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SUPPORTING INFORMATION

Title: Organoborane Reagents in the *C*-Alkylation of Aromatic Aldimines
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General: Melting points were determined with a Gallenkamp instrument and are given uncorrected. UV spectra were recorded with a Hewlett-Packard 8452A spectrophotometer. NMR spectra were recorded with a Bruker AC 200 (200 MHz for ^{1}H and 50.3 MHz for ^{13}C) or Bruker ARX 400 (400 MHz for ^{1}H and 100.6 MHz for ^{13}C). Chemical shifts are given relative to residual $CHCl_3$ (δ_H = 7.24 ppm) and $CDCl_3$ (δ_C = 77.0 ppm) in deuterochloroform. EI-MS data were recorded with a HP-MS 5988A spectrometer operating at 70 eV. HR-MS data were recorded with a VG Autospec spectrometer. Thin-layer chromatography was carried out on TLC aluminium sheets with silica gel 60 F_{254} (Merck). For liquid chromatography silicagel 60 (70–230 mesh) was employed. All experiments were performed under an inert atmosphere (Ar) in oven–dried glassware, sealed with a rubber septum, using anhydrous solvents.
General procedure for the synthesis of imines: A mixture of aldehyde (0.05 mL), amine (0.05 mol) in benzene (40 mL) was heated under reflux using a Dean–Stark water separator. Once the reaction ended (2 to 3 h) the solvent was removed *in vacuo*. The crude imines were used without further purification. Compounds **1a**,[1] **1b**,[2] **1c**,[3] **1d**,[4] **1e**,[3] have been described previously, and spectroscopic data were in agreement with those published.

***N*-(2,3-dimethoxyphenyl)-*N*-[(*E*)-(4-methoxyphenyl)methylidene]amine (1f)**: Yellow powder. M.p. 71–73 ºC. R_f = 0.64 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 280 (4.14), 334 (4.12). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 8.87 (s, 1 H), 7.74 (d, *J* = 7.8 Hz, 1 H), 7.26 (d, *J* = 8.7 Hz, 2 H), 7.12 (t, *J* = 7.8 Hz, 1 H), 6.98 (d, *J* = 7.8 Hz, 1 H), 6.92 (d, *J* = 8.7 Hz, 2 H), 3.90 (s, 3 H), 3.89 (s, 3 H), 3.82 (s, 3 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 158.2, 154.3, 152.7, 149.8, 145.1, 130.0, 122.3, 124.2, 118.7, 114.6, 114.2, 61.8, 55.8, 55.4 ppm. EI-MS: *m/z* (%) = 271 (7) $[M]^+$, 123 (100), 108 (88).
Reaction of imines with dicyclohexylboron chloride: General Procedure. The imine (1 mmol) was dissolved in dichloromethane (10 mL) contained in a dry, nitrogen-flushed, 25 mL round-bottomed flask. Dicyclohexylboron chloride (1 mmol, 1 M hexane solution) was added via syringe and the solution was allowed to stir for 2 h at room temp. A solution of hydrogen peroxide (0.2 mL, 30%) was then added and the mixture stirred for 30 min. Later a solution of sodium hydroxide (1 mL, 1 M) was added and stirred for 30 min. The crude reaction was washed with water (2 × 10 mL) and the organic phase dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The ^{1}H NMR of this crude exhibited the almost exclusive presence of amine derivatives (yields 80–85%). Purification by column chromatography (10% ethyl acetate in hexane) afforded the amines in 60–70% yield.
***N*-[1-cyclohexyl-1-(4-methoxyphenyl)methyl]-*p*-anisidine (2a)**: Pale yellow liquid. Yield: 60%. R_f = 0.78 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 244 (4.10), 322 (3.44). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.17 (d, *J* = 8.5 Hz, 2 H), 6.81 (d, *J* = 8.5 Hz, 2 H), 6.65 (d, *J* = 8.5 Hz, 2 H), 6.43 (d, *J* = 8.5 Hz, 2 H), 3.97 (d, *J* = 6.3 Hz, 1 H), 3.76 (s, 3 H), 3.66 (s, 3 H), 2.0–0.9 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 158.3, 151.6, 142.2, 134.8, 128.2, 114.7, 114.3, 113.5, 63.6, 55.7, 55.1, 45.0, 30.1, 29.6, 26.4 ppm. EI-MS *m/z* (%) = 325 (4) $[M]^+$, 243 (18), 242 (100), 134 (14), 121 (76). HR-MS: calcd. for $C_{21}H_{27}NO_2$ 325.2042; found 325.2047.
***N*-[1-cyclohexyl-1-(4-methoxyphenyl)methyl]-*m*-anisidine (2b):** Pale yellow liquid. Yield: 68%. R_f = 0.82 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 244 (3.47). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.16 (d, *J* = 8.5 Hz, 2 H), 6.94 (t, *J* = 8.0 Hz, 1 H), 6.80 (d, *J* = 8.5 Hz, 2 H), 6.14 (dd, *J* = 8.0, 2.4 Hz, 1 H), 6.11 (dd, *J* = 8.0, 2.4 Hz, 1 H), 6.01 (t, *J* = 2.4 Hz, 1 H), 4.02 (d, *J* = 6.1 Hz, 1 H), 3.75 (s, 3 H), 3.65 (s, 3 H), 2.0–0.8 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 160.6, 158.4, 149.2, 134.6, 129.7, 128.1, 113.6, 106.4, 102.1, 99.1, 62.8, 55.2, 54.9, 44.9, 30.1, 29.6, 26.4 ppm. EI-MS *m/z* (%) = 325 (1) $[M]^+$, 243 (21), 242 (100), 121 (65). HR-MS: calcd. for $C_{21}H_{27}NO_2$ 325.2042; found 325.2031.
***N*-[1-cyclohexyl-1-(4-methoxyphenyl)methyl]-*o*-anisidine (2c)**: Yellow liquid. Yield: 65%. R_f = 0.82 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 246 (4.33). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.18 (d, *J* = 8.5 Hz, 2 H), 6.80 (d, *J* = 8.5 Hz, 2 H), 6.72 (dd, *J* = 8.0, 1.6 Hz, 1 H), 6.66 (dt, *J* = 8.0, 1.6 Hz, 1 H), 6.53 (dt, *J* = 8.0, 1.8 Hz, 1 H), 6.29 (dd, *J* = 8.0, 1.8 Hz, 1 H), 4.74 (s, 1 H), 4.03 (d, *J* = 6.2 Hz, 1 H), 3.87 (s, 3 H), 3.75 (s, 3 H), 2.0–0.9 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 158.2, 146.5,

137.7, 134.8, 128.5, 121.1, 115.7, 113.4, 110.6, 109.1, 62.6, 55.5, 55.1, 44.9, 30.0, 29.6, 26.4 ppm. EI-MS *m/z* (%) = 325 (4) $[M]^+$, 243 (17), 242 (100), 14 (11), 121 (66). HR-MS: calcd. for $C_{21}H_{27}NO_2$ 325.2042; found 325.2055.

***N*-[1-cyclohexyl-1-(3-methoxyphenyl)methyl]-*p*-anisidine (2d)**: Pale yellow liquid. Yield: 70%. R_f = 0.82 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 246 (4.06), 3.16 (3.50). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.20 (t, *J* = 8.5 Hz, 1 H), 6.86 (d, *J* = 8.5 Hz, 1 H), 6.83 (d, *J* = 2.5 Hz, 1 H), 6.72 (dd, *J* = 8.5, 2.5 Hz, 1 H), 6.65 (d, *J* = 8.5 Hz, 2 H), 6.44 (d, *J* = 8.5 Hz, 2 H), 3.99 (d, *J* = 6.1 Hz, 1 H), 3.77 (s, 3 H), 3.67 (s, 3 H), 2.0–0.8 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 159.5, 151.6, 144.8, 142.1, 129.0, 119.8, 114.7, 114.3, 113.2, 111.5, 64.3, 55.7, 55.1, 44.8, 30.2, 29.7, 26.4 ppm. EI-MS *m/z* (%) = 325 (4) $[M]^+$, 242 (100). HR-MS: calcd. for $C_{21}H_{27}NO_2$ 325.2042; found 325.2043.

***N*-[1-cyclohexyl-1-(2-methoxyphenyl)methyl]-*p*-anisidine (2e)**: Pale yellow oil. Yield: 65%. R_f = 0.78 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 244 (4.10), 320 (3.35). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.19 (t, *J* = 8.5 Hz, 1 H), 7.16 (t, *J* = 8.5 Hz, 1 H), 6.9–6.8 (m, 2 H), 6.65 (d, *J* = 8.3 Hz, 2 H), 6.47 (d, *J* = 8.3 Hz, 2 H), 4.42 (d, *J* = 6.6 Hz, 1 H), 3.86 (s, 3 H), 3.66 (s, 3 H), 2.0–0.9 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 157.2, 151.5, 142.1, 130.7, 128.1, 127.4, 120.3, 114.6, 114.3, 110.4, 58.6, 55.7, 55.3, 43.2, 30.3, 29.7, 26.4 ppm. EI-MS *m/z* (%) = 325 (3) $[M]^+$, 243 (17), 242 (100), 121 (16). HR-MS: calcd. for $C_{21}H_{27}NO_2$ 325.2042; found 325.2042.

***N*-[1-cyclohexyl-1-(2,3-dimethoxyphenyl)methyl]-*p*-anisidine (2f)**: Pale yellow oil. Yield: 66%. R_f = 0.73 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 242 (4.20), 318 (3.48). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 6.93 (t, *J* = 7.9 Hz, 1 H), 6.78 (dd, *J* = 7.9, 1.7 Hz, 1 H), 6.74 (dd, *J* = 7.9, 1.7 Hz, 1 H), 6.65 (d, *J* = 8.5 Hz, 2 H), 6.49 (d, *J* = 8.5 Hz, 2 H), 4.36 (d, *J* = 7.3 Hz, 1 H), 3.88 (s, 3 H), 3.83 (s, 3 H), 3.62 (s, 3 H), 2.0–0.9 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 152.4, 151.5, 146.8, 142.3, 136.5, 123.4, 119.9, 114.7, 114.3, 110.5, 60.5, 59.1, 55.7, 55.5, 43.9, 30.7, 29.8, 26.5 ppm. EI-MS *m/z* (%) = 355 (3) $[M]^+$, 273 (21), 272 (100), 136 (18), 123 (19), 122 (23), 108 (15). HR-MS: calcd. for $C_{22}H_{29}NO_3$ 355.214744; found 355.2153.

***N*-[1-cyclohexyl-1-(2-hydroxy-3-methoxyphenyl)methyl]-*p*-anisidine** [5]: Yellow oil. Yield: 15%. R_f = 0.66 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 244 (4.09), 280 (3.65), 308 (3.28). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 6.8–6.5 (m, 7 H), 3.83 (s, 3 H), 4.15 (d, *J* = 7.0 Hz, 1 H), 3.65 (s, 3 H), 2.0–0.9 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 153.0, 147.1, 144.9, 141.3, 126.6, 120.8, 118.8, 116.3, 114.5, 109.3, 63.5, 55.7, 55.6, 43.6, 30.0, 29.7, 26.3 ppm. EI-MS *m/z* (%) = 341 (1) $[M]^+$, 218 (31), 138 (26), 137 (100). HR-MS: calcd. for $C_{21}H_{27}NO_3$ 341.1991; found 341.1990.

One–Pot reaction of aldehyde, amine and dialkylboron chloride: The corresponding alkene (7.6 mmol) was added to a 1 M solution of monochloroborane–methyl sulfide complex in dichloromethane (3 mL) at 0 ºC. The mixture was warmed to room temperature and stirred for 3 h. A solution of aldehyde (1 mmol) and amine (1 mmol) in dichlorometane was then added and the mixture was stirred at room temperature for 2 h. A solution of hydrogen peroxide (0.2 mL, 30%) was added and the mixture stirred for 30 min. Later a solution of sodium hydroxide (1 mL, 1 M) was added and stirred for 30 min. The crude reaction was washed with water (2 x 10 mL) and the organic phase dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The ^{1}H NMR of this crude exhibited the almost exclusive presence of amine derivatives (yields 70–80%). Purification by column chromatography (10% ethyl acetate in hexane) afforded the amines in 78–32% yield.

***N*-[1-cyclohexyl-1-(4-fluorophenyl)methyl]-*p*-anisidine (2g)**: Yellow oil. Yield: 78%. R_f = 0.78 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 272 (4.47). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.22 (dd, *J* = 8.7, 5.0 Hz, 2 H), 6.95 (t, *J* = 8.7 Hz, 2 H), 6.64 (d, *J* = 8.8 Hz, 2 H), 6.40 (d, *J* = 8.8 Hz, 2 H), 3.99 (t, *J* = 5.8 Hz, 1 H), 3.66 (s, 3 H), 1.87–0.83 (m, 11 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$) δ = 164.8, 159.2, 151.7, 141.8, 138.4, 128.6, 115.1, 114.7, 114.3, 63.6, 55.7, 44.9, 31.8, 29.6, 26.3 ppm. EI-MS *m/z* (%) = 314 (2), 313 (8) $[M]^+$, 231 (16), 230 (100), 134 (5), 109 (15). HR-MS: calcd. for $C_{20}H_{24}NOF$ 313.1842; found 313.1845.

***N*-[1-cyclohexyl-1-(4-chlorophenyl)methyl]-*p*-anisidine (2h)**: Yellow oil. Yield: 38%. R_f = 0.78 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 274 (3.47), 320 (3.11). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.24 (d, *J* = 8.8 Hz, 2 H), 7.18 (d, *J* = 8.8 Hz, 2 H), 6.64 (d, *J* = 8.8 Hz, 2 H), 6.38 (d, *J* = 8.8 Hz, 2 H), 3.99 (d, *J* = 5.8 Hz, 1 H), 3.66 (s,

3 H), 1.81–0.85 (m, 11 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 151.8, 141.6, 141.3, 132.2, 128.6, 128.3, 114.7, 114.3, 63.7, 55.7, 44.8, 30.0, 29.4, 26.3 ppm. EI-MS *m/z* (%) = 331 (4), 329 (13) $[M]^+$, 249 (7), 248 (37), 247 (19), 246 (100), 207 (12), 149 (65), 127 (10), 125 (34), 123 (17), 111 (27). HR-MS: calcd. for $C_{20}H_{24}NOCl$ 3329.1546; found 329.1547.

***N*-[1-cyclohexyl-1-(4-methylphenyl)methyl]-*p*-anisidine (2i)**: Yellow oil. Yield: 50%. R_f = 0.82 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 320 (4.02). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.14 (d, *J* = 8.5 Hz, 2 H), 7.06 (d, *J* = 8.5 Hz, 2 H), 6.63 (d, *J* = 9.2 Hz, 2 H), 6.42 (d, *J* = 9.2 Hz, 2 H), 3.98 (t, *J* = 6.5 Hz, 1 H), 3.65 (s, 3 H), 2.28 (s, 3 H), 1.88–0.86 (m, 11 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 151.5, 142.2, 134.1, 132.1, 128.7, 127.1, 114.6, 114.2, 63.9, 55.6, 44.8, 30.1, 29.5, 26.4, 21.0 ppm. EI-MS *m/z* (%) = 309 (15) $[M]^+$, 227 (31), 226 (100), 134 (16), 105 (31). HR-MS: calcd. for $C_{21}H_{27}NO$ 309.2093; found 309.2095.

***N*-[1-cyclohexyl-1-(4-methoxyphenyl)methyl]-*p*-toluidine (2j)**: Yellow oil. Yield: 62%. R_f = 0.82 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 312 (3.57). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.16 (d, *J* = 8.8 Hz, 2 H), 6.84 (d, *J* = 8.8 Hz, 2 H), 6.79 (d, *J* = 8.8 Hz, 2 H), 6.42 (d, *J* = 8.8 Hz, 2 H), 4.00 (d, *J* = 5.8 Hz, 1 H), 3.75 (s, 3 H), 2.14 (s, 3 H), 1.88–0.94 (m, 11 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 158.3, 145.0, 135.0, 129.5, 128.2, 113.5, 63.6, 55.2, 44.8, 30.0, 29.6, 26.4, 20.3 ppm. EI-MS *m/z* (%) = 309 (5) $[M]^+$, 227 (17), 226 (100), 121 (26). HR-MS: calcd. for $C_{21}H_{27}NO$ 309.2093; found. 309.2096.

***N*-[1-cyclohexyl-1-(4-methylphenyl)methyl]-*p*-toluidine (2k)**: Yellow oil. Yield: 65%. R_f = 0.86 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 280 (4.05), 334 (4.03). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.14 (d, *J* = 8.8 Hz, 2 H), 7.06 (d, *J* = 8.8 Hz, 2 H), 6.84 (d, *J* = 8.1 Hz, 2 H), 6.39 (d, *J* = 8.1 Hz, 2 H), 4.03 (d, *J* = 6.4 Hz, 1 H), 2.28 (s, 3 H), 2.14 (s, 3 H), 1.92–0.92 (m, 11 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 145.5, 139.7, 136.0, 129.5, 128.8, 127.1, 125.8, 113.2, 63.3, 44.8, 30.2, 29.4, 26.3, 21.0, 20.3 ppm. EI-MS *m/z* (%) = 293 (3) $[M]^+$, 211 (12), 210 (100). HR-MS: calcd. for $C_{21}H_{27}N$ 309.2144; found 309.2139.

***N*-(1-cyclohexyl-1-phenyl methyl) aniline (2l)**: Yellow oil. Yield: 57%. R_f = 0.84 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 304 (2.87). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.27-7.24 (m, 5 H), 7.03 (t, *J* = 7.7 Hz, 2 H), 6.57 (t, *J* = 7.7 Hz, 1 H), 6.47 (d, *J* = 7.7 Hz, 2 H), 4.09 (d, *J* = 5.8 Hz, 1 H), 1.92–0.92 (m, 11 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 147.7, 142.6, 129.0, 128.1, 127.2, 126.7, 116.9, 113.1, 63.4, 44.8, 30.2, 29.4, 26.3 ppm. EI-MS *m/z* (%) = 265 (5) $[M]^+$, 183 (14), 182 (100), 104 (6). HR-MS: calcd. for $C_{19}H_{23}N$ 265.1830; found 265.1840.

***N*-[1-(*n*-hexyl)-1-(4-methoxyphenyl)methyl]-*p*-anisidine (2m)**: Yellow oil. Yield: 26%. R_f = 0.75 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 320 (3.49). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.22 (d, *J* = 8.9 Hz, 2 H), 6.82 (d, *J* = 8.9 Hz, 2 H), 6.65 (d, *J* = 8.4 Hz, 2 H), 6.44 (d, *J* = 8.4 Hz, 2 H), 4.14 (t, *J* = 6.7 Hz, 1 H), 3.76 (s, 3 H), 3.67 (s, 3 H), 1.81-1.60 (m, 2 H), 1.31-1.16 (m, 4 H), 0.84 (t, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 158.4, 128.3, 127.9, 127.5, 114.7, 113.8, 113.5, 58.8, 55.7, 55.1, 38.8, 31.7, 29.2, 26.2, 22.6, 14.0 ppm. EI-MS *m/z* (%) = 328 (1), 327 (14) $[M]^+$, 243 (11), 242 (63), 205 (18), 134 (11), 123 (15), 122 (11), 121 (100). HR-MS: calcd. for $C_{21}H_{29}NO_2$ 327.2198; found 327.2188.

***N*-[1-(*n*-hexyl)-1-(4-fluorophenyl)methyl]-*p*-anisidine (2n)**: Yellow oil. Yield: 34%. R_f = 0.78 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 318 (3.44). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.26 (dd, *J* = 8.7, *J* = 5.0 Hz, 2 H), 6.96 (t, *J* = 8.7 Hz, 2 H), 6.65 (d, *J* = 8.7 Hz, 2 H), 6.41 (d, *J* = 8.7 Hz, 2 H), 4.16 (t, *J* = 6.6 Hz, 1 H), 3.66 (s, 3 H), 1.78–1.63 (m, 2 H), 1.36–1.16 (m, 8 H), 0.84 (t, *J* = 6.6 Hz, 3 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 162.8, 160.4, 151.9, 141.4, 140.1, 127.9, 127.8, 115.4, 115.1, 114.7, 114.4, 58.5, 55.6, 39.1, 31.7, 29.1, 26.2, 22.6, 14.0 ppm. EI-MS *m/z* (%) = 316 (6), 315 (26) $[M]^+$, 231 (23), 230 (100), 149 (79), 135 (16), 134 (15), 123 (29), 122 (23), 109 (72). HR-MS: calcd. for $C_{20}H_{26}FNO$ 315.1998; found 315.1997.

***N*-[1-(*n*-hexyl)-1-(4-methylphenyl)methyl]-*p*-anisidine (2o)**: Yellow oil. Yield: 32%. R_f **=** 0.82 (30% ethyl acetate in hexane). UV ($CHCl_3$) λ_{max} (log ε): 318 (3.44). ^{1}H NMR (200 MHz, $CDCl_3$) δ = 7.20 (d, *J* = 8.0 Hz, 2 H), 7.09 (d, *J* = 8.0 Hz, 2 H), 6.67 (d, *J* = 9.1 Hz, 2 H), 6.49 (d, *J* = 9.1 Hz, 2 H), 4.16 (t, *J* = 6.5 Hz, 1 H), 3.68 (s, 3 H), 2.30 (s, 3 H), 1.85-1.67 (m, 2 H), 1.41–1.17 (m, 8 H), 0.86 (t, *J* = 7.3 Hz, 3 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 152.2, 140.8, 136.4, 129.1, 126.4, 115.1, 114.6, 59.4, 55.6, 38.5,

31.7, 29.1, 26.3, 22.6, 21.0, 14.0 ppm. EI-MS *m/z* (%) = 312 (11), 311 (41) $[M]^+$, 227 (36), 226 (100), 134 (13), 123 (23), 105 (53). HR-MS: calcd. for $C_{21}H_{29}NO$ 311.2249; found 311.2250.

Acetylation of 2a: A mixture of acetic anhydride (5 mmol), pyridine (6 mmol) and the clean crude of **2a** (1 mmol) was stirred for 12 h at room temperature. The reaction mixture is poured over ice–water and extracted with $CHCl_3$. The organic phase was washed with HCl (2%, 2 × 10 mL) , water (2 × 10mL), $NaHCO_3$ (2%, 2 × 10 mL), water (2 × 10 mL), dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. Purification by column chromatography (10% ethyl acetate in hexane) afforded the corresponding *N*-acetyl derivative in 88% yield as yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.90 (bs, 1 H), 6.82 (d, *J* = 7.0 Hz, 2 H), 6.69 (d, *J* = 7.0 Hz, 2 H), 6.60 (bs, 1 H), 5.99 (bs, 1 H), 5.68 (d, *J* = 9.0 Hz, 1 H), 3.76 (s, 3 H), 3.73 (s, 3 H), 2.15 (m, 1 H), 1.9–1.8 (m, 2 H), 1.73 (s, 3 H), 1.65 (m, 1 H), 1.4–1.2 (m, 6 H), 0.9 (m,1 H) ppm. ^{13}C NMR (100.6 MHz, $CDCl_3$) δ = 170.9, 158.9, 158.7, 132.7, 132.4, 130.8, 130.7, 113.9, 113.1, 63.0, 55.3, 55.1, 38.1 30.7, 30.4, 26.5, 26.0, 25.9, 23.4 ppm. EI-MS *m/z* (%) = 367 (4) $[M]^+$, 284 (19), 242 (27), 203 (67), 121 (100). HR-MS: calcd. for $C_{23}H_{29}NO_3$ 367.2147; found 367.2141.

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