Supporting Information

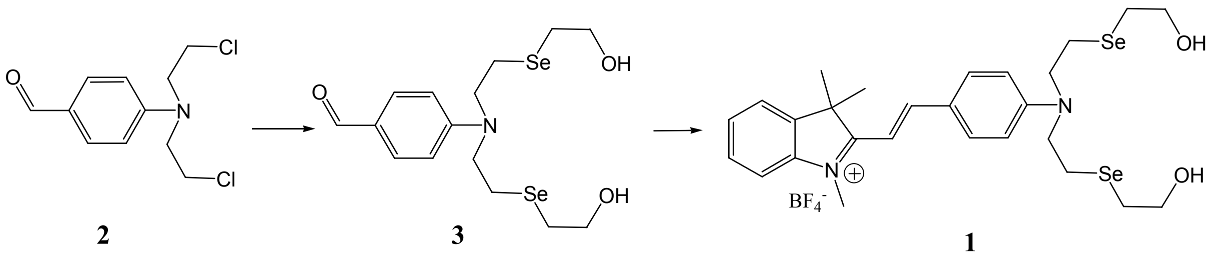
Materials and methods

1. Materials

All synthetic reagents were used as supplied. Solvents were dried and distilled using standard procedures. 1,2-bis(2-hydroxyethyl)diselenide[S1] and 2[S2] were prepared according to the literature procedures.

2. Instruments

Silica gel (230-400 mesh) was used for flash chromatography separations. ^{1}H and ^{13}C NMR spectra were recorded respectively at 400 MHz and 100 MHz on a Bruker spectrometer at 400 (^{1}H NMR) MHz and 100 (^{13}C NMR) MHz. Chemical shifts are expressed in ppm. Positive ion FAB mass spectra were recorded on a ZAB-HS mass spectrometer (VG, U. K.). Elemental analyses were performed on a Vanio-EL elemental analyzer (Analysensysteme GmbH, Germany). UV absorption spectra were recorded on a UV-3600 UV-VIS spectrophotometer (Shimadzu, Japan). Fluorescence measurements were performed using an F-4600 fluorescence spectrophotometer (Hitachi, Japan) equipped with a plotter unit and a quartz cell (1 cm × 1 cm).



Preparation of 4-*N,N*-bis(2'-hydroxyethylselenoethyl)aminobenzoaldehyde 3.

To a 250 mL reactor, was charged 1,2-(2-hydroxyethyl)diselenide (3.183 g, 12.73 mmol), NaOH (1.526 g, 38.19 mmol), $NaBH_4$ (1.451g, 38.19 mmol) and ethanol (130 mL). The reaction mixture was washed with nitrogen flow for 15 min, and then refluxed under nitrogen atmosphere for 1 h. The resulting reaction mixture was cooled to room temperature, and 3 *N* HCl (40 mL) was added dropwise to destroy the excess $NaBH_4$. After the addition of HCl solution, the mixture was stirred for 1 h at room temperature, and then 5 *N* NaOH (20 mL) was added dropwise to the reaction mixture. To this mixture, 4-*N,N*-bis(2'-chloroethyl)aminobenzoaldehyde **2** (2.00 g, 8.13 mmol) in THF (60 mL) was added dropwise. The result mixture was stirred at 60 °C for 24 h. The reactant was extracted with dichloromethane (30 mL × 3). The combined organic phase was dried over $MgSO_4$. The product was purified by column chromatography (SiO_2, hexane/ethyl acetate, v/v, 1:2). The product was obtained as yellow powder (1.10 g) in 32% yield; m.p. 162-163 °C ; MS (FAB): 426.0 $(M+H)^+$; cald: 426.0; ^1H NMR(400 MHz, $CDCl_3$), 9.75 (s, 1H), 7.75(d, J = 8.4 Hz, 2H), 6.71 (d, J = 8.4 Hz, 2H), 3.86 (s, 4H), 3.73 (s, 4H), 2.83 (t, J = 6.4 Hz, 8H), 2.49 (s, 2H). ^{13}C NMR(100 MHz, $CDCl_3$): 190.31, 151.51, 132.51, 126.06, 111.22, 62.13, 61.89, 52.12, 27.82, 20.69; elemental analysis calcd (%) for $C_{15}H_{23}NO_3Se_2{\cdot}H_2O$: C: 40.83, H: 5.71, N: 3.17; Found: C: 40.73, H: 5.92, N: 2.92.

Preparation of chemosensor 1.

To a 50 ml flask, was charged 1,2,3,3'-tetramethylindolium iodide (486 mg, 1.615 mmol), **3** (684 mg, 1.615 mmol) and anhydrous ethanol (10 mL). The reaction mixture was washed with nitrogen flow for 30 min to remove oxygen, and then stirred at 95 °C for 14 h in dark. After cooling to room temperature, $NaBF_4$ (1.60 g, 12.70 mmol) was added in one portion to the reaction mixture. The suspension was refluxed for another 3 h in dark. The solvent was removed under reduced pressure. The residue was dissolved in dichloromethane (100 mL) and filtered; the solid salt was washed with dichloromethane (2 × 15 mL). The filtrate was condensed to dryness. The residue was dissolved with hot ethanol (10 mL). After cooling to room temperature, **1** was crystallized from the solvent to get pure **1** in 83% yield; m.p. 185-186 °C; MS (FAB): 581.12 $(M-BF_4^-)^+$; cald: 581.12. ^1H NMR(400 MHz, DMSO-d_6): 8.30 (d, J = 15.6 Hz, 1H), 8.06 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 7.2 Hz, 1H), 7.72 (d, J = 7.6 Hz, 1H), 7.56 (t, J = 7.4 Hz, 1H), 7.51 (t, J = 6.8 Hz, 1H), 7.28 (d, J = 15.6 Hz, 1H), 6.89 (d, J = 8.4 Hz, 2H), 3.98 (s, 3H), 3.79 (t, 4H), 3.66 (t, 4H), 2.82 (t, 4H), 2.74 (t, 4H), 1.75(s, 6H); ^{13}C NMR(100 MHz, DMSO-d_6): 179.48, 153.39, 151.56, 142.18, 141.57, 133.67, 128.29, 127.31, 122.29, 122.16, 113.27, 111.81, 105.27, 61.23, 50.83, 50.55, 39.31, 39.10, 38.89, 32.75, 26.07, 25.73, 19.79; elemental analysis calcd (%) for $C_{27}H_{37}BF_4N_2O_2Se_2$: C: 48.67%, H: 5.60 %, N: 4.20%; Found: C: 48.58%, H: 5.68%, N: 4.06%.

References:

S1: X. Hu, Z. Tian, X. Lu and Y. Chen, *Synth. Commun.* 1997, **27**, 553.

S2: X. M. Meng, L. Liu, H. Y. Hu, M. Z. Zhu, M. X. Wang, J. Shi and Q. X. Guo, *Tetrahedron Lett.* 2006, **47**, 7961.

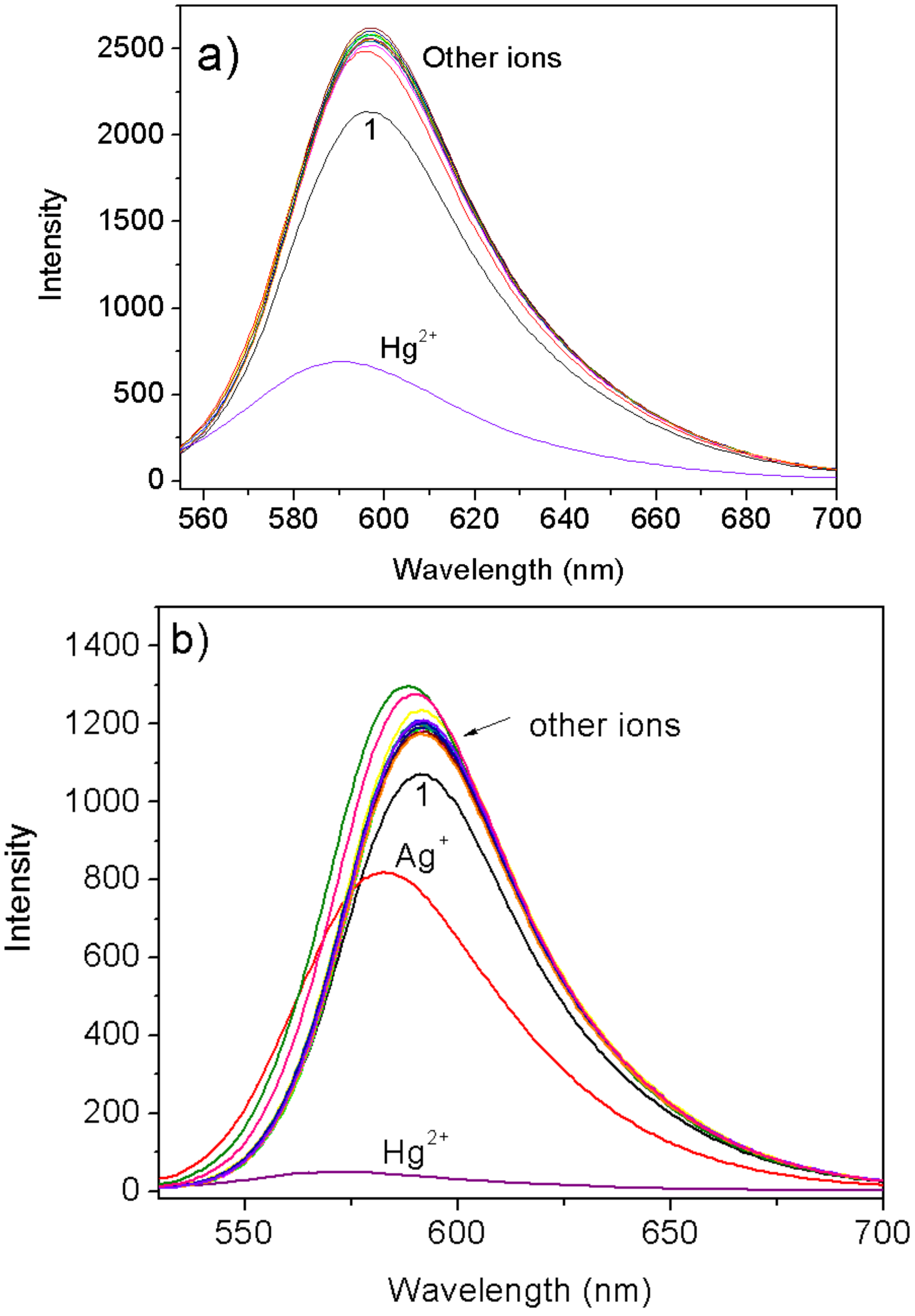


Figure S1. Fluorescence spectra of **1** (1 μM) upon the addition of the nitrate salts (10.0 equiv) of Na^+, K^+, NH_4^+, Mg^{2+}, Ca^{2+}, Pb^{2+}, Hg^{2+}, Co^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+}, Cu^{2+}, Cr^{3+}, Fe^{3+} and Ag^+ a) in ethanol/H_2O (1:1, v/v) and b) in H_2O.

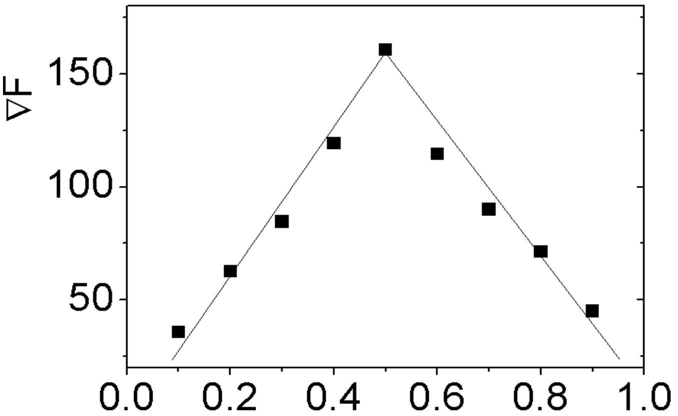


Figure S2. Job's plot of 1 in water showing the 1:1 stoichiometry of the complex between Hg^{2+} ion and 1. The total of chemosensor and Hg^{2+} is 1 μM. Fluorescence is recorded at 591 nm.

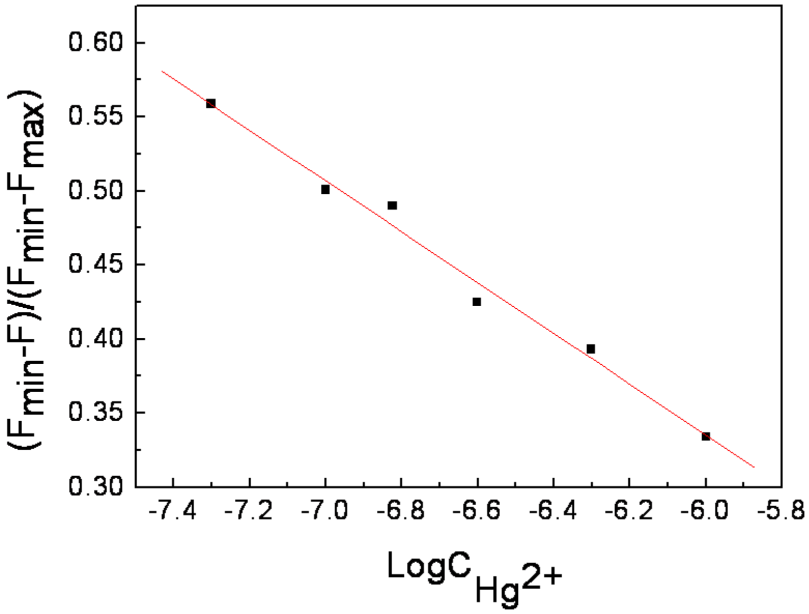


Figure S3. Emission (at 590 nm) of **1** at different concentrations of Hg^{2+} (0.05, 0.1, 0.15, 0.25, 1, 2 μM) added, normalized between the minimum emission (0.0 μM Hg^{2+}) and the emission at 2 μM Hg^{2+}. The detection limit was determined to be 5.0×10^{-8} M.

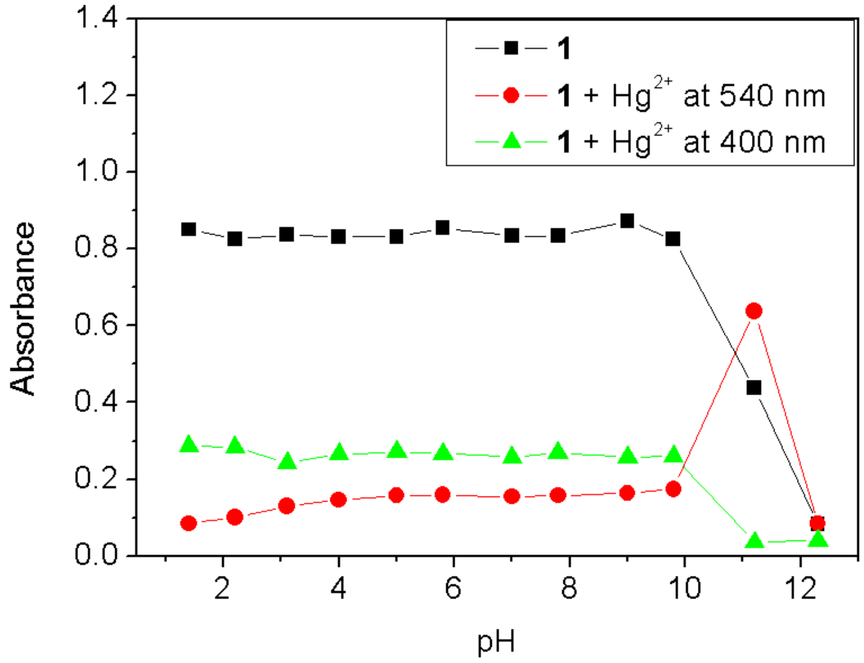


Figure S4. pH profiles of the absorption of **1** (10 µM) in the absence and presence of Hg^{2+} in EtOH/H_2O (1:1, v/v).

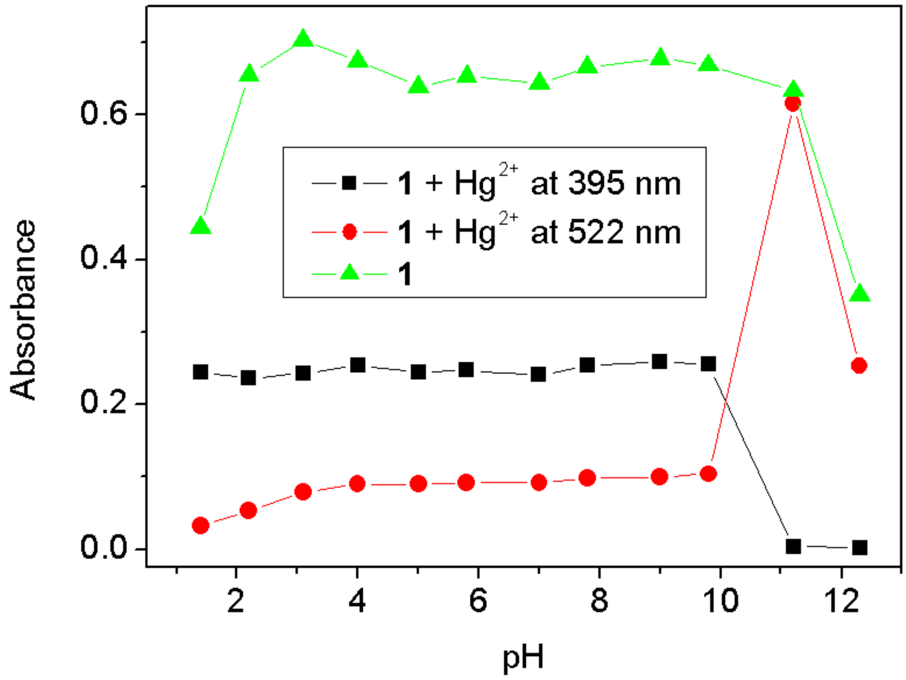


Figure S5. pH profiles of the absorption of **1** (10 µM) in the absence and presence of Hg^{2+} in H_2O.

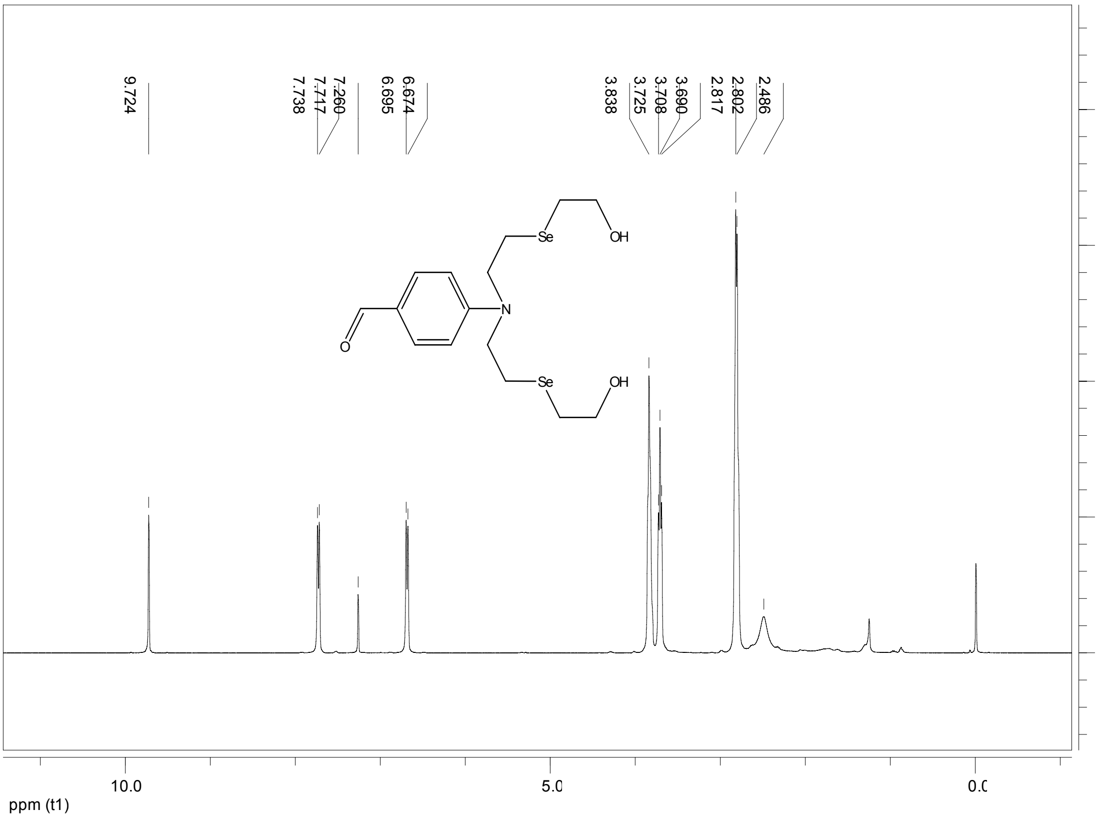


Figure S6. ^{1}H NMR of compound **3** (400 MHz, $CDCl_3$).

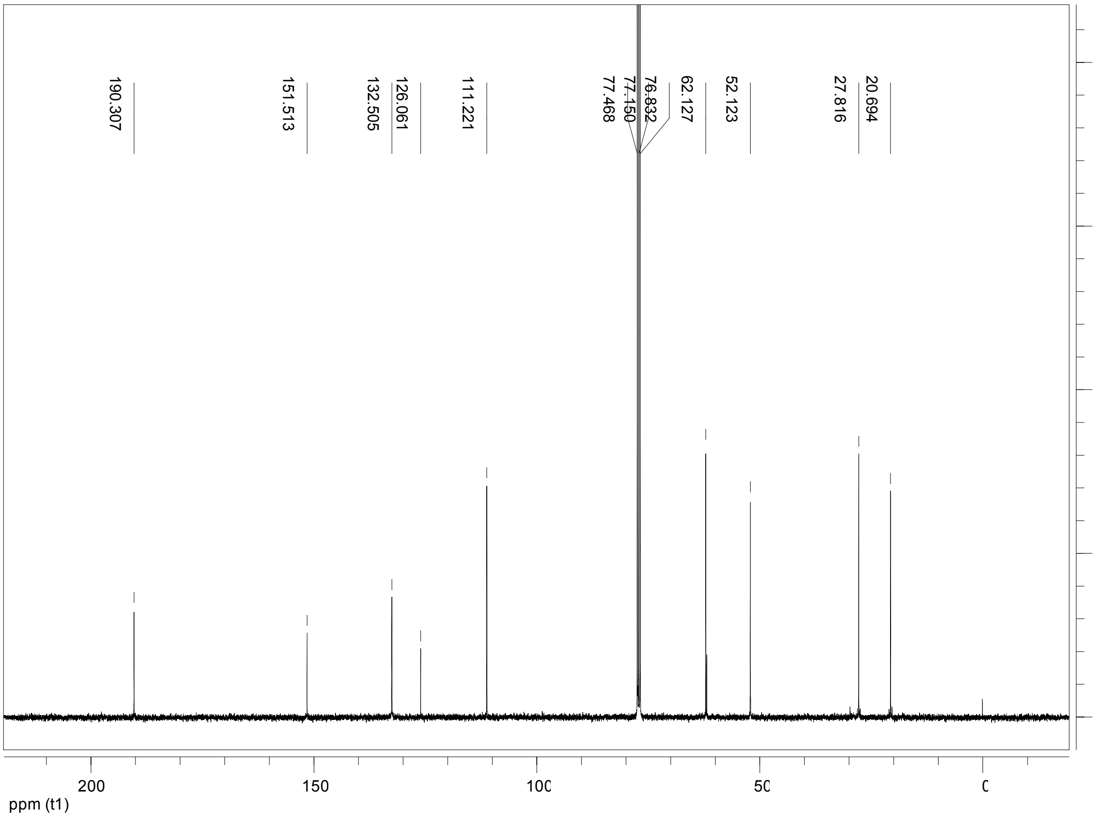


Figure S7. ^{13}C NMR of compound **3** (100 MHz, $CDCl_3$).

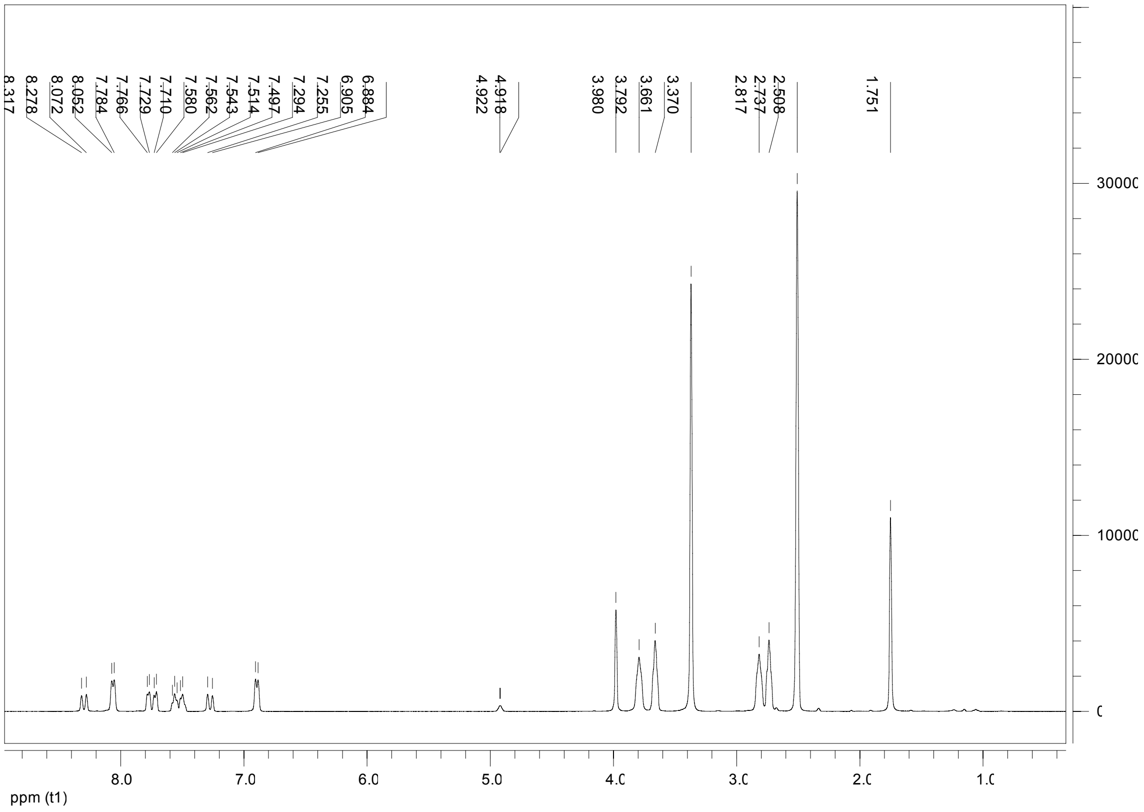


Figure S8. ^{1}H NMR of compound **1** (400 MHz, DMSO-d$_6$).

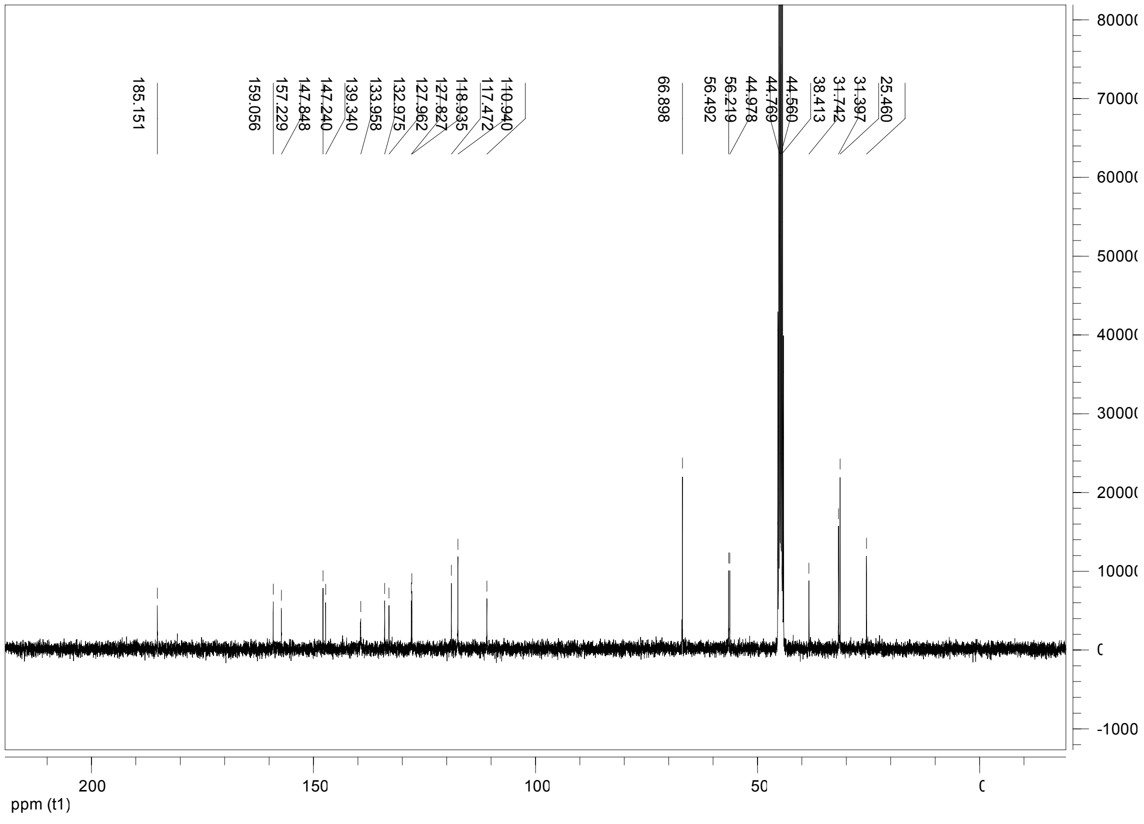


Figure S9. ^{13}C NMR of compound **1** (100 MHz, DMSO-d$_6$).