SUPPLEMENTARY MATERIAL TO

Novel methylene bridged ethylenediamine-type ligands: synthesis and spectral characterization

LJILJANA E. MIHAJLOVIĆ-LALIĆ, ALEKSANDAR SAVIĆ, GABRIJELA BRADAN, TIBOR J. SABO and SANJA GRGURIĆ-ŠIPKA*

Faculty of Chemistry, University of Belgrade, Studentski trg 12–16, 11000 Belgrade, Serbia


Structures of \( C_1 \) and \( C_2 \) with atomic numbering.

ANALYTIC AND SPECTRAL DATA FOR \( C_1 \) AND \( C_2 \)

\textbf{Compound} \( C_1 \). Yield: 83 \%; Anal. Caled. for \( C_{29}H_{52}N_2O_4 \): C, 70.69, H, 10.64, N, 5.69 \%. Found: C, 70.38; H, 10.36; N, 5.76 \%; IR (ATR, \( \text{cm}^{-1} \)): 2928, 2852, 1734, 1468, 1451, 1375, 1252, 1165, 994; \( ^1 \text{H}-\text{NMR} \) (500 MHz, CDCl\(_3\), \( \delta \) / ppm): 0.82–0.90 (4H, \( m \), H5\( a' \), H5\( b' \)), 0.92 (12H, \( d \), \( J = 7.0 \text{ Hz} \), (CH\(_3\)_2CH), 1.07–1.31 (8H, \( m \), H4, H7, H6b), 1.47–1.52 (2H, \( m \), CH\(_2\)Cy), 1.60–1.66 (10H, \( m \), H6a, H5a, H7, CH\(_2\)Cy), 1.76 (2H, \( d \), \( J = 12.5 \text{ Hz} \), H5b), 1.92 (2H, \( m \), (CH\(_3\)_2CHCH\(_2\)O), 2.82–2.88 (2H, \( m \), NCH\(_2\)CH\(_2\)N), 2.95–3.01 (2H, \( m \), NCH\(_2\)CH\(_2\)N), 3.36–3.39 (2H, \( m \), OCH\(_2\)N), 3.63 (2H, s, NCH\(_2\)N), 3.86 (4H, \( d \), \( J = 6.5 \text{ Hz} \), CH\(_2\)OOC); \( ^{13} \text{C}-\text{NMR} \) (125 MHz, CDCl\(_3\), \( \delta \) / ppm): 19.29 ((CH\(_3\)_2CH), 26.25 (C6a), 26.28 (C6b), 26.59 (C7), 27.80 ((CH\(_3\)_2CH), 33.20 (C5b), 33.74 (C5a), 34.50 (C4), 38.89 (CH\(_2\)Cy), 48.23 (NCH\(_2\)CH\(_2\)N), 62.05 (OCCH\(_2\)N), 69.55 (NCH\(_2\)N), 70.59 (CH\(_2\)OOC), 173.31 (C1). ESI-MS (\( m/z \), (relative abundance, \%)): 481.58 (M–CH\(_2\)+3H\(^+\), 100), 493.40 (M\(^+\), 37.16).

*Corresponding author. E-mail: sanjag@chem.bg.ac.rs

S137
Compound **C2.** Yield: 71 %; Anal. Calcd. for C₃₁H₅₆N₂O₄: C, 71.49, H, 10.84, N, 5.38 %. Found: C, 71.10; H, 10.44; N, 5.49 %; IR (ATR, cm⁻¹): 2924, 2851, 1732, 1683, 1449, 1367, 1306, 1252, 1164, 971; H-NMR (500 MHz, CDCl₃, δ / ppm) 0.82–0.88 (4H, m, H₅a', H₅b'), 0.90 (12H, d, J = 6.5 Hz, (CH₃)₂CH), 1.06–1.31 (8H, m, H₄, H₇', H₆b), 1.46–1.53 (2H, m, CH₂Cy), 1.59–1.70 (10H, m, H₆a, H₅a, H₇, CH₂Cy), 1.75 (2H, d, J = 13.0 Hz, H₅b), 2.80–2.86 (2H, m, NCH₂CH₂N), 2.94–3.00 (2H, m, NCH₂CH₂N), 3.33–3.35 (2H, m, OCCHN), 3.61 (2H, s, NCH₂N), 4.11 (4H, m, CH₂CH₂OOC); C-NMR (50 MHz, CDCl₃, δ / ppm): 11.32 and 16.61 ((CH₃)₂CH), 22.56 ((CH₃)₂CH), 25.16 (C₆a), 26.27 (C₆b), 26.60 (C₇), 33.19 (C₅b), 33.77 (C₅a), 34.52 (C₄), 37.50 ((CH₃)₂CH₂C), 38.87 (CH₂Cy), 48.30 (NCH₂CH₂N), 62.16 (OCCHN), 63.04 (CH₂OOC), 69.60 (NCH₂N), 173.47 (C₁); ESI-MS (m/z, (relative abundance, %)): 509.43 (M–CH₂⁺H⁺, 49), 521.43 (M⁺, 100).

![Chemical structures of compounds C2](image)

**Fig. S-1.** Recently synthesized compounds with confirmed antitumor activity.
Fig. S-2. $^1$H-NMR spectrum of C1 recorded in CDCl$_3$.

Fig. S-3. $^{13}$C-NMR spectrum of C1 recorded in CDCl$_3$. 
Fig. S-4. HSQC NMR spectrum of C1 recorded in CDCl₃.

Fig. S-5. ¹H-NMR spectrum of C2 recorded in CDCl₃.
Supplementary Material

Fig. S-6. $^{13}$C-NMR spectrum of C2 recorded in CDCl$_3$.

Fig. S-7. COSY NMR spectrum of C2 recorded in CDCl$_3$.