



SUPPLEMENTARY MATERIAL TO
**The synthesis of some new hydrazone derivatives
containing the benzothiazole moiety**

AHMET ÖZDEMİR^{1*}, GÜLHAN TURAN-ZITOUNI¹, ZAFER ASIM KAPLANCIKLİ¹
and MEHLİKA DİLEK ALTINTOP¹

¹Anadolu University, Faculty of Pharmacy, Department of
Pharmaceutical Chemistry, 26470, Eskişehir, Turkey

J. Serb. Chem. Soc. 77 (2) (2012) 141–146

CHARACTERISTICS AND SPECTRAL DATA OF THE SYNTHESIZED COMPOUNDS

2-Oxo-3(2H)-benzothiazoleacetic acid, [2-(4-methylphenoxy)ethylidene]hydrazide (1a). Yield 71 %; m.p. 213–214 °C; Anal. Calcd. for C₁₈H₁₇N₃O₃S: C, 60.83; H, 4.82; N, 11.82 %. Found: C, 60.70; H, 4.79; N, 11.74 %; IR (KBr, cm⁻¹): 3195, 1770, 1682, 1525, 1490; ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 2.25 (3H, *s*), 4.63–4.70 (2H, *m*), 4.73 and 5.02 (2H, *s*), 6.86–6.95 (2H, *m*), 7.07–7.15 (2H, *m*), 7.17–7.27 (2H, *m*), 7.30–7.38 (1H, *m*), 7.55–7.61 (1H, *t*, *J* = 5.0 Hz), 7.64–7.68 (1H, *m*), 11.73 (1H, *m*); ¹³C-NMR (100 MHz, DMSO-*d*₆ / δ ppm): 20.05 (CH₃), 43.23 (CH₂), 66.72 (CH₂), 111.40 (CH), 114.53 (2CH), 121.09, 122.73 (CH), 123.12 (CH), 126.51 (CH), 129.79, 129.89 (2CH), 137.22, 143.40 (CH), 155.75, 167.18, 162.66; MS-FAB⁺ (*m/z*): 356 (M+1).

2-Oxo-3(2H)-benzothiazoleacetic acid, [2-(4-tert-butylphenoxy)ethylidene]hydrazide (1b). Yield 88 %; m.p. 209–211 °C; Anal. Calcd. for C₂₁H₂₃N₃O₃S: C, 63.46; H, 5.83; N, 10.57 %. Found: C, 63.79; H, 5.72; N, 10.64 %; IR (KBr, cm⁻¹): 3181, 1769, 1681, 1545, 1510; ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.25 (9H, *s*), 4.70–4.74 (2H, *m*), 4.76 and 5.06 (2H, *s*), 6.90–6.97 (2H, *m*), 7.17–7.40 (5H, *m*), 7.56–7.63 (1H, *t*, *J* = 5.0 Hz), 7.64–7.68 (1H, *m*), 11.74–11.79 (1H, *m*); ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 31.28 (3CH₃), 33.75, 43.25 (CH₂), 66.68 (CH₂), 111.40 (CH), 114.17 (2CH), 121.11, 122.72 (CH), 123.12 (CH), 126.15 (2CH), 126.50 (CH), 137.22, 143.26, 143.45 (CH), 155.60, 167.16, 162.65; MS-FAB⁺ (*m/z*): 398 (M+1).

2-Oxo-3(2H)-benzothiazoleacetic acid, [2-(4-methoxyphenoxy)ethylidene]hydrazide (1c). Yield 70 %; m.p. 203–204 °C; Anal. Calcd. for C₁₈H₁₇N₃O₄S: C, 58.21; H, 4.61; N, 11.31 %. Found: C, 58.33; H, 4.70; N, 11.38 %; IR (KBr, cm⁻¹):

* Corresponding author. E-mail: ahmeto@anadolu.edu.tr

3175, 1770, 1683, 1602, 1500; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$, δ / ppm): 3.70 (3H, s), 4.62–4.69 (2H, m), 4.73 and 5.02 (2H, s), 6.86–7.00 (4H, m), 7.17–7.27 (2H, m), 7.30–7.38 (1H, m), 7.54–7.72 (2H, m), 11.75–11.78 (1H, bs); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$, δ / ppm): 43.24 (CH_2), 55.32 (CH_3), 67.25 (CH_2), 111.40 (CH), 114.63 (2CH), 115.70 (2CH), 121.08, 122.74 (CH), 123.13 (CH), 126.51 (CH), 137.22, 143.54 (CH), 151.83, 153.72, 167.18, 162.65; MS-FAB⁺ (m/z): 372 (M+1).

2-Oxo-3(2H)-benzothiazoleacetic acid, [2-(2,4-dimethylphenoxy)ethylidene]-hydrazide (Id). Yield 77 %; m.p. 218–220 °C; Anal. Calcd. for $\text{C}_{19}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$: C, 61.77; H, 5.18; N, 11.37 %. Found: C, 61.79; H, 5.13; N, 11.34 %; IR (KBr, cm^{-1}): 3190, 1768, 1682, 1555, 1498; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$, δ / ppm): 2.15 (3H, s), 2.21 (3H, s), 4.66–4.71 (2H, m), 4.75 and 5.02 (2H, s), 6.85–7.00 (3H, m), 7.17–7.39 (3H, m), 7.56–7.69 (1H, t, $J = 5.0$ Hz), 7.70 (1H, d, $J = 7.7$ Hz), 11.71–11.77 (1H, bs); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$, δ / ppm): 15.93 (CH_3), 20.05 (CH_3), 43.22 (CH_2), 67.02 (CH_2), 111.41 (CH), 111.77 (CH), 121.07, 122.74 (CH), 123.13 (CH), 125.66, 126.51 (CH), 127.09 (CH), 129.42, 131.29 (CH), 137.23, 143.57 (CH), 153.87, 167.17, 162.65; MS-FAB⁺ (m/z): 370 (M+1).

2-Oxo-3(2H)-benzothiazoleacetic acid, [2-(2,6-dimethylphenoxy)ethylidene]-hydrazide (Ie). Yield 79 %; m.p. 226–227 °C; Anal. Calcd. for $\text{C}_{19}\text{H}_{19}\text{N}_3\text{O}_3\text{S}$: C, 61.77; H, 5.18; N, 11.37 %. Found: C, 61.97; H, 5.11; N, 11.39 %; IR (KBr, cm^{-1}): 3339, 1770, 1680, 1595, 1486; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$, δ / ppm): 2.26 (6H, s), 4.46–4.53 (2H, m), 4.73 and 5.01 (2H, s), 6.92–6.99 (1H, m), 7.02–7.08 (2H, m), 7.18–7.40 (3H, m), 7.63–7.70 (1H, m), 7.81–7.89 (1H, t, $J = 5.2$ Hz), 11.73 (1H, m); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$, δ / ppm): 16.13 (2 CH_3), 43.26 (CH_2), 71.08 (CH_2), 111.40 (CH), 121.07, 122.72 (CH), 123.11 (CH), 124.07 (CH), 126.50 (CH), 128.80 (2CH), 130.30 (2C), 137.23, 143.76 (CH), 155.23, 167.19, 162.71; MS-FAB⁺ (m/z): 370 (M+1).

2-Thioxo-3(2H)-benzothiazoleacetic acid, [2-(4-methylphenoxy)ethylidene]-hydrazide (If). Yield 80 %; m.p. 138–140 °C; Anal. Calcd. for $\text{C}_{18}\text{H}_{17}\text{N}_3\text{O}_2\text{S}_2$: C, 58.20; H, 4.61; N, 11.31 %. Found: C, 58.42; H, 4.47; N, 11.18 %; IR (KBr, cm^{-1}): 3200, 1683, 1588, 1523, 1491; $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$, δ / ppm): 2.22 (3H, s), 4.27–4.58 (2H, s), 4.67–4.68 (2H, d, $J = 5.0$ Hz), 6.86–6.92 (2H, m), 7.09 (2H, d, $J = 8.2$ Hz), 7.33–7.40 (1H, m), 7.43–7.49 (1H, m), 7.56–7.75 (1H, t, $J = 5.0$ Hz), 7.84 (1H, d, $J = 8.2$ Hz), 8.01 (1H, d, $J = 7.3$ Hz), 11.70–11.94 (1H, 2bs); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$, δ / ppm): 20.04 (CH_3), 35.21 (CH_2), 66.73 (CH_2), 114.52 (2CH), 121.09 (CH), 121.75 (CH), 124.43 (CH), 126.32 (CH), 129.73 (2CH), 134.69, 143.00 (CH), 152.47, 152.56, 155.74, 163.18, 165.83; MS-FAB⁺ (m/z): 372 [M+1].

2-Thioxo-3(2H)-benzothiazoleacetic acid, [2-(4-tert-butylphenoxy)ethylidene]-hydrazide (Ig). Yield 78 %; m.p. 142–144 °C; Anal. Calcd. for $\text{C}_{21}\text{H}_{23}\text{N}_3\text{O}_2\text{S}_2$: C, 60.99; H, 5.61; N, 10.16 %. Found: C, 61.00; H, 5.64; N, 10.18 %; IR (KBr,

cm⁻¹): 3178, 1682, 1574, 1523, 1487; ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 1.24 (9H, *s*), 4.27–4.58 (2H, *s*), 4.70 (2H, *d*, *J* = 5.0 Hz), 6.88–6.95 (2H, *m*), 7.27–7.32 (2H, *m*), 7.34–7.40 (1H, *m*), 7.43–7.50 (1H, *m*), 7.56–7.75 (1H, *t*, *J* = 5.0 Hz), 7.84–7.85 (1H, *2d*, *J* = 8.1 Hz), 8.02 (1H, *t*, *J* = 7.3 Hz), 11.70–11.97 (1H, *2bs*); ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 31.26 (3CH₃), 33.73, 35.18 (CH₂), 66.68 (CH₂), 114.16 (2CH), 121.08 (CH), 121.78 (CH), 124.44 (CH), 126.11 (2CH), 126.32 (CH), 134.67, 143.07 (CH), 143.19, 152.46, 155.60, 163.15, 165.85; MS-FAB⁺ (*m/z*): 414 [M+1].

2-Thioxo-3(2H)-benzothiazoleacetic acid, [2-(4-methoxyphenoxy)ethylidene]hydrazide (Ih). Yield 81 %; m.p. 145–147 °C; Anal. Calcd. for C₁₈H₁₇N₃O₃S₂: C, 55.80; H, 4.42; N, 10.84 %. Found: C, 55.97; H, 4.53; N, 10.92 %; IR (KBr, cm⁻¹): 3196, 1683, 1566, 1518, 1490; ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 3.70 (3H, *s*), 4.26–4.58 (2H, *s*), 4.65 (2H, *d*, *J* = 5.0 Hz), 6.84–6.90 (2H, *m*), 6.95–6.98 (2H, *m*), 7.34–7.40 (1H, *m*), 7.43–7.49 (1H, *m*), 7.55–7.74 (1H, *t*, *J* = 5.0 Hz), 7.84–7.85 (1H, *d*, *J* = 7.8 Hz), 8.02 (1H, *t*, *J* = 7.0 Hz), 11.69–11.96 (1H, *2bs*); ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 35.21 (CH₂), 55.29 (CH₃), 67.24 (CH₂), 114.59 (2CH), 115.67 (2CH), 121.08 (CH), 121.78 (CH), 124.44 (CH), 126.33 (CH), 134.67, 143.15 (CH), 151.81, 152.46, 153.70, 163.16, 165.85; MS-FAB⁺ (*m/z*): 388 [M+1].

2-Thioxo-3(2H)-benzothiazoleacetic acid, [2-(2,4-dimethylphenoxy)ethylidene]hydrazide (Ii). Yield 79 %; m.p. 136–137 °C; Anal. Calcd. for C₁₉H₁₉N₃O₂S₂: C, 59.20; H, 4.97; N, 10.90 %. Found: C, 59.28; H, 4.93; N, 10.91 %; IR (KBr, cm⁻¹): 3195, 1682, 1562, 1528, 1494; ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 2.14 (3H, *s*), 2.19 (3H, *s*), 4.26–4.58 (2H, *s*), 4.67–4.68 (2H, *d*, *J* = 5.0 Hz), 6.84–6.90 (1H, *m*), 6.91–6.98 (2H, *m*), 7.34–7.40 (1H, *m*), 7.44–7.50 (1H, *m*), 7.56–7.74 (1H, *t*, *J* = 5.0 Hz), 7.84 (1H, *d*, *J* = 7.8 Hz), 8.02 (1H, *t*, *J* = 7.4 Hz), 11.67–11.91 (1H, *2bs*). ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 15.91 (CH₃), 20.04 (CH₃), 35.20 (CH₂), 66.97 (CH₂), 111.75 (CH), 121.07 (CH), 121.77 (CH), 124.44 (CH), 125.63, 126.32 (CH), 127.05 (CH), 129.34, 131.26 (CH), 134.67, 143.17 (CH), 152.46, 153.85, 163.15, 165.83 (C); MS-FAB⁺ (*m/z*): 386 [M+1].

2-Thioxo-3(2H)-benzothiazoleacetic acid, [2-(2,6-dimethylphenoxy)ethylidene]hydrazide (Ij). Yield 75 %; m.p. 86–88 °C; Anal. Calcd. for C₁₉H₁₉N₃O₂S₂: C, 59.20; H, 4.97; N, 10.90 %. Found: C, 59.08; H, 4.84; N, 10.98 %; IR (KBr, cm⁻¹): 3210, 1684, 1599, 1520, 1493; ¹H-NMR (400 MHz, DMSO-*d*₆, δ / ppm): 2.24 (6H, *s*), 4.27–4.56 (2H, *s*), 4.46 (2H, *d*, *J* = 5.3 Hz), 6.91–6.96 (1H, *m*), 7.01–7.05 (2H, *m*), 7.37 (1H, *m*), 7.47 (1H, *m*), 7.65–7.72 (1H, *t*, *J* = 5.3 Hz), 7.80–7.86 (1H, *m*), 8.01 (1H, *t*, *J* = 8.3 Hz), 11.69–11.87 (1H, *2bs*); ¹³C-NMR (100 MHz, DMSO-*d*₆, δ / ppm): 16.13 (2CH₃), 35.14 (CH₂), 71.11 (CH₂), 121.06 (CH), 121.75 (CH), 124.03 (CH), 124.42 (CH), 126.31 (CH), 128.77 (2CH), 130.30 (2C), 134.68, 143.33, 152.46, 155.20, 163.23, 165.81; MS-FAB⁺ (*m/z*): 386 [M+1].