

SUPPLEMENTARY MATERIAL TO

Synthesis and spectral characterization of novel 1,5-benzodiazepine oxime derivatives

LINA REKOVIC^{1*}, LIDIJA KOSYCHOVA^{1,2}, IRINA BRATKOVSKAJA¹ and REGINA
VIDZIUNAITE¹

¹ *Institute of Biochemistry, Life Sciences Center, Vilnius University, Saulėtekio al. 7, Vilnius
10223, Lithuania*

² *Klaipeda University, H. Manto 84, LT-91001 Klaipeda, Lithuania*

ANALYTICAL AND SPECTRAL DATA FOR SYNTHESISED COMPOUNDS

1,3,4,5-tetrahydro-2H-1,5-benzodiazepin-2-one oxime (1). Cream-colored crystals, yield 2,41g, 68.1%, mp 147-148 °C (Ethanol), IR ($\nu_{\max}$, cm⁻¹): 3388, 3262, 3060, 1665, 1491, 1447, 1435, 1407, 1299, 965, 765. ¹H NMR (DMSO-d₆) δ : 2.34 (m, 2H, CH₂), 3.37 (m, 2H, CH₂), 5.34 (br.s, 1H, NH), 6.57-6.92 (m, 4H, ArH), 7.70 (s, 1H, OH or NH), 9.39 (s, 1H, OH or NH) ppm; ¹³C NMR (DMSO-d₆) δ : 29.3 (C-3), 46.2 (C-4), 118.8, 119.2, 121.6, 122.9 (C-9a), 128.5, 138.6 (C-5a), 150.7 (C-2) ppm. Anal. Calcd for C₉H₁₁N₃O (177.20): C, 61.00; H, 6.26; N, 23.71. Found: C, 59.92; H, 6.14; N, 24.31.

4-methyl-1,3,4,5-tetrahydro-2H-1,5-benzodiazepin-2-one oxime (2). Cream-colored crystals, yield 2,94g, 78,4 %, mp 171-172 °C (Ethanol), IR ($\nu_{\max}$, cm⁻¹): 3386, 3295, 3066, 1670, 1508, 1486, 1442, 1405, 1289, 976, 765. ¹H NMR (DMSO-d₆) δ : 1.13 (d, 3H, J=6.3 Hz, CH₃), 2.01 (dd, 1H, J=6,9 Hz, J=14.0 Hz, CH₂), 2.33 (dd, 1H, J=4,4 Hz, J=14.1 Hz, CH₂), 3.62 (m, 1H, CH), 4.91 (s, 1H, NH), 6.65-6.93 (m, 4H, ArH), 7.80 (br.s, 1H, NH or OH), 9.32 (s, 1H, OH or NH) ppm. ¹³C NMR (DMSO-d₆) δ : 23.0 (CH₃), 34.8 (C-3), 52.8 (C-4), 119.9, 120.4, 121.0, 122.7, 130.7, 138.0, 148.9 (C-2) ppm. Anal. Calcd for C₁₀H₁₃N₃O (191.23): C, 62.81; H, 6.85; N, 21.97. Found: C, 62,64 ; H, 6.79; N, 22.08.

3-methyl-1,3,4,5-tetrahydro-2H-1,5-benzodiazepin-2-one oxime (3). Cream-colored crystals, yield 2,63g, 68.8%, mp 131-132° (Ethanol), IR ($\nu_{\max}$, cm⁻¹): 3350, 3176, 3048, 1660, 1497, 1460, 1424, 1411, 1299, 955, 767. ¹H NMR (DMSO-d₆) δ : 1.01 (d, 3H, J=7.0 Hz, CH₃), 2.61 (m, 1H, CH), 3.11 (dd, 1H, J=9.0 Hz, J= 11.5 Hz, CH₂), 3.37 (dd, 1H, J=3.3 Hz, J= 11.5 Hz, CH₂), 5.43 (br.s, 1H, NH), 6.54-6.91 (m, 4H, ArH), 7.61 (s, 1H, NH or OH), 9.51 (s, 1H, NH or OH) ppm. ¹³C NMR (DMSO-d₆) δ : 14.6 (CH₃), 33.9 (C-3), 52.2 (C-4), 118.4, 118.5, 121.2, 122.5, 128.0, 138.5, 152.9 (C-2) ppm. Anal. Calcd for C₁₀H₁₃N₃O (191.23): C, 62.81; H, 6.85; N, 21.97. Found: C, 62, 59; H, 6.74; N, 22.12.

*Corresponding author. E-mail: lina.rekovic@gmail.com