Supporting information

Vanadyl complexes with dansyl-labelled di-picolinic acid ligands:
synthesis, phosphatase inhibition activity and cellular uptake studies

Juliet Collins, Agostino Cilibrizzi, Marina Fedorova, Gillian Whyte, Lok Mak, Inna Guterman, Robin Leatherbarrow, Rudiger Woscholski, Ramon Vilar

Table of Contents

Figures S1 and S2. $^1$H NMR spectra of ligands 8 and 13

Figure S3. Fluorescence spectra of 8, 14 and 15 in buffer solution

Figure S4. Fluorescence changes of ligand 8 and complex 14 in the presence or absence of LMW-PTP and EDTA

Figure S5. Fluorescence intensity of ligand 13 and complex 15 in the presence or absence of LMW-PTP and EDTA

Figure S6. Activity of LMW-PTP in the presence of ligands 8 and 13

Figure S7. Activity of VHR in the presence of ligands 8 and 13
Figure S1: $^1$H NMR spectrum of 8 (400MHz, CD$_3$OD)

Figure S2: $^1$H NMR spectrum of 13 (400MHz, CD$_3$OD)
Figure S3. Fluorescence spectra of 8, 14, and 15 at 30µM in 100 mM Tris buffer containing 1 mM DTT. Readings were taken using a Varian fluorescence spectrometer in a 96-well plate with excitation of 340 nm and emission recorded from 400-650 nm.

Figure S4. Fluorescence intensity at 560 nm of a 30 µM solution of ligand 8 (yellow bars) and complex 14 (green bars) in the presence or absence of LMW-PTP (Enz) and 1mM EDTA in Tris buffer containing 1mM DTT. Reading was taken after 5 minutes incubation at room temperature. Intensity is recorded ±standard deviation of triplicate repeats.
Figure S5. Fluorescence intensity at 560 nm of a 30 μM solution of ligand 13 (yellow bars) and complex 15 (green bars) in the presence or absence of LMW-PTP (Enz) and 1mM EDTA in Tris buffer containing 1mM DTT. Reading was taken after 2 hours incubation at room temperature. Intensity is recorded ± standard deviation of triplicate repeats.
Figure S6. Activity of LMW-PTP in the presence of increasing amounts of ligands 8 and 13. LMW-PTP activity was measured using the OMFP method (see Experimental Details – Phosphatase inhibition assays). The ligand solutions (prepared from 10 mM stock solution in DMSO and further diluted in water containing 1% DMSO to the required concentrations) were incubated with the enzyme in the buffer (100 mM Tris, pH = 7.4, containing 1 mM DTT) for 10 minutes at room temperature before reaction was initiated by addition of OMFP. Reading was taken over 30 minutes at 60 s intervals.
**Figure S7.** Activity of VHR in the presence of increasing amounts of ligands 8 and 13. VHR activity was measured using the OMFP method (see Experimental Details – Phosphatase inhibition assays). The ligand solutions (prepared from 10 mM stock solution in DMSO and further diluted in water containing 1% DMSO to the required concentrations) were incubated with the enzyme in the buffer (100 mM Tris, pH = 7.4, containing 1 mM DTT) for 10 minutes at room temperature before reaction was initiated by addition of OMFP. Reading was taken over 30 minutes at 60 s intervals.