

This is an Open Access document downloaded from ORCA, Cardiff University's institutional repository:<https://orca.cardiff.ac.uk/id/eprint/125828/>

This is the author's version of a work that was submitted to / accepted for publication.

Citation for final published version:

Estrada-Ortiz, Natalia, Lopez-Gonzales, Elena, Woods, Ben, Stürup, Stefan, de Graaf, Inge A. M., Groothuis, Geny M. M. and Casini, Angela 2019. Ex vivo toxicological evaluation of experimental anticancer gold(I) complexes with lansoprazole-type ligands. *Toxicology Research* 8 (6) , pp. 885-895. 10.1039/C9TX00149B

Publishers page: <http://dx.doi.org/10.1039/C9TX00149B>

Please note:

Changes made as a result of publishing processes such as copy-editing, formatting and page numbers may not be reflected in this version. For the definitive version of this publication, please refer to the published source. You are advised to consult the publisher's version if you wish to cite this paper.

This version is being made available in accordance with publisher policies. See <http://orca.cf.ac.uk/policies.html> for usage policies. Copyright and moral rights for publications made available in ORCA are retained by the copyright holders.



Supporting Information

***Ex vivo* toxicological evaluation of experimental anticancer gold(I) complexes with lansoprazole-type ligands**

N. Estrada-Ortiz,^a E. Lopez-Gonzales,^a Ben Woods,^b Stefan Stürup,^c I. A. M. de Graaf,^a G. M. M. Groothuis,^{a*} A. Casini^{a,b,d*}

Gold compounds analysis

Compound 1: Anal. Calcd for C₃₄H₂₉AuBF₇N₃O₂PS (915.42): C, 44.61; H, 3.19; N, 4.59. Found: C, 44.38; H, 3.19; N, 4.57. ¹H NMR (CDCl₃): δ 8.33 (br, 1H, H⁶), 7.74 (br, 2H, H^{3'}, H^{6'}), 7.53 (br, 15H, PPh₃), 7.36 (m, *J*_{H-H} = 9.1, 6.0, 3.0 Hz, 2H, H^{4'}, H^{5'}), 6.67 (d, *J*_{H-H} = 5.6 Hz, 1H, H⁵), 4.76 (AB, *J*_{AB} = 13.6 Hz, 2H, CH₂SO), 4.36 (br, 2H, OCH₂CF₃), 2.18 (br, 3H, CH₃). ³¹P NMR (CDCl₃): δ 31.2 ppm (s, PPh₃). ESI-MS (CH₃CN, pos. mode) for C₃₄H₂₉AuF₃N₃O₂PS: exp. 305.1465 (calc. 305.1978).

Compound 2: Anal. Calcd for C₂₂H₂₅AuF₃N₆O₂PS (722.47): C, 36.57; H, 3.49; N, 11.63. Found C, 36.57; H, 3.43; N, 11.48. ¹H NMR (CDCl₃): δ 8.36 (d, *J*_{H-H} = 5.6 Hz, 1H, H⁶), 7.74 (br, 2H, H^{3'}, H^{6'}), 7.23 (m, *J*_{H-H} = 9.2, 6.0, 3.2 Hz, 2H, H^{4'}, H^{5'}), 6.68 (d, *J*_{H-H} = 5.6 Hz, 1H, H⁵), 4.70 (q, AB, *J*_{AB} = 13.5 Hz, 2H, CH₂SO), 4.57 (q, AB, *J*_{AB} = 13.5 Hz, 6H, NCH₂N), 4.41 (q, *J*_{H-F} = 8.0 Hz, 2H, OCH₂CF₃), 4.36 (s, 6H, N-CH₂-P), 2.28 (s, 3H, CH₃). ¹H NMR (acetone-*d*₆): δ 8.34 (d, *J*_{H-H} = 5.4 Hz, 1H, H⁶), 7.62 (m, AA' part of an AA'BB'), *J*_{H-H} = 9.0, 6.0, 3.3 Hz, 2H, H^{3'}, H^{6'}), 7.12 (m, BB' part, *J*_{H-H} = 9.0, 5.7, 2.7 Hz, 2H, H^{4'}, H^{5'}), 7.07 (d, *J*_{H-H} = 5.4 Hz, 1H, H⁵), 4.83 (q, *J*_{H-F} = 8.4 Hz, 2H, OCH₂CF₃), 4.71 (AB, *J*_{AB} = 12.9 Hz, 6H, N-CH₂-N), 4.57 (s, 2H, CH₂SO), 4.52 (s, 6H, N-CH₂-P), 2.27 (s, 3H, CH₃). ³¹P NMR (CDCl₃): δ -58.6 ppm (s, PTA).

Compound 3: Anal. Calcd for C₅₂H₄₃Au₂BF₇N₃O₂P₂S (1373.66): C, 45.47; H, 3.16; N, 3.06. Found: C, 45.43; H, 3.12; N, 3.05. ¹H NMR (CDCl₃): δ 7.95 (d, br, *J*_{H-H} = 4.8 Hz, 1H, H⁶), 7.81 (m, AA' part of an AA'BB', *J*_{H-H} = 8.8, 5.6, 2.8 Hz, 2H, H^{3'}, H^{6'}), 7.59 (m, br, 30H, PPh₃), 7.39 (m, br, BB' part, *J*_{H-H} = 9.2, 5.2, 3.2 Hz, 2H, H^{4'}, H^{5'}), 6.78 (d, *J*_{H-H} = 5.6 Hz, 1H, H⁵), 4.78 (q, AB, *J*_{H-H} = 13.2 Hz, 2H, CH₂SO), 4.27 (qd, *J*_{H-F} = 8.0, 3.2 Hz, 2H, CH₂CF₃), 1.93 (s, 3H, CH₃). ³¹P NMR (CDCl₃): δ 31.0 and 33.2 ppm.

Figures

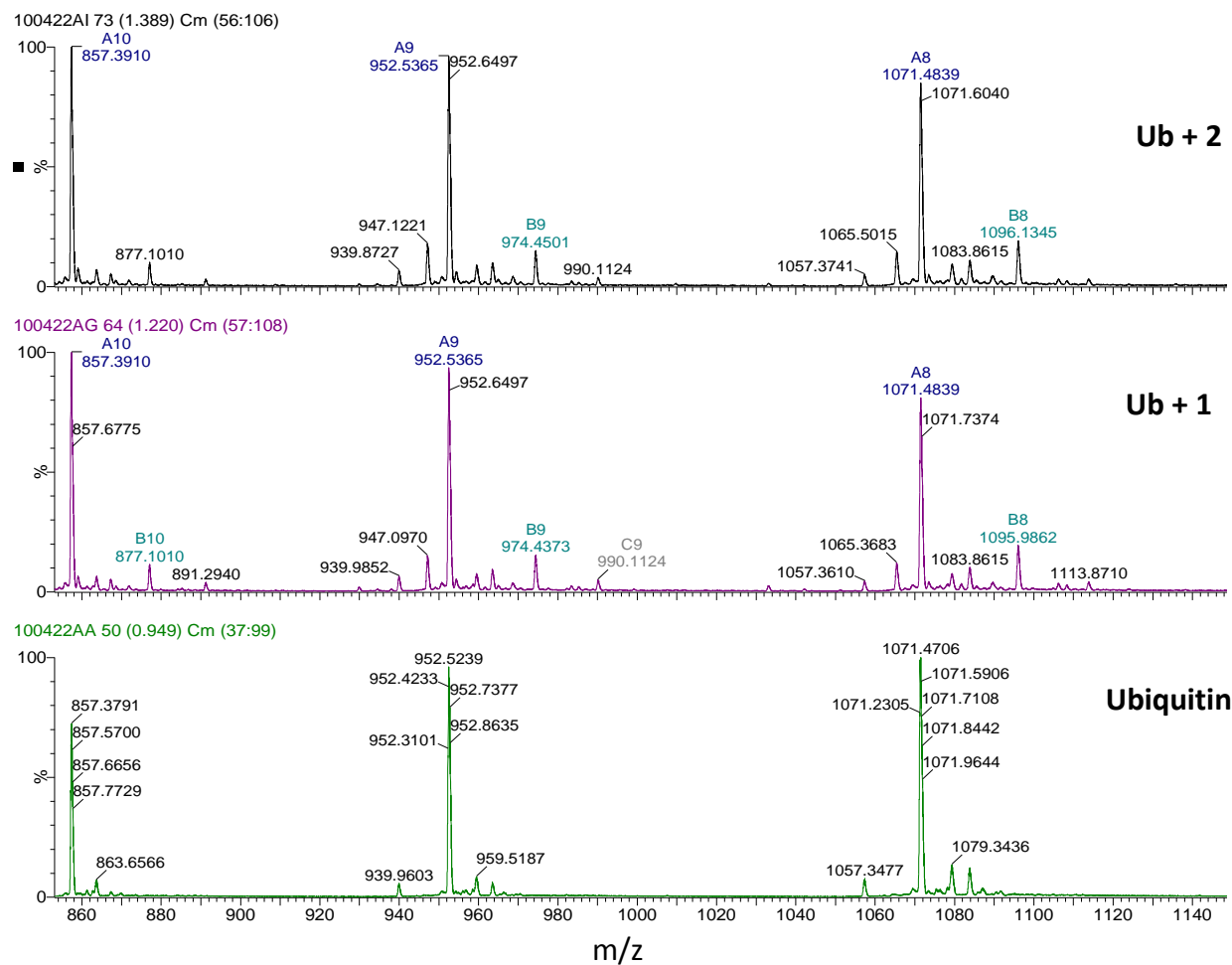


Figure S1 – Multicharged ESI mass spectra of Ub alone (bottom) or incubated with **1** and **2** (gold complex/Ub ratio = 3:1) for 24 h at 37 °C.