonates the acidic olefinic hydrogen intramolecularly (route II) to afford the new iminoalkyllithium **9**, which then couples to give **11**. The structure of **11** was confirmed by its $^{13}$C-NMR spectrum: the imino carbon resonated at $\delta$ = 164.0 and had no attached hydrogen according to the APT spectrum.

Other examples of route I and examples of routes III and IV are given in Scheme 3. Phenylmagnesium bromide and benzylmagnesium bromide displace benzotriazole in naphthol derivatives of type **15** affording the naphthols **13** in good yields as recently observed[7]. By contrast, we now find that two equivalents of methylmagnesium iodide or *n*BuLi react with **15** at −78 °C to give, after normal workup, imines **14** on route I. This could be attributed to the higher basicity of methylmagnesium iodide and *n*BuLi as compared to phenyl and benzyl Grignard reagents. Thus, while the more basic reagents deprotonate the N−C$_\alpha$ proton (forming a carbanion), the more nucleophilic reagents displace the benzotriazole. Quenching the reaction (of **15a** with *n*BuLi) with methyl iodide prior to workup gave products **16** and **17** (intermolecular nucleophilic attack on route III), of which **16** was the major product. This can be explained by the stability of the lithio phenolate in the low dielectric medium of tetrahydrofuran. Compound **16** exists as a mixture of two isomers in chloroform as indicated by the presence of two sets of signals in the NMR spectra. This phenomenon could be attributed to the hydrogen bonding between the imino nitrogen and the hydroxy group making the conversion from *syn*- to *anti*-conformer and vice versa very slow. When the spectrum was recorded in the more polar dimethyl sulfoxide, the two sets of signals coalesced. When intramolecular hydrogen bonding is not possible as in compound **17**, only one set of signals is observed in chloroform.

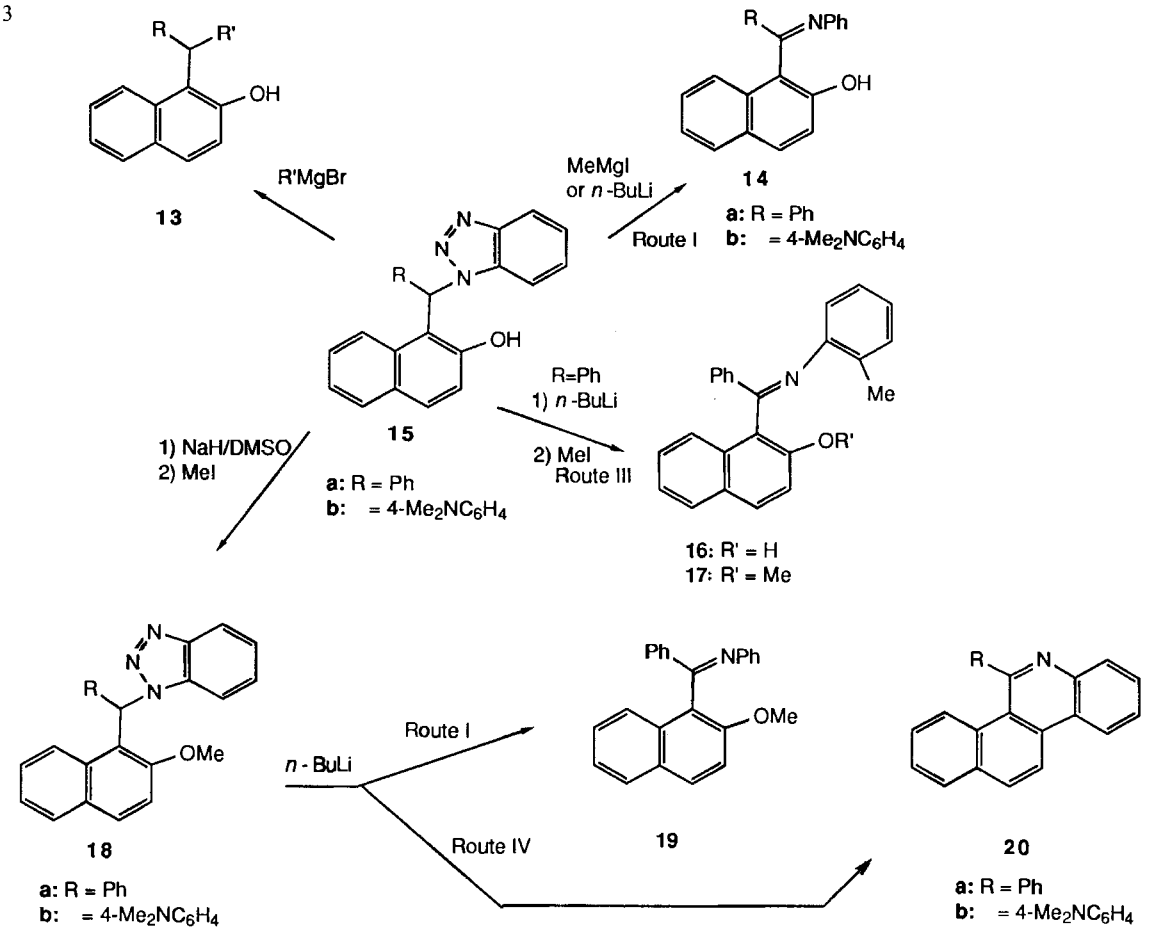
Conversion of the hydroxy group in **15** to a methoxy group (to give **18**) should favour intramolecular nucleophilic attack at this position (route IV). Treatment of **18a** with *n*BuLi at −78 °C and then at room temperature for 12 h indeed gave the cyclized product **20a** in a yield of 33%, accompanied by the imine **19** (45%). The $^1$H- and $^{13}$C-NMR spectra of **20a** displaced no signals in the aliphatic region showing that the methoxy group was not present. The structure of **20a** was further confirmed by the mass spectroscopic and elemental analysis data. Increasing the reaction time to 2 days gave the cyclized product **20a** exclusively. Similarly **20b** was obtained in a yield of 80%.

In conclusion, the presence of an anion adjacent to the benzotriazol-1-yl group makes the triazole ring susceptible to ring opening. This is the first example of benzotriazole ring opening under such mild conditions.

## Experimental

Melting points: Hot-stage microscope. All melting points are uncorrected. – $^1$H NMR: Varian VXR-300 NMR spectrometer (300 MHz) with TMS [$\delta$(TMS) = 0.00] as the internal reference. – $^{13}$C

Scheme 3



NMR: Varian VXR-300 NMR spectrometer (75 MHz), referenced to the central line of $CDCl_3$ ($\delta$ = 77.00). $CDCl_3$ was used as the solvent for both $^1H$ and $^{13}C$ NMR except when stated otherwise. – High-resolution MS: Kratos/AE1-MS 30 mass spectrometer. – Microanalyses: Carbo Erba 1106 elemental analyzer. – THF was distilled from sodium/benzophenone prior to use. – Lithiation reactions were carried out under the protection of dry nitrogen. All glassware was dried in an oven overnight prior to use. All moisture-sensitive reagents were transferred by means of pre-dried syringes. – 4-[1-(Benzotriazol-1-yl)ethyl]-*N,N*-dimethylaniline (**10a**)[8], 1-(diphenylmethyl)benzotriazole (**10b**)[9], 4-(benzotriazol-1-ylmethyl)-*N,N*-dimethylaniline (**10c**)[8], and compounds **15a,b**[7] were prepared according to literature methods. – Yields, melting points, analyses etc. see Table 1.

Table 1. Preparation of imines

| Comp. | Yield (%) | Crystal form M.p. [°C] | $\delta^{13}C$ C=N | Molecular formula (Mol. mass) | Calcd. / Found C | H | N |
|---|---|---|---|---|---|---|---|
| **11** | 27 | micro. 246–248 | 164.0 | $C_{30}H_{30}N_4$ (446.6) | 80.65 / 80.68 | 6.81 / 6.77 | 12.36 / 12.55 |
| **12a** | 74 | plates 109–111 | 164.3 | $C_{16}H_{18}N_2$ (238.3) | 80.63 / 80.28 | 7.61 / 7.81 | 11.75 / 11.46 |
| **12b** | 51 | micro. 110–112[a] | 168.2 | | | | |
| **14a** | 64(69)[b] | micro. 176–178[c] | 164.6 | | | | |
| **14b** | 78 | micro. 211–213 | 164.3 | $C_{25}H_{22}N_2O$ (366.5) | 81.94 / 81.94 | 6.05 / 6.15 | 7.64 / 7.58 |
| **16** | 89 | micro. 165–167 | 163.6 | $C_{24}H_{19}NO$ (337.4) | 85.43 / 85.20 | 5.68 / 5.84 | 4.15 / 3.81 |
| **17** | 4 | plates 102–104 | 163.3 | $C_{25}H_{21}NO$ (351.5) | 85.44 / 85.63 | 6.02 / 6.17 | 3.99 / 3.82 |
| **19** | 45 | micro. 108–110 | 165.1 | $C_{24}H_{19}NO$ (337.4) | 85.43 / 85.10 | 5.68 / 5.68 | 4.15 / 4.04 |
| **20a** | 33(82)[d] | needles 156–158 | 159.2 | $C_{23}H_{15}N$ (324.4) | 90.46 / 90.34 | 4.95 / 4.96 | 4.59 / 4.47 |
| **20b** | 78 | plates 220–222 | 159.3 | $C_{25}H_{20}N_2$ (348.5) | 86.18 / 85.99 | 5.79 / 5.76 | 8.04 / 7.84 |

[a] Ref.[10] 112–113°C. – [b] 64% (yield with MeMgI); 69% (yield with *n*-BuLi). – [c] Ref.[11] 178°C. – [d] 33% (yield with reaction time 12 h); 82% (yield with reaction time 48 h).

*General Procedure for the Preparation of Compounds* **11, 12, 14, 19,** *and* **20**: To a solution of the substrate (1.0 mmol) in 10 ml of THF was added at –78°C the appropriate amount of *n*BuLi (2.5 M in hexane) (For the preparation of **11, 12, 19,** and **20**, 1.1 mmol of *n*BuLi, for **14**, 2.2 mmol of *n*BuLi was added.). The resulting solution was allowed to warm slowly to room temp. overnight, poured into 20 ml of water, extracted with diethyl ether (3 × 30 ml), and the combined extracts were dried with $MgSO_4$. The solvent was removed in vacuo and the residue purified by chromatography on a silica gel column.

*1,2-Bis[4-(dimethylamino)phenyl]-1,2-bis(phenylimino)ethane* (**11**): From **10c** (250 mg, 1.0 mmol). The crude product was chromatographed with petroleum ether (40–60°C)/ethyl acetate (15:1); yield 60 mg (27%) of **11**. – $^1H$ NMR: $\delta$ = 3.01 (s, 12H), 6.47 (d, $J$ = 7.6 Hz, 4H), 6.65 (d, $J$ = 9.2 Hz, 4H), 6.9–7.1 (m, 6H), 7.79 (d, $J$ = 9.2 Hz, 4H). – $^{13}C$ NMR: $\delta$ = 40.1, 111.8, 120.5, 123.8, 126.2, 128.1, 130.0, 150.2, 151.8, 164.0.

*N,N-Dimethyl-4-[1-(phenylimino)ethyl]aniline* (**12a**): From **10a** (270 mg, 1.0 mmol). The crude product was chromatographed with petroleum ether (40–60°C)/ethyl acetate (12:1) to yield 176 mg (74%) of **12a**. – $^1H$ NMR: $\delta$ = 2.15 (s, 3H), 2.99 (s, 6H), 6.69 (d, $J$ = 8.8 Hz, 2H), 6.76–6.80 (m, 2H), 6.99–7.05 (m, 1H), 7.27–7.33 (m, 2H), 7.88 (d, $J$ = 8.8 Hz, 2H). – $^{13}C$ NMR: $\delta$ = 16.8, 40.1, 111.2, 119.8, 122.5, 127.1, 128.4, 128.7, 151.8, 152.3, 164.3.

*N-(Diphenylmethylene)aniline* (**12b**): From **10b** (280 mg, 1.0 mmol). The crude product was chromatographed with petroleum ether (40–60°C)/ethyl acetate (15:1); yield 130 mg (51%) of **12b**. – $^1H$ NMR: $\delta$ = 6.71 (d, $J$ = 7.4 Hz, 2H), 6.90 (t, $J$ = 7.4 Hz, 1H), 7.1–7.5 (m, 10H), 7.7–7.8 (m, 2H). – $^{13}C$ NMR: $\delta$ = 120.9, 123.1, 127.8, 128.1, 128.4, 128.5, 129.3, 129.5, 130.7, 136.2, 139.6, 151.2, 168.2.

*1-(N-Phenylbenzimidoyl)-2-naphthol* (**14a**): From **15a** (350 mg, 1.0 mmol). The crude product was chromatographed with petroleum ether (40–60°C)/ethyl acetate (10:1); yields 200 mg (64%) (with MeMgI) and 220 mg (69%) (with *n*BuLi). – $^1H$ NMR: $\delta$ = 6.7–7.0 (m, 5H), 7.1–7.5 (m, 7H), 7.6–7.8 (m, 4H), 9.46 (s, 1H). – $^{13}C$ NMR: $\delta$ = 115.8, 117.0, 118.5, 122.0, 122.4, 123.3, 125.8, 126.8, 127.1, 127.2, 127.4, 129.1, 129.7, 130.9, 138.2, 150.6, 151.6, 164.6.

*1-[4-(Dimethylamino)-N-phenylbenzimidoyl]-2-naphthol* (**14b**): From **15b** (400 mg, 1.0 mmol). The crude product was chromatographed with petroleum ether (40–60°C)/ethyl acetate (8:1); yield 280 mg (78%) of **14b**. – $^1H$ NMR: $\delta$ = 2.95 (s, 6H), 6.58 (d, $J$ = 9.2 Hz, 2H), 6.68–6.74 (m, 1H), 6.83–6.95 (m, 4H), 7.09 (d, $J$ = 8.9 Hz, 2H), 7.14–7.28 (m, 2H), 7.50 (d, $J$ = 8.4 Hz, 1H), 7.56–7.65 (m, 3H), 9.0 (broad, 1H). – $^{13}C$ NMR: $\delta$ = 39.7, 110.8, 116.9, 117.3, 119.3, 122.2, 122.3, 124.2, 125.9, 126.4, 127.2, 127.3, 129.1, 129.5, 131.7, 151.5, 151.6, 164.3.

*2-Methoxy-1-(N-phenylbenzimidoyl)naphthalene* (**19**): From **18a** (360 mg, 1.0 mmol), reaction time 12 h. **19** was obtained under the standard reaction conditions as the first fraction from a chromatography of the crude product with petroleum ether (40–60°C)/$CH_2Cl_2$ (1:1); yield 150 mg (45%). – $^1H$ NMR: $\delta$ = 3.66 (s, 3H), 6.7–6.8 (m, 2H), 6.93 (t, $J$ = 7.5 Hz, 2H), 7.07 (d, $J$ = 9.1 Hz, 1H), 7.2–7.4 (m, 6H), 7.52 (d, $J$ = 8.8 Hz, 1H), 7.63 (d, $J$ = 7.9 Hz, 1H), 7.70 (d, $J$ = 9.0 Hz, 1H), 7.74–7.79 (m, 2H). – $^{13}C$ NMR: $\delta$ = 55.9, 112.3, 119.1, 119.6, 123.2, 123.6, 124.7, 126.9, 127.8, 128.0, 128.2, 128.3, 130.4, 130.6, 131.8, 139.0, 151.4, 153.6, 165.1.

*5-Phenylbenzo[i]phenanthridine* (**20a**) was obtained from 360 mg (1.0 mmol) of **18a**: (1) in a yield of 100 mg (33%) as the second fraction from the above chromatography (compound **19**); or (2) in a yield of 250 mg (82%) after a reaction time of 48 h at room temp. from chromatography with petroleum ether (40–60°C)/$CH_2Cl_2$ (2:1). – $^1H$ NMR: $\delta$ = 7.15–7.25 (m, 1H), 7.4–7.8 (m, 9H), 7.88 (d, $J$ = 7.2 Hz, 1H), 8.09 (d, $J$ = 8.8 Hz, 1H), 8.26 (dd, $J$ = 8.2, 1.3 Hz, 1H), 8.58 (d, $J$ = 9.2 Hz, 2H). – $^{13}C$ NMR: $\delta$ = 119.8, 121.4, 122.4, 123.5, 125.8, 126.3, 126.7, 128.2, 128.3, 128.4, 128.8, 128.9, 129.0, 129.9, 130.2, 132.2, 133.1, 134.3, 144.0, 144.5, 159.2.

*5-[4-(Dimethylamino)phenyl]benzo[i]phenanthridine* (**20b**): From **18b** 410 mg (1.0 mmol). The crude product was chromatographed with petroleum ether (40–60°C)/ethyl acetate (12:1); yield 270 mg (78%) of **20b**. – $^1H$ NMR: $\delta$ = 2.96 (s, 6H), 6.76 (d, $J$ = 8.9 Hz, 2H), 7.18–7.22 (m, 1H), 7.4–7.6 (m, 4H), 7.66–7.73 (m, 1H), 7.81 (dd, $J$ = 7.9, 1.3 Hz, 1H), 8.00 (d, $J$ = 8.8 Hz, 1H), 8.05 (d, $J$ = 8.6 Hz, 1H), 8.19–8.24 (m, 1H), 8.45–8.51 (m, 2H). – $^{13}C$ NMR: $\delta$ = 40.4, 112.5, 119.6, 121.4, 122.2, 123.1, 125.4, 126.0, 126.1, 128.1, 128.3, 128.7, 129.6, 129.9, 130.6, 131.8, 132.4, 133.0, 134.1, 144.1, 150.6, 159.3.

*Procedure for the Preparation of Compounds* **16** *and* **17**: To a solution of 0.70 g (2.0 mmol) of **15a** in 40 ml of THF was added

at −78°C 1.6 ml (4.0 mmol) of *n*BuLi (2.5 M in hexane). The resulted solution was stirred at −78°C for 1 h and at room temp. for 2 h. It was again cooled to −78°C, and 0.60 g (4.2 mmol) of methyl iodide was added. After allowing the solution to reach room temp. ovcrnight, 30 ml of ammonium chloride solution (20%) was added. The organic phase was separated and the aqueous phase extracted with diethyl ether (3 × 30 ml). The combined organic phases were washed with water and dried with $MgSO_4$. The solvent was removed in vacuo to give an oily residue, which was chromatographed with petroleum ether (40−60°C)/ethyl acetate (2:1) to give compound **17** (40 mg, 4%) as the first fraction and compound **16** (0.60 g, 89%) as the third fraction.

*1-[N-(2-Methylphenyl)benzimidoyl]-2-naphthol* (**16**): From **15a** (0.70 g, 2.0 mmol), yield 0.60 g (89%) of **16**. − $^1$H NMR ([D$_6$]-DMSO): δ = 2.33 (s, 3H), 6.6−6.8 (m, 3H), 6.9−7.1 (m, 1H), 7.1−7.5 (m, 7H), 7.7−7.8 (m, 4H), 10.1 (broad, 1H). − $^{13}$C NMR ([D$_6$]DMSO): δ = 17.8, 116.0, 116.9, 117.7, 122.8, 123.3, 123.6, 125.4, 126.6, 127.3, 127.9, 128.2, 128.5, 129.1, 129.6, 130.0, 130.6, 131.7, 138.8, 149.2, 152.7, 163.6.

*1-[N-(2-Methylphenyl)benzimidoyl]-2-methoxynaphthalene* (**17**): From **15a** (0.70 g, 2.0 mmol), yield 40 mg (4%) of **17**. − $^1$H NMR: δ = 2.36 (s, 3H), 3.70 (s, 3H), 6.4−6.5 (m, 1H), 6.58−6.76 (m, 2H), 6.9−7.4 (m, 7H), 7.52−7.56 (m, 1H), 7.7−7.8 (m, 4H). − $^{13}$C NMR: δ = 17.9, 55.8, 112.4, 117.2, 119.5, 123.4, 123.5, 123.6, 124.8, 125.2, 126.8, 128.1, 128.2, 128.3, 129.5, 129.8, 130.3, 130.4, 132.5, 139.3, 149.5, 153.7, 163.3.

*General Procedure for the Preparation of Compounds* **18**: 0.24 g (6 mmol) of sodium hydride (60% in mineral oil) was added to an ice-cold solution of the corresponding **15** (5 mmol) in 10 ml of dimethyl sulfoxide. The solution was stirred at room temp. for 15 min, and 1.4 g (10 mmol) of methyl iodide was added. The resulted solution was stirred at room temp. for 10 min, poured into 30 ml of ice-cold water and extracted with diethyl ether (3 × 30 ml). The combined extracts were washed with water (4 × 30 ml) and dried with $MgSO_4$. The solvent was removed in vacuo to give the pure compound **18a** or the residue chromatographed to give pure **18b**.

*1-[α-(Benzotriazol-1-yl)benzyl]-2-methoxynaphthalene* (**18a**): From **15a** (1.76 g, 5.0 mmol), yield 1.66 g (91%) of **18a**, m.p. 167−169°C. − $^1$H NMR: δ = 3.70 (s, 3H), 7.1−7.3 (m, 11H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.83 (d, *J* = 9.0 Hz, 1H), 8.00−8.05 (m, 1H), 8.16 (d, *J* = 8.5 Hz, 1H), 8.35 (s, 1H). − $^{13}$C NMR: δ = 56.4, 58.8, 110.7, 113.1, 118.0, 119.7, 123.5, 123.7, 124.7, 126.8, 126.9, 127.4, 127.8, 128.4, 129.6, 131.5, 132.4, 134.0, 137.5, 145.6, 155.5.

$C_{24}H_{19}N_3O$ (365.4)
Calcd. C 78.88 H 5.24 N 11.50
Found C 78.77 H 5.27 N 11.37

*1-[α-(Benzotriazol-1-yl)-4-(dimethylamino)benzyl]-2-methoxynaphthalene* (**18b**): From **15b** (1.97 g, 5.0 mmol). The crude product was chromatographed with petroleum ether (40−60°C)/ethyl acetate (4:1) to give pure **18b** (1.73 g, 85%), m.p. 156−158°C. − $^1$H NMR: δ = 2.92 (s, 6H), 3.62 (s, 3H), 6.65 (d, *J* = 9.0 Hz, 2H), 7.05−7.12 (m, 3H), 7.2−7.4 (m, 5H), 7.75 (dd, *J* = 7.8, 1.1 Hz, 1H), 7.84 (d, *J* = 9.0 Hz, 1H), 7.98−8.03 (m, 1H), 8.13 (d, *J* = 8.5 Hz, 1H), 8.23 (s, 1H). − $^{13}$C NMR: δ = 40.3, 56.4, 59.6, 111.3, 112.2, 113.4, 118.9, 119.6, 123.2, 123.5, 123.6, 124.3, 126.5, 126.9, 128.5, 129.5, 129.6, 130.9, 132.4, 134.1, 145.7, 150.1, 155.3.

$C_{26}H_{24}N_4O$ (408.5)
Calcd. C 76.45 H 5.92 N 13.72
Found C 76.40 H 5.99 N 13.72

CAS Registry Numbers

**10a**: 132377-89-0 / **10b**: 73006-65-2 / **10c**: 29546-14-3 / **11**: 132377-92-5 / **12a**: 132377-93-6 / **12b**: 574-45-8 / **14a**: 15431-97-7 / **14b**: 132377-94-7 / **15a**: 132377-90-3 / **15b**: 132377-91-4 / **16**: 132377-99-2 / **17**: 132378-00-8 / **18a**: 132377-95-8 / **18b**: 132377-96-9 / **19**: 132377-97-0 / **20a**: 54607-46-4 / **20b**: 132377-98-1

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