

Supplementary data for article:

Pantelić, N. Đ.; Zmejkovski, B. B.; Trifunović-Macedoljan, J.; Savić, A.; Stanković, D.; Damjanović, A.; Juranic, Z.; Kaluđerović, G. N.; Sabo, T. Gold(III) Complexes with Esters of Cyclohexyl-Functionalized Ethylenediamine-N,N'-Diacetate. *Journal of Inorganic Biochemistry* **2013**, *128*, 146–153. <https://doi.org/10.1016/j.jinorgbio.2013.08.002>

SUPPLEMENTARY MATERIAL

For:

Gold(III) complexes with esters of cyclohexyl-functionalized ethylenediamine-*N,N'*-diacetate

Nebojša Pantelić^a, Bojana B. Zmejkovski^b, Jelena Trifunović-Macedoljan^c, Aleksandar Savić^d, Dalibor Stanković^d, Ana Damjanović^c, Zorica Juranić^c, Goran N. Kaluđerović^{e*},
and Tibor J. Sabo^{d*}

^aFaculty of Agriculture, University of Belgrade, Nemanjina 6, Belgrade-Zemun, Serbia; ^bDepartment of Chemistry, Institute of Chemistry, Technology and Metallurgy, University of Belgrade, Studentski trg 14, 11000 Belgrade, Serbia; ^cInstitute of Oncology and Radiology, 11000 Belgrade, Serbia; ^dFaculty of Chemistry, University of Belgrade, P.O. Box 158, 11001 Belgrade, Serbia; ^eInstitut für Chemie, Martin-Luther-Universität Halle-Wittenberg, Kurt-Mothes-Straße 2, D-06120 Halle, Germany.

E-mail addresses: goran.kaluderovic@chemie.uni-halle.de (G. N. Kaluđerović);
tsabo@chem.bg.ac.rs (T. J. Sabo)

The supplementary material contains numerical experimental data for complexes **1–6** to support the text.

Experimental data:

1. Yield: 56 mg, 55%. Anal. Calcd for C₂₂H₄₀N₂O₄AuCl₂PF₆: C, 32.65; H, 4.98; N, 3.46%. Found: C, 32.70; H, 4.94; N, 3.38%. ¹H NMR (500 MHz, CDCl₃): δ 0.98 (m, C⁷H₂, 4H), 1.21 (m, C^{5,6}H₂, 8H), 1.80 (m, C³H₂, C⁴H and C^{5,6}H₂, 14H), 3.57 (m, C⁸H₂, 4H), 3.89 (s, CH₃–OOC–, 6H), 4.06 (m, C²H, 2H), 4.69 (s, NH, 2H). ¹³C NMR (125 MHz, CDCl₃): δ 25.9 (C⁶), 32.3 (C⁴), 33.0 (C⁷), 33.8 (C⁵), 37.8 (C³), 44.3 (C⁸), 54.0 (CH₃–OOC–) 59.2 (C²), 170.6 (C¹). IR (ATR): ν_{max} = 2927, 2852, 1732, 1447, 1261, 1227, 848 cm⁻¹. UV/Vis (CHCl₃): λ_{max} (ε, 8120 M⁻¹ cm⁻¹, 7780 M⁻¹ cm⁻¹) 315.00 nm, 323.50 nm; ESI–MS (CH₃CN), positive: m/z: 663.20 [M]⁺, 664.20 [M + H]⁺. E_p = – 304 mV.

2. Yield: 66 mg, 62%. Anal. Calcd for C₂₄H₄₄N₂O₄AuCl₂PF₆: C, 34.42; H, 5.29; N,

3.34%. Found: C, 34.76; H, 5.58; N, 3.46%. ^1H NMR (200 MHz, CDCl_3): δ 1.00 (m, C^7H_2 , 4H), 1.23 (m, $\text{C}^{5,6}\text{H}_2$, 8H), 1.35 (t, $\text{CH}_3\text{CH}_2\text{-OOC-}$, 6H), 1.80 (m, C^3H_2 , C^4H and $\text{C}^{5,6}\text{H}_2$, 14H), 3.55 (m, C^8H_2 , 4H), 4.02 (t, C^2H , 2H), 4.34 (m, $\text{CH}_3\text{CH}_2\text{-OOC-}$, 4H), 4.71 (s, NH , 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 13.9 ($\text{CH}_3\text{CH}_2\text{-OOC-}$), 25.9 (C^6), 32.2 (C^4), 32.8 (C^7), 33.1, 33.9 (C^5), 37.8 (C^3), 44.1 (C^8), 59.2 (C^2) 63.5 ($\text{CH}_3\text{CH}_2\text{-OOC-}$), 170.1 (C^1); IR (ATR): $\nu_{\text{max}} = 2929, 2854, 1734, 1449, 1283, 1229, 850 \text{ cm}^{-1}$; UV/Vis (CHCl_3): $\lambda_{\text{max}} (\epsilon, 11520 \text{ M}^{-1} \text{ cm}^{-1}) 317.55 \text{ nm}$; ESI-MS (CH_3CN), positive: $m/z: 691.23 [M]^+$, 692.24 [$M + \text{H}$] $^+$. $E_p = -304 \text{ mV}$.

3. Yield: 62 mg, 57%. Anal. Calcd for $\text{C}_{26}\text{H}_{48}\text{N}_2\text{O}_4\text{AuCl}_2\text{PF}_6 \cdot 2\text{H}_2\text{O}$: C, 34.64; H, 5.81; N, 3.11%. Found: C, 34.44; H, 5.50; N, 3.12%. ^1H NMR (200 MHz, CDCl_3): δ 0.98 (t, $\text{CH}_3\text{CH}_2\text{CH}_2\text{-OOC-}$, 6H; m, C^7H_2 , 4H), 1.24 (m, $\text{C}^{5,6}\text{H}_2$, 8H), 1.80 (m, C^3H_2 , C^4H , $\text{C}^{5,6}\text{H}_2$ and $\text{CH}_3\text{CH}_2\text{CH}_2\text{-OOC-}$, 18H), 3.48 (m, C^8H_2 , 4H), 3.98 (t, C^2H , 2H), 4.23 (t, $\text{CH}_3\text{CH}_2\text{CH}_2\text{-OOC-}$, 4H), 4.70 (s, NH , 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 10.3 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{-OOC-}$), 21.7 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{-OOC-}$), 25.9 (C^6), 32.3 (C^4), 33.1 (C^7), 33.9 (C^5), 38.1 (C^3), 44.4 (C^8), 59.2 (C^2) 68.7 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{-OOC-}$), 170.7 (C^1). IR (ATR): $\nu_{\text{max}} = 2929, 2853, 1734, 1451, 1277, 1224, 849 \text{ cm}^{-1}$; UV/Vis (CHCl_3): $\lambda_{\text{max}} (\epsilon, 9010 \text{ M}^{-1} \text{ cm}^{-1}) 315.84 \text{ nm}$; ESI-MS (CH_3CN), positive: $m/z: 719.27 [M]^+$, 720.27 [$M + \text{H}$] $^+$. $E_p = -304 \text{ mV}$.

4. Yield: 62 mg, 55%. Anal. Calcd for $\text{C}_{28}\text{H}_{52}\text{N}_2\text{O}_4\text{AuCl}_2\text{PF}_6$: C, 37.64; H, 5.86; N, 3.13%. Found: C, 37.90; H, 6.00; N, 3.36%. ^1H NMR (200 MHz, CDCl_3): δ 0.96 (t, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$, 6H; m, C^7H_2 , 4H), 1.21 (m, $\text{C}^{5,6}\text{H}_2$, 8H), 1.41 (q, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$, 4H), 1.80 (m, C^3H_2 , C^4H , $\text{C}^{5,6}\text{H}_2$ and $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$, 18H), 3.57 (m, C^8H_2 , 4H), 4.04 (t, C^2H , 2H), 4.30 (m, $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$, 4H), 4.71 (s, NH , 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 13.5 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$), 18.9 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$), 25.9 (C^6), 30.2 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$), 32.2 (C^4), 33.2 (C^7), 33.8 (C^5), 37.8 (C^3), 44.2 (C^8), 59.3 (C^2), 67.3 ($\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{-OOC-}$), 170.3 (C^1). IR (ATR): $\nu_{\text{max}} = 2929, 2853, 1729, 1451, 1260, 1210, 845 \text{ cm}^{-1}$; UV/Vis (CHCl_3): $\lambda_{\text{max}} (\epsilon, 8310 \text{ M}^{-1} \text{ cm}^{-1}) 319.25 \text{ nm}$; ESI-MS (CH_3CN), positive: $m/z: 747.29 [M]^+$, 748.29 [$M + \text{H}$] $^+$. $E_p = -276 \text{ mV}$.

5. Yield: 58 mg, 52%. Anal. Calcd for $\text{C}_{28}\text{H}_{52}\text{N}_2\text{O}_4\text{AuCl}_2\text{PF}_6$: C, 37.64; H, 5.86; N,

3.13%. Found: C, 37.96; H, 6.15; N, 3.35%. ^1H NMR (200 MHz, CDCl_3): δ 0.97 (d, $(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$, 12H; m, C^7H_2 , 4H), 1.22 (m, $\text{C}^{5,6}\text{H}_2$, 8H), 1.80 (m, C^3H_2 , C^4H and $\text{C}^{5,6}\text{H}_2$, 14H), 2.01 (hept, $(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$, 2H) 3.53 (m, C^8H_2 , 4H), 4.06 (m, C^2H , 2H), 4.06 (d, $(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$, 4H), 4.69 (s, *NH*, 2H). ^{13}C NMR (50 MHz, CDCl_3): δ 18.9 ($(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$), 25.9 (C^6), 27.5 ($(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$), 32.2 (C^4), 33.2 (C^7), 33.9 (C^5), 38.0 (C^3), 44.3 (C^8), 59.3 (C^2), 73.3 ($(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$), 170.5 (C^1). IR (ATR): $\nu_{\text{max}} = 2929, 2853, 1730, 1450, 1266, 1216, 848 \text{ cm}^{-1}$; UV/Vis (CHCl_3): λ_{max} (ϵ , $8470 \text{ M}^{-1} \text{ cm}^{-1}$) 324.36 nm; ESI-MS (CH_3CN), positive: m/z : 747.29 [M] $^+$, 748.30 [$M + \text{H}$] $^+$. $E_p = -237 \text{ mV}$.

6. Yield: 66 mg, 57%. Anal. Calcd for $\text{C}_{30}\text{H}_{56}\text{N}_2\text{O}_4\text{AuCl}_2\text{PF}_6$: C, 39.09; H, 6.12; N, 3.04%. Found: C, 38.98; H, 6.13; N, 3.11%. ^1H NMR (200 MHz, CDCl_3): δ 0.95 (d, $(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$, 12H; m, C^7H_2 , 4H), 1.24 (m, $\text{C}^{5,6}\text{H}_2$, 8H), 1.50-1.90 (m, C^3H_2 , C^4H , $\text{C}^{5,6}\text{H}_2$, $(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$ and $(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$, 20H), 3.43 (m, C^8H_2 , 4H), 3.92 (m, C^2H , 2H), 4.30 (m, $(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$, 4H), 4.71 (s, *NH*, 2H). δ ^{13}C NMR (50 MHz, CDCl_3): 11.1 ($(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$), 16.3 ($(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$), 22.3 ($(\text{CH}_3)_2\text{CHCH}_2\text{CH}_2\text{-OOC-}$), 25.9 (C^6), 32.4 (C^4), 33.1 (C^7), 33.8 (C^5), 36.9 (C^3), 44.5 (C^8), 59.2 (C^2), 65.8 ($(\text{CH}_3)_2\text{CHCH}_2\text{-OOC-}$), 171.0 (C^1). IR (ATR): $\nu_{\text{max}} = 2929, 2854, 1731, 1453, 1260, 1212, 851 \text{ cm}^{-1}$; UV/Vis (CHCl_3): λ_{max} (ϵ , $6630 \text{ M}^{-1} \text{ cm}^{-1}$) 320.95 nm; ESI-MS (CH_3CN), positive: m/z : 775.33 [M] $^+$, 776.33 [$M + \text{H}$] $^+$. $E_p = -273 \text{ mV}$.