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Arynes in Three-Component Coupling Reaction: Straightforward Synthesis of Benzo-Annulated Iminolactones

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General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL EX-270 (^{1}H, 270 MHz; ^{13}C, 67.8 MHz) spectrometer or a JEOL Lambda-400 (^{1}H, 400 MHz; ^{13}C, 99.5 MHz) spectrometer using residual chloroform (^{1}H), $CDCl_3$ (^{13}C) or THF-d_8 (^{13}C) as an internal standard. High-resolution mass spectra were obtained with a Hitachi M–80B spectrometer. The preparative recycling gel permeation chromatography was performed with GL Science PU 614 equipped with Shodex GPC H-2001L and -2002L columns (benzene as an eluent). Column chromatography was carried out using Merck Aluminium oxide 90 active neutral. Unless otherwise noted, commercially available reagents were used without purification. THF was distilled from sodium/benzophenone ketyl. 18-Crown-6 was recrystallized from distilled MeCN. KF (spray-dried) was vacuum dried at 100 °C for 12 h.

Aryne Precursors. 2-(Trimethylsilyl)phenyl triflate (**1a**),[1] 1-(trimethylsilyl)-2-naphthyl triflate (**1b**),[2] 2-(trimethylsilyl)-1-naphthyl triflate (**1c**),[3] 6-methyl-2-(trimethylsilyl)phenyl triflate (**1d**),[4] 3-methoxy-2-(trimethylsilyl)phenyl triflate (**1e**),[5] 4-methyl-2-(trimethylsilyl)phenyl triflate (**1g**),[6] 4,5-dimethyl-2-(trimethylsilyl)phenyl triflate (**1h**),[7] 6-(trimethylsilyl)-5-indanyl triflate (**1i**)[7] and 3,6-dimethoxy-2-(trimethylsilyl)phenyl triflate (**1j**)[7] were prepared according to literature procedures. 4-Fluoro-2-(trimethylsilyl)phenyl triflate (**1f**) was prepared from 2-bromo-4-fluorophenol (WAKO Chemical) in a similar manner as reported.[8]

Isocyanides. All isocyanides were synthesized from the corresponding amines by a literature method.[9]

Three-Component Coupling of Arynes, Isocyanides and Aldehydes. ***A General Procedure.*** To a THF solution (1.0 mL) of KF (0.035 g, 0.60 mmol), 18-crown-6 (0.16 g, 0.60 mmol), **2** (0.15 mmol) and **3** (0.30 mmol) was added **1** (0.30 mmol), and the resulting mixture was stirred at 0 °C. After the time specified in Table

1, Scheme 2 or 3, the mixture was diluted with ethyl acetate, filtered through a Celite plug, and concentrated. Alumina column chromatography (25% ethyl acetate/hexane as an eluent) followed by gel permeation chromatography (benzene as an eluent) gave the corresponding product.

***N*-(3-Phenyl-(3*H*)-isobenzofuranylidene)-*tert*-octylamine (4a).** Isolated in 65% yield as a white powder: ^{1}H NMR ($CDCl_3$) δ 1.03 (s, 9 H), 1.47 (s, 6 H), 1.77 (s, 2 H), 6.37 (s, 1 H), 7.13-7.17 (m, 1 H), 7.27-7.41 (m, 7 H), 7.78-7.82 (m, 1 H); ^{13}C NMR (THF-d_8) δ 30.9, 32.4, 32.7, 56.2, 57.9, 85.6, 122.9, 124.2, 127.4, 129.2, 129.5, 129.7, 131.8, 133.0, 140.7, 147.1, 152.5; HRMS Calcd for $C_{22}H_{27}NO$: M^+, 321.2093. Found: *m*/*z* 321.2100.

***N*-[3-(4-Methoxyphenyl)-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4b).** Isolated in 73% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.02 (s, 9 H), 1.44 (s, 6H), 1.75 (s, 2 H), 3.80 (s, 3 H), 6.33 (s, 1 H), 6.88 (d, *J* = 8.7 Hz, 2 H), 7.11-7.14 (m, 1 H), 7.19 (d, *J* = 8.7 Hz, 2 H), 7.38-7.41 (m, 2 H), 7.77-7.81 (m, 1 H); ^{13}C NMR (THF-d_8) δ 30.86, 30.92, 32.4, 32.7, 55.4, 56.2, 57.8, 85.5, 114.8, 123.0, 124.1, 128.9, 129.1, 131.6, 132.5, 147.2, 161.1; HRMS Calcd for $C_{22}H_{26}NO_2$: M^+-Me, 336.1964. Found: *m*/*z* 336.1985.

***N*-[3-(4-Trifluoromethylphenyl)-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4c).** Isolated in 69% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.04 (s, 9 H), 1.48 (s, 6 H), 1.78 (s, 2 H), 6.42 (s, 1 H), 7.14-7.17 (m, 1 H), 7.40-7.44 (m, 4 H), 7.64 (d, *J* = 8.5 Hz, 2 H), 7.81-7.85 (m, 1 H); ^{13}C NMR (THF-d_8) δ 30.8, 31.0, 32.4, 32.8, 56.3, 58.0, 84.5, 122.9, 124.4, 124.5 ($J_{C\text{-}F}$ = 272.1 Hz), 126.4 ($J_{C\text{-}F}$ = 3.3 Hz), 127.8, 129.0, 129.6, 131.1($J_{C\text{-}F}$ = 32.8 Hz), 132.0, 132.7, 145.2, 154.2; HRMS Calcd for $C_{23}H_{26}F_3NO$: M^+-Me, 389.1966. Found: *m*/*z* 389.1974.

***N*-[3-(3-Methoxyphenyl)-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4d).** Isolated in 65% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.04 (s, 9 H), 1.48 (s, 6 H), 1.78 (s, 2 H), 3.77 (s, 3 H), 6.34 (s, 1 H), 6.80-6.92 (m, 3 H), 7.15-7.18 (m, 1 H), 7.28-7.41 (m, 3H), 7.78-7.83 (m, 1 H); ^{13}C NMR (THF-d_8) δ 30.8, 31.0, 32.4, 32.8, 55.4, 56.2, 57.9, 85.4, 112.7, 114.6, 119.2, 122.9, 124.1, 129.2, 130.4, 131.7, 132.8, 142.3, 147.0, 154.7, 161.1; HRMS Calcd for $C_{22}H_{26}NO_2$: M^+-Me, 336.1964. Found: *m*/*z* 336.1974.

***N*-[3-(2,4-Dimethylphenyl)-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4e).** Isolated in 61% yield as a white powder: ^{1}H NMR ($CDCl_3$) δ 1.02 (s, 9 H), 1.44 (s, 6

H), 1.75 (s, 2 H), 2.31 (s, 3 H), 2.42 (s, 3 H), 6.60 (s, 1 H), 6.89-6.93 (m, 2 H), 7.04 (s, 1 H), 7.12-7.16 (m, 1 H), 7.39-7.42 (m, 2 H), 7.79-7.82 (m, 1H); ^{13}C NMR (THF-d_8) δ 19.3, 21.1, 30.8, 30.9, 32.4, 32.7, 56.2, 57.8, 83.5, 123.0, 124.2, 127.5, 128.2, 129.1, 131.6, 132.4, 133.8, 135.1, 137.1, 138.9, 146.5, 154.9; HRMS Calcd for $C_{24}H_{31}NO$: M^+, 349.2406. Found: *m*/*z* 349.2416.

***N*-[3-(1-Naphthyl)-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4f).** Isolated in 73% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.03 (s, 9 H), 1.48 (s, 6 H), 1.78 (s, 2 H), 7.19 (s, 1 H), 7.23-7.25 (m, 1 H), 7.36-7.45 (m, 4 H), 7.53-7.64 (m, 2 H), 7.84-7.94 (m, 3 H), 8.19-8.22 (m, 1 H); ^{13}C NMR (THF-d_8) δ 30.9, 31.0, 32.4, 32.7, 56.3, 57.9, 82.9, 123.1, 124.4, 125.2, 126.0, 126.7, 127.3, 128.8, 129.3, 129.7, 129.9, 131.6, 132.2, 133.5, 135.2, 136.0, 146.6, 154.6; HRMS Calcd for $C_{26}H_{29}NO$: M^+, 371.2249. Found: *m*/*z* 371.2237.

***N*-[3-(2-Thienyl)-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4g).** Isolated in 50% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.00 (s, 9 H), 1.44 (s, 3 H), 1.45 (s, 3 H), 1.71 (d, *J* = 14.4 Hz, 1 H), 1.82 (d, *J* = 14.4 Hz, 1 H), 6.64 (s, 1 H), 7.00 (dd, *J* = 4.9 Hz, 3.5 Hz, 1 H), 7.12 (d, *J* = 3.1 Hz, 1 H), 7.28-7.32 (m, 2 H), 7.41-7.49 (m, 2 H), 7.81 (d, *J* = 7.2 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 30.40, 30.44, 31.8, 32.0, 54.8, 57.5, 80.0, 122.1, 123.7, 126.0, 126.5, 126.7, 128.9, 131.0, 131.9, 142.5, 144.7, 153.5; HRMS Calcd for $C_{20}H_{25}NOS$: M^+, 327.1657. Found: *m*/*z* 327.1630.

***N*-(3-Ethyl-(3*H*)-isobenzofuranylidene)-*tert*-octylamine (4h).** Isolated in 50% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 0.96 (t, *J* = 8.0 Hz, 3 H), 1.06 (s, 9 H), 1.41 (s, 6 H), 1.70-1.77 (m, 3 H), 2.00-2.10 (m, 1 H), 5.41 (dd, *J* = 6.8 Hz, 4.1 Hz, 1 H), 7.26 (d, *J* = 7.2 Hz, 1 H), 7.35-7.39 (m, 1 H), 7.42-7.46 (m, 1 H), 7.74 (d, *J* = 7.5 Hz, 1 H); ^{13}C NMR (THF-d_8) δ 9.4, 29.5, 30.85, 30.94, 32.4, 32.7, 56.2, 57.7, 84.8, 121.9, 124.1, 129.0, 131.4, 134.0, 146.6, 154.9; HRMS Calcd for $C_{18}H_{27}NO$: M^+, 273.2093. Found: *m*/*z* 273.2088.

***N*-(3-*tert*-Butyl-(3*H*)-isobenzofuranylidene)-*tert*-octylamine (4i).** Isolated in 40% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.00 (s, 9 H), 1.03 (s, 9 H), 1.45 (s, 3 H), 1.47 (s, 3 H), 1.70 (d, *J* = 14.4 Hz, 1 H), 1.80 (d, *J* = 14.4 Hz, 1 H), 5.11 (s, 1 H), 7.36-7.43 (m, 3 H), 7.75 (d, *J* = 7.3 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 25.6, 29.9, 30.3, 31.9, 32.0, 36.2, 55.2, 57.1, 90.6, 122.6, 123.5, 128.2, 130.1, 133.8, 143.6, 154.8; HRMS Calcd for $C_{20}H_{31}NO$: M^+, 301.2406. Found: *m*/*z* 301.2423.

***N*-[3-(1-Naphthyl)-(3*H*)-isobenzofuranylidene]-*tert*-butylamine (4j).** Isolated in 49% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.44 (s, 9 H), 7.18-7.24 (m, 2 H), 7.35-7.46 (m, 4 H), 7.52-7.62 (m, 2 H), 7.84-7.94 (m, 3 H), 8.16 (d, *J* = 7.6 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 30.1, 53.8, 82.3, 122.1, 123.3, 123.8, 124.6, 125.4, 125.9, 126.6, 128.3, 128.7, 129.0, 129.2, 131.0, 131.9, 134.0, 134.4, 145.4, 156.1; HRMS Calcd for $C_{21}H_{18}NO$: M^+-Me, 300.1387. Found: *m*/*z* 300.1349.

***N*-[3-(4-Methoxyphenyl)-(3*H*)-isobenzofuranylidene]-1-adamantylamine (4k).** Isolated in 77% yield as a white powder: ^{1}H NMR ($CDCl_3$) δ 1.50-1.61 (m, 6 H), 1.97 (s, 9 H), 3.70 (s, 3 H), 6.22 (s, 1 H), 6.79 (d, *J* = 8.7 Hz, 2 H), 7.00-7.02 (m, 1 H), 7.07 (d, *J* = 8.7 Hz, 2 H), 7.28-7.32 (m, 2 H), 7.74-7.76 (m, 1 H); ^{13}C NMR (THF-d_8) δ 31.0, 37.7, 43.7, 54.8, 55.4, 85.5, 114.8, 122.9, 124.2, 128.8, 129.0, 131.7, 132.6, 133.0, 147.4, 155.5, 161.0; HRMS Calcd for $C_{25}H_{27}NO_2$: M^+, 373.2042. Found: *m*/*z* 373.2004.

***N*-[3-(4-Methoxyphenyl)-(3*H*)-benzo[*e*]isobenzofuranylidene]-1-*tert*-octylamine (4l).** Isolated in 40% (from **1b**) or 41% (from **1c**) yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 9 H), 1.54 (s, 6 H), 1.88 (s, 2 H), 3.81 (s, 3 H), 6.38 (s, 1 H), 6.89 (d, *J* = 8.6 Hz, 2 H), 7.18 (d, *J* = 8.6 Hz, 1 H), 7.23 (d, *J* = 8.6 Hz, 2 H), 7.56 (t, *J* = 6.8 Hz, 1 H), 7.68 (t, *J* = 8.4 Hz, 1 H), 7.85-7.90 (m, 2 H), 9.59 (d, *J* = 8.1 Hz, 1 H); ^{13}C NMR ($CDCl_3$) δ 30.7, 30.8, 32.0, 32.1, 54.9, 55.3, 57.9, 83.9, 114.1, 119.2, 125.0, 125.4, 126.4, 127.9, 128.1, 128.4, 129.0, 131.3, 132.0, 133.5, 145.9, 154.9, 159.9; HRMS Calcd for $C_{27}H_{31}NO_2$: M^+, 401.2355. Found: m/z 401.2325.

***N*-[3-(4-Methoxyphenyl)-4-methyl-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4m).** Isolated in 45% yield as a pale yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.02 (s, 9 H), 1.43 (s, 3 H), 1.44 (s, 3 H), 1.74 (d, *J* = 14.2 Hz, 1 H), 1.79 (d, *J* = 14.2 Hz, 1 H), 2.73 (s, 3 H), 3.80 (s, 3 H), 6.24 (s, 1 H), 6.87-6.93 (m, 3 H), 7.14-7.26 (m, 4 H); ^{13}C NMR (THF-d_8) δ 18.5, 30.5, 30.7, 31.9, 32.0, 54.9, 55.3, 57.6, 83.4, 114.0, 119.3, 128.1, 128.9, 130.1, 130.4, 132.1, 137.4, 146.5, 154.3, 159.6; HRMS Calcd for $C_{24}H_{31}NO_2$: M^+, 350.2120. Found: *m*/*z* 350.2075.

***N*-[3-(4-Methoxyphenyl)-4-methoxy-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4n).** Isolated in 16% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 0.99 (s, 9 H), 1.42 (s, 6 H), 1.73 (s, 2 H), 3.70 (s, 3 H), 3.80 (s, 3 H), 6.36 (s, 1 H), 6.82-6.87 (m, 3 H), 7.19 (d, *J* = 8.6 Hz, 2 H), 7.36-7.38(m, 2 H); ^{13}C NMR ($CDCl_3$) δ 30.1, 30.4, 31.8, 32.0, 54.8, 55.2, 55.5, 57.3, 83.4, 112.3, 113.6, 115.4, 128.5, 130.4, 130.8, 133.5,

154.3, 154.6, 159.5; HRMS Calcd for $C_{24}H_{31}NO_3$: M^+, 381.2304. Found: *m*/*z* 381.2358.

***N*-[3-(4-Methoxyphenyl)-5-fluoro-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4o).** Isolated in 38% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.02 (s, 9 H), 1.44 (s, 6 H), 1.77 (s, 2 H), 3.81 (s, 3 H), 6.29 (s, 1 H), 6.80 (dd, J = 2.2 Hz, $J_{H\text{-}F}$ = 8.1 Hz, 1 H), 6.90 (d, J = 8.6 Hz, 2 H), 7.08 (td, J = 8.9 Hz, 2.2 Hz, 1 H), 7.17 (d, J = 8.6 Hz, 2 H), 7.64-7.81 (m, 1 H); ^{13}C NMR ($CDCl_3$) δ 30.3, 30.4, 31.8, 32.0, 55.0, 55.3, 57.5, 83.9, 109.1 ($J_{C\text{-}F}$ = 24.4 Hz), 114.16, 114.23, 116.4 ($J_{C\text{-}F}$ = 23.1 Hz), 125.5 ($J_{C\text{-}F}$ = 9.8 Hz), 128.1, 130.7, 148.1 ($J_{C\text{-}F}$ = 9.8 Hz), 153.3, 160.0, 164.8 ($J_{C\text{-}F}$ = 251.0 Hz), ; HRMS Calcd for $C_{23}H_{28}FNO_2$: M^+, 369.2104. Found: m/z 369.2076.

A mixture of *N*-[3-(4-methoxyphenyl)-6-methyl-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4p) and *N*-[3-(4-methoxyphenyl)-5-methyl-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4p'). Isolated in 58% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.02 (s, 18 H), 1.44 (s, 12 H), 1.76-1.77 (m, 4 H), 2.35 (s, 3 H), 2.40 (s, 3 H), 3.798 (s, 3 H), 3.804 (s, 3 H), 6.27 (s, 1 H), 6.29 (s, 1 H), 6.86-6.91 (m, 5 H), 7.00 (d, J = 7.8 Hz, 1 H), 7.17-7.25 (s, 6 H), 7.60 (s, 1 H), 7.67 (d, J = 7.8 Hz, 1 H); ^{13}C NMR (THF-d_8) δ 21.2, 21.6, 30.96, 31.00, 31.03, 31.07, 32.4, 32.7, 55.4, 56.0, 56.1, 57.7, 57.8, 85.25, 85.35, 114.69, 114.71, 122.7, 123.2, 123.8, 124.1, 128.9, 130.1, 130.7, 132.7, 133.5, 139.1, 142.16, 144.6, 147.6, 154.7, 154.8, 161.0; HRMS Calcd for $C_{24}H_{31}NO_2$: M^+, 365.2355. Found: *m*/*z* 365.2365.

***N*-[3-(4-Methoxyphenyl)-5,6-dimethyl-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4q).** Isolated in 61% yield as a yellow oil: ^{1}H NMR ($CDCl_3$) δ 1.02 (s, 9 H), 1.44 (s, 6 H), 1.76 (d, J = 14.5 Hz, 1 H), 1.80 (s, J = 14.5 Hz, 1 H), 2.25 (s, 3 H), 2.30 (s, 3 H), 3.80 (s, 3 H), 6.26 (s, 1 H), 6.86-6.91 (m, 3 H), 7.18 (d, J = 8.7 Hz, 2 H), 7.57 (s, 1 H); ^{13}C NMR (THF-d_8) δ 19.7, 20.3, 30.5, 30.6, 31.9, 32.0, 54.5, 55.2, 57.2, 84.2, 114.0, 122.7, 123.9, 128.1, 129.8, 131.8, 137.3, 140.3, 143.9, 155.0, 159.7; HRMS Calcd for $C_{25}H_{33}NO_2$: M^+, 379.2511. Found: *m*/*z* 379.2516.

***N*-[3-(4-Methoxyphenyl)-5,6-propylen-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4r).** Isolated in 31% yield as a colorless oil: ^{1}H NMR ($CDCl_3$) δ 1.03 (s, 9 H), 1.45 (s, 6 H), 1.78 (s, 2 H), 2.09 (quintet, J = 7.24 Hz, 2 H), 2.85 (dd, J = 14.4, 7.1 Hz, 2 H), 2.92 (t, J = 7.2 Hz, 2 H), 3.80 (s, 3 H), 6.27 (s, 1 H), 6.89 (d, J = 8.7 Hz, 2 H), 6.93 (s, 1 H), 7.20 (d, J = 8.7 Hz, 2 H), 7.64 (s, 1 H); ^{13}C NMR ($CDCl_3$) δ 25.8, 30.4, 30.6, 31.9, 32.0, 32.2, 32.7, 54.7, 55.2, 57.2, 84.3, 114.0, 117.5, 118.9, 128.3, 130.3,

131.8, 144.7, 145.3, 148.4, 155.1, 159.7; HRMS Calcd for $C_{26}H_{33}NO_2$: M^+, 391.2511. Found: m/z 391.2522.

***N*-[3-(4-Methoxyphenyl)-4,7-dimethoxy-(3*H*)-isobenzofuranylidene]-*tert*-octylamine (4s).** Isolated in 26% yield as a white powder: ^{1}H NMR ($CDCl_3$) δ 1.06 (s, 9 H), 1.42 (s, 3 H), 1.44 (s, 3 H), 1.66 (d, *J* = 14.2 Hz, 1 H), 1.73 (d, *J* = 14.2 Hz, 1 H), 3.61 (s, 3 H), 3.79 (s, 3 H), 3.87 (s, 3 H), 6.27 (s, 1 H), 6.79 (s, 2 H), 6.83 (d, *J* = 8.9 Hz, 2 H), 7.19 (d, *J* = 8.9 Hz, 2 H); ^{13}C NMR (THF-d_8) δ 29.3, 29.6, 31.8, 32.0, 55.2, 55.9, 56.3, 57.7, 82.3, 112.1, 113.5, 113.8, 120.9, 128.4, 131.0, 136.2, 147.9, 150.7, 153.3, 159.3; HRMS Calcd for $C_{25}H_{33}NO_4$: M^+, 411.2410. Found: *m*/*z* 411.2430.

Three-Component Coupling Using 3j or 3k. To a THF solution (2.5 mL) of KF (0.087 g, 1.5 mmol) and 18-crown-6 (0.40 g, 1.5 mmol), **2a** (0.052 g, 0.38 mmol), **3j** (or **3k**) (0.020 g, 0.15 mmol) was added **1a** (0.22 g, 0.75 mmol), and the resulting mixture was stirred at 0 °C for 7 h. The mixture was diluted with ethyl acetate, filtered through a Celite plug, and concentrated. Alumina column chromatography (25% ethyl acetate/hexane as an eluent) followed by gel permeation chromatography (benzene as an eluent) gave the corresponding product.

1,4-Bis(1-*tert*-octylimino-1,3-dihydro-3-isobenzofuranyl)benzene (a mixture of diastereomers) (4t). Isolated in 38% yield as a yellow powder: ^{1}H NMR ($CDCl_3$) δ 1.03 (s, 18 H), 1.458 (s, 6 H), 1.464 (s, 6 H), 1.76 (s, 4 H), 6.36 (s, 1 H), 6.37 (s, 1 H), 7.13-7.15 (m, 2 H), 7.30 (d, *J* = 3.6 Hz, 4 H), 7.39-7.41 (m, 4 H), 7.79-7.81 (m, 2 H); ^{13}C NMR (THF-d_8) δ 30.27, 30.32, 30.4, 31.9, 32.0, 54.9, 55.0, 57.4, 84.1, 84.2, 121.9, 123.6, 126.8, 126.9, 128.3, 128.6, 130.9, 131.8, 131.9, 139.8, 145.3, 145.4, 154.0; HRMS Calcd for $C_{37}H_{45}N_2O_2$: M^+-Me, 549.3479. Found: *m*/*z* 549.3519.

1,3-Bis(1-*tert*-octylimino-1,3-dihydro-3-isobenzofuranyl)benzene (a mixture of diastereomers) (4u). Isolated in 35% yield as a white powder: ^{1}H NMR ($CDCl_3$) δ 0.99 (s, 18 H, *minor*), 1.04 (s, 18 H, *major*), 1.39-1.41 (m, 12 H, *minor*), 1.47-1.48 (m, 12 H, *major*), 1.75(brs, 4 H, *minor*), 1.77 (brs, 4 H, *major*), 6.34 (brs, 2 H), 7.08-7.15 (m, 2 H), 7.24-7.25 (m, 3 H), 7.34-7.42 (m, 7 H), 7.81-7.82 (m, 2 H); ^{13}C NMR ($CDCl_3$) δ 30.2, 30.3, 30.4, 31.8, 31.9, 31.995, 32.044, 55.0, 55.1, 57.4, 57.5, 84.3, 121.9, 123.7, 124.5, 124.8, 126.5, 126.8, 128.3, 128.7, 129.2, 129.3, 131.76, 131.84, 140.07, 141.12, 145.26, 145.34, 152.3, 154.2; Anal. Calcd for $C_{38}H_{48}N_2O_2$: C, 80.81; H, 8.57; N, 4.96. Found: C, 80.59; H, 8.28; N, 5.21.

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