



SUPPLEMENTARY MATERIAL TO
**An efficient, microwave-assisted, one-pot synthesis of novel
5,6,7,8-tetrahydroquinoline-3-carbonitriles**

DIPTI K. DODIYA*, HARESH K. RAM, AMIT R. TRIVEDI and VIRESH H. SHAH

Department of Chemistry, Saurashtra University, Rajkot-360 005, Gujarat, India

J. Serb. Chem. Soc. 76 (6) (2011) 823–830

ANALYTICAL AND SPECTRAL DATA OF SYNTHESIZED COMPOUNDS

2-Methoxy-4-phenyl-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3a). Yield: 91 %; m.p. 189–192 °C; Anal. Calcd. for C₁₇H₁₆N₂O: C, 77.25; H, 6.10; N, 10.6 %. Found: C, 77.13; H, 6.02; N, 10.68 %. IR (KBr, cm⁻¹): 2225, 1606, 1263, 1066. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.66–2.83 (8H, *m*, 4 –CH₂), 3.75 (3H, *s*, –OCH₃), 7.06–7.37 (5H, *m*, Ar–H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 23.1, 23.6, 24.8, 32.1, 55.9, 96.2, 117.7, 122.6, 127.5, 130.2, 138.7, 152.2, 160.2, 163.7. MS (*m/z*): 264 (M⁺).

2-Methoxy-4-(4-chlorophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3b). Yield: 96 %; m.p. 213–215 °C; Anal. Calcd. for C₁₇H₁₅ClN₂O: C, 68.34; H, 5.06; N, 9.38 %. Found: C, 68.25; H, 5.12; N, 9.26 %. IR (KBr, cm⁻¹): 2225, 1608, 1263, 1068. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.68–2.89 (8H, *m*, 4–CH₂), 3.80 (3H, *s*, –OCH₃), 7.10–7.12 (2H, *d*, *J* = 8.36 Hz, Ar–H), 7.35–7.37 (2H, *d*, *J* = 8.32 Hz, Ar–H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 23.4, 23.8, 24.5, 32.5, 55.7, 96.5, 117.4, 123.0, 128.2, 129.5, 134.4, 136.7, 151.7, 160.5, 162.8. MS (*m/z*): 298 (M⁺).

2-Methoxy-4-(4-fluorophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3c). Yield: 92 %; m.p. 224–226 °C; Anal. Calcd. for C₁₇H₁₅FN₂O: C, 72.32; H, 5.36; N, 9.92 %. Found: C, 72.21; H, 5.28; N, 9.80 %. IR (KBr, cm⁻¹): 2222, 1604, 1278, 1066. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.62–2.90 (8H, *m*, 4–CH₂), 3.77 (3H, *s*, –OCH₃), 6.98–7.00 (2H, *t*, Ar–H), 7.24–7.26 (2H, *m*, Ar–H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 23.2, 23.7, 24.2, 31.9, 55.6, 96.2, 116.8, 117.6, 122.8, 129.3, 133.8, 151.4, 159.9, 162.9, 163.5. MS (*m/z*): 282 (M⁺).

* Corresponding author. E-mail: diptid85@gmail.com

2-Methoxy-4-(4-methoxyphenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3d). Yield: 89 %; m.p. 193–195 °C; Anal. Calcd. for C₁₈H₁₈N₂O₂: C, 73.45; H, 6.16; N, 9.52 %. Found: C, 73.36; H, 6.03; N, 9.39 %. IR (KBr, cm⁻¹): 2227, 1597, 1263, 1066. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.71–2.81 (8H, *m*, 4-CH₂), 3.75 (3H, *s*, -OCH₃), 3.89 (3H, *s*, -OCH₃), 6.76–6.78 (2H, *d*, *J* = 7.96 Hz, Ar-H), 6.98–7.00 (2H, *d*, *J* = 8.08 Hz, Ar-H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 23.4, 23.9, 24.6, 32.3, 55.7, 96.5, 114.9, 117.1, 122.8, 129.1, 130.5, 151.5, 160.3, 161.7, 162.8. MS (*m/z*): 294 (M⁺).

2-Methoxy-4-(4-hydroxyphenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3e). Yield: 94 %; m.p. 207–210 °C; Anal. Calcd. for C₁₇H₁₆N₂O₂: C, 77.67; H, 6.52; N, 10.06 %. Found: C, 77.72; H, 6.41; N, 9.93 %. IR (KBr, cm⁻¹): 2231, 1606, 1276, 1037. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.62–2.85 (8H, *m*, 4-CH₂), 3.80 (3H, *s*, -OCH₃), 4.71 (1H, *s*, -OH), 7.14–7.16 (2H, *d*, *J* = 8.1 Hz, Ar-H), 7.23–7.25 (2H, *d*, *J* = 8.15 Hz, Ar-H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 23.2, 23.7, 24.2, 31.9, 55.4, 95.8, 117.3, 122.6, 127.3, 129.8, 136.1, 138.5, 151.6, 160.3, 162.8. MS (*m/z*): 280 (M⁺).

2-Ethoxy-4-phenyl-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3f). Yield: 89 %; m.p. 233–235 °C; Anal. Calcd. for C₁₈H₁₈N₂O: C, 77.67; H, 6.52; N, 10.06 %; Found: C, 77.52; H, 6.38; N, 10.18 %. IR (KBr, cm⁻¹): 2225, 1627, 1292, 1097. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.31–1.36 (3H, *t*, -CH₃), 1.64–2.85 (8H, *m*, 4-CH₂), 4.39–4.45 (2H, *q*, -CH₂), 6.91–7.44 (5H, *m*, Ar-H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 14.3, 23.1, 23.9, 24.0, 31.5, 65.4, 96.5, 117.4, 122.8, 127.8, 129.8, 138.3, 152.2, 159.9, 162.8. MS (*m/z*): 278 (M⁺).

2-Ethoxy-4-(4-chlorophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3g). Yield: 94 %; m.p. 216–218 °C; Anal. Calcd. for C₁₈H₁₇ClN₂O: C, 69.12; H, 5.48; N, 8.96 %. Found: C, 69.19; H, 5.42; N, 8.89 %. IR (KBr, cm⁻¹): 2222, 1602, 1278, 1064. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.40–1.44 (3H, *t*, -CH₃), 1.68–2.85 (8H, *m*, 4-CH₂), 4.44–4.50 (2H, *q*, CH₂), 6.98–7.00 (2H, *d*, *J* = 7.9 Hz, Ar-H), 7.23–7.25 (2H, *d*, *J* = 8.28 Hz, Ar-H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 14.5, 22.9, 23.8, 24.7, 32.1, 65.4, 96.1, 117.7, 122.8, 128.4, 129.1, 134.8, 137.1, 151.7, 161.2, 162.9. MS (*m/z*): 312 (M⁺).

2-Ethoxy-4-(4-fluorophenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3h). Yield: 92 %; m.p. 244–226 °C; Anal. Calcd. for C₁₈H₁₇FN₂O: C, 72.95; H, 5.78; N, 9.45 %. Found: C, 72.87; H, 5.69; N, 9.52 %. ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.41–1.44 (3H, *t*, -CH₃), 1.63–2.88 (8H, *m*, 4-CH₂), 4.45–4.51 (2H, *q*, -CH₂), 7.26–7.28 (2H, *t*, Ar-H), 7.82–7.84 (2H, *t*, Ar-H). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 14.4, 23.1, 23.8, 24.5, 31.5, 64.9, 96.4, 117.1, 117.8, 123.1, 129.6, 133.7, 151.2, 160.2, 162.7, 163.2. MS (*m/z*): 296 (M⁺).

2-Ethoxy-4-(4-methoxyphenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3i). Yield: 92 %; m.p. 188–190 °C; Anal. Calcd. for C₁₉H₂₀N₂O₂: C, 74.00; H, 6.54; N, 9.08 %. Found: C, 73.89; H, 6.45; N, 9.12 %. IR (KBr, cm⁻¹): 2227, 1589,

1296, 1093. $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$, δ / ppm): 1.41–1.44 (3H, *t*, $-\text{CH}_3$), 1.62–2.87 (8H, *m*, $4-\text{CH}_2$), 3.80 (3H, *s*, $-\text{OCH}_3$), 4.45–4.48 (2H, *q*, $-\text{CH}_2$), 7.49–7.51 (2H, *d*, $J = 8.13$ Hz, Ar-H), 7.78–7.80 (2H, *d*, $J = 8.32$ Hz, Ar-H). $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$, δ / ppm): 14.5, 23.2, 23.9, 24.2, 31.9, 55.7, 65.2, 96.5, 114.6, 117.5, 123.1, 129.4, 130.3, 151.2, 160.8, 161.9, 162.5. MS (m/z): 308 (M^+).

2-Ethoxy-4-(4-hydroxyphenyl)-5,6,7,8-tetrahydroquinoline-3-carbonitrile (3j).

Yield: 88 %; m.p. 232–234 °C; Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_2$: C, 78.05; H, 6.89; N, 9.58 %; Found: C, 78.17; H, 6.72; N, 9.43 %. IR (KBr, cm^{-1}): 2222, 1635, 1278, 1097. $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$, δ / ppm): 1.27–1.31 (3H, *t*, $-\text{CH}_3$), 1.63–2.85 (8H, *m*, $4-\text{CH}_2$), 4.15–4.20 (2H, *q*, $-\text{CH}_2$), 4.77 (1H, *s*, $-\text{OH}$), 6.76–6.78 (2H, *d*, $J = 7.89$ Hz, Ar-H), 6.98–7.00 (2H, *d*, $J = 8.03$ Hz, Ar-H). $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$, δ / ppm): 14.8, 22.9, 23.2, 23.9, 31.4, 64.9, 95.9, 117.5, 122.2, 127.4, 129.8, 135.8, 139.1, 151.2, 160.7, 163.2. MS (m/z): 294 (M^+).