

Electrocatalytic dimerization of diarylacetonitriles mediated by sodium halides

Michail N. Elinson*, **Alexey I. Ilovaisky**, **Valentina M. Merkulova**, **Tatiana A. Zaimovskaya**, **Pavel A. Belyakov** and **Gennady I. Nikishin**

Tetraphenylsuccinonitrile 2a

White solid; yield 0.96 g (100%); mp 209–212 °C (lit. ¹ mp 208–211 °C).

¹H NMR (300 MHz, CDCl₃) δ : 7.20–7.40 (m, 20H, Ar).

2,3-Bis(4-methylphenyl)-2,3-diphenylsuccinonitrile 2b

White solid; yield 0.98 g (95%); mp 197–199 °C (lit. ² mp 149 °C).

¹H NMR (300 MHz, CDCl₃) δ : 2.33 (s, 3H, CH₃), 2.34 (s, 3H, CH₃), 7.02–7.09 (m, 4H, Ar), 7.13–7.34 (m, 14H, Ar).

¹³C NMR (75 MHz, CDCl₃) δ : 20.95 (2CH₃), 58.92 (2C), 121.21 (2CN), 127.92 (4CH), 128.35 (2CH), 128.66 (4CH), 129.89 (2CH), 129.94 (2CH), 129.96 (2CH), 130.02 (2CH), 133.99 (C), 134.01 (C), 137.20 (C), 137.22 (C), 138.40 (2C).

Found (%): C, 87.17; H, 5.74; N, 6.64. Calc. for C₃₀H₂₄N₂ (%): C, 87.35; H, 5.86; N, 6.79.

2,3-Bis(4-chlorophenyl)-2,3-diphenylsuccinonitrile 2c

White solid; yield 1.10 g (97%); mp 187–189 °C.

¹H NMR (300 MHz, CDCl₃) δ : 7.10–7.40 (m, 18H, Ar).

¹³C NMR (75 MHz, CDCl₃) δ : 58.64 (2C), 120.49 (2CN), 128.32 (4CH), 128.34 (4CH), 128.82 (CH), 128.95 (CH), 129.82 (2CH), 129.88 (2CH), 131.31 (2CH), 131.37 (2CH), 134.98 (C), 135.13 (C), 135.26 (C), 135.28 (C), 136.14 (C), 136.15 (C).

IR (KBr, ν /cm⁻¹): 3060, 2242, 1496, 1448, 1408, 1104, 1016, 804, 752, 700.

Found (%): C, 74.37; H, 4.24; Cl, 15.36; N, 5.94. Calc. for C₂₈H₁₈Cl₂N₂ (%): C, 74.18; H, 4.00; Cl, 15.64; N, 6.18.

2,3-Bis(4-bromophenyl)-2,3-diphenylsuccinonitrile 2d

White solid; yield 1.26 g (93%); mp 170 °C (lit. ² mp 170 °C).

¹H NMR (300 MHz, CDCl₃) δ : 7.10–7.18 (m, 4H, Ar), 7.25–7.47 (m, 14H, Ar).

^{13}C NMR (75 MHz, CDCl_3) δ : 58.66 (2C), 120.43 (2CN), 123.24 (C), 123.40 (C), 128.32 (2CH), 128.35 (2CH), 128.84 (CH), 128.96 (CH), 129.81 (2CH), 129.86 (2CH), 131.28 (2CH), 131.31 (2CH), 131.56 (2CH), 131.62 (2CH), 135.76 (C), 135.79 (C), 136.01 (C), 136.03 (C).

2,3-Bis(4-iodophenyl)-2,3-diphenylsuccinonitrile 2e

White solid; yield 1.41 g (89%); mp 166–167 °C.

^1H NMR (300 MHz, CDCl_3) δ : 6.99 (d, $J = 8.8$ Hz, 4H, Ar), 7.23–7.38 (m, 10H, Ar), 7.55–7.65 (m, 4H, Ar).

^{13}C NMR (75 MHz, CDCl_3) δ : 58.70 (2C), 95.12 (C), 95.27 (C), 120.38 (2CN), 128.28 (2CH), 128.34 (2CH), 128.82 (CH), 128.93 (CH), 129.81 (2CH), 129.86 (2CH), 131.68 (2CH), 131.72 (2CH), 135.97 (C), 136.00 (C), 136.45 (C), 136.51 (C), 137.21 (2CH), 137.27 (2CH).

IR (KBr, ν/cm^{-1}): 3060, 2240, 1488, 1448, 1396, 1004, 800, 752, 696.

Found (%): C, 52.98; H, 3.06; I, 40.02; N, 4.22. Calc. for $\text{C}_{28}\text{H}_{18}\text{I}_2\text{N}_2$ (%): C, 52.86; H, 2.85; I, 39.89; N, 4.40.

2,3-Bis(2,4-dimethylphenyl)-2,3-diphenylsuccinonitrile 2f

White solid; yield 0.96 g (87%); mp 198–202 °C.

^1H NMR (300 MHz, CDCl_3) δ : 1.91 (s, 6H, CH_3), 2.19 (s, 6H, CH_3), 6.52–6.65 (m, 2H, Ar), 6.90–7.03 (m, 2H, Ar), 7.03–7.17 (m, 6H, Ar), 7.21–7.34 (m, 4H, Ar), 7.65 (s, 2H, Ar).

^{13}C NMR (75 MHz, CDCl_3) δ : 21.15 (2 CH_3), 21.61 (2 CH_3), 58.67 (2C), 119.39 (2CN), 126.52 (2CH), 127.80 (C), 127.94 (C), 128.00 (2CH), 128.48 (2CH), 128.74 (C), 128.79 (C), 129.30 (2CH), 130.01 (2CH), 131.18 (2CH), 132.31 (2CH), 133.09 (2CH), 133.89 (C), 134.12 (C), 134.94 (C), 135.87 (C).

IR (KBr, ν/cm^{-1}): 3060, 2924, 2236, 1492, 1448, 820, 744, 724, 704.

Found (%): C, 87.41; H, 6.68; N, 6.20. Calc. for $\text{C}_{32}\text{H}_{28}\text{N}_2$ (%): C, 87.24; H, 6.41; N, 6.36.

2,3-Bis(4-methoxyphenyl)-2,3-diphenylsuccinonitrile 2g

White solid; yield 0.90 g (81%); mp 194–195 °C.

^1H NMR (300 MHz, CDCl_3) δ : 3.80 (s, 3H, OCH_3), 3.81 (s, 3H, OCH_3), 6.74–6.81 (m, 4H, Ar), 7.13–7.21 (m, 4H, Ar), 7.22–7.37 (m, 10H, Ar).

^{13}C NMR (75 MHz, CDCl_3) δ : 55.21 (2 OCH_3), 58.75 (C), 58.77 (C), 113.20 (2CH), 113.28 (2CH), 121.25 (2CN), 127.94 (2CH), 128.01 (2CH), 128.31 (CH), 128.37 (CH), 128.86 (C), 128.89 (C), 129.85 (2CH), 130.01 (2CH), 131.27 (2CH), 131.44 (2CH), 137.27 (C), 137.32 (C), 159.37 (C), 159.42 (C).

IR (KBr, ν/cm^{-1}): 3060, 2932, 2840, 2244, 1608, 1512, 1304, 1256, 1184, 1036.

Found (%): C, 81.28; H, 5.56; N, 6.12. Calc. for $\text{C}_{30}\text{H}_{24}\text{N}_2\text{O}_2$ (%): C, 81.06; H, 5.44; N, 6.30.

Methoxy(4-methoxyphenyl)phenylacetonitrile 3

Colourless oil; yield 1.11 g (88%); R_f 0.6 (hexane–ethyl acetate, 4:1).

^1H NMR (300 MHz, CDCl_3) δ : 3.43 (s, 3H, OCH_3), 3.82 (s, 3H, OCH_3), 6.91 (d, $J = 8.8$ Hz, 2H, Ar), 7.33–7.45 (m, 5H, Ar), 7.47–7.54 (m, 2H, Ar).

^{13}C NMR (75 MHz, CDCl_3) δ : 53.85 (OCH_3), 55.15 (OCH_3), 82.05 (C), 113.91 (2CH), 118.26 (CN), 126.33 (2CH), 127.85 (2CH), 128.56 (2CH), 128.85 (CH), 130.62 (C), 138.87 (C), 159.96 (C).

IR (film, ν/cm^{-1}): 2936, 2832, 2240, 1608, 1512, 1308, 1256, 1176, 1076, 1032.

MS (m/z , relative intensity %): 253 (25) $[\text{M}]^+$, 222 (80) $[\text{M}-\text{OCH}_3]^+$, 195 (15), 176 (30), 135 (28), 105 (26), 92 (24), 77 (100), 51 (49).

Found (%): C, 76.08; H, 6.15; N, 5.31. Calc. for $\text{C}_{16}\text{H}_{15}\text{NO}_2$ (%): C, 75.87; H, 5.97; N, 5.53.

References

1. A. J. Papa, *J. Org. Chem.*, 1966, **31**, 1430.
2. W. Hochstein and U. Schöllkopf, *Liebigs Ann. Chem.*, 1978, 1823.