Electronic Supplementary Material (ESI) for Journal of Materials Chemistry B.

Supporting Information

Fluorescence imaging of lysosomal hydrogen selenide under oxygen-controlled conditions

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Contents

1. Synthesis of Se-1

Synthesis of compound 1

1,4-Diamino-2-nitrobenzene (554 mg, 3.62 mmol) and 4-bromo-1,8-naphthalic anhydride (1.0 g, 3.62 mmol) were dissolved in 2-methoxylethanol (30 mL) and stirred under reflux for 24 h. Cooling of the reaction solution yielded a precipitate, which was isolated by filtration. A pale yellow solid (1.37 g, 92%) were obtained as product.

Synthesis of compound 2

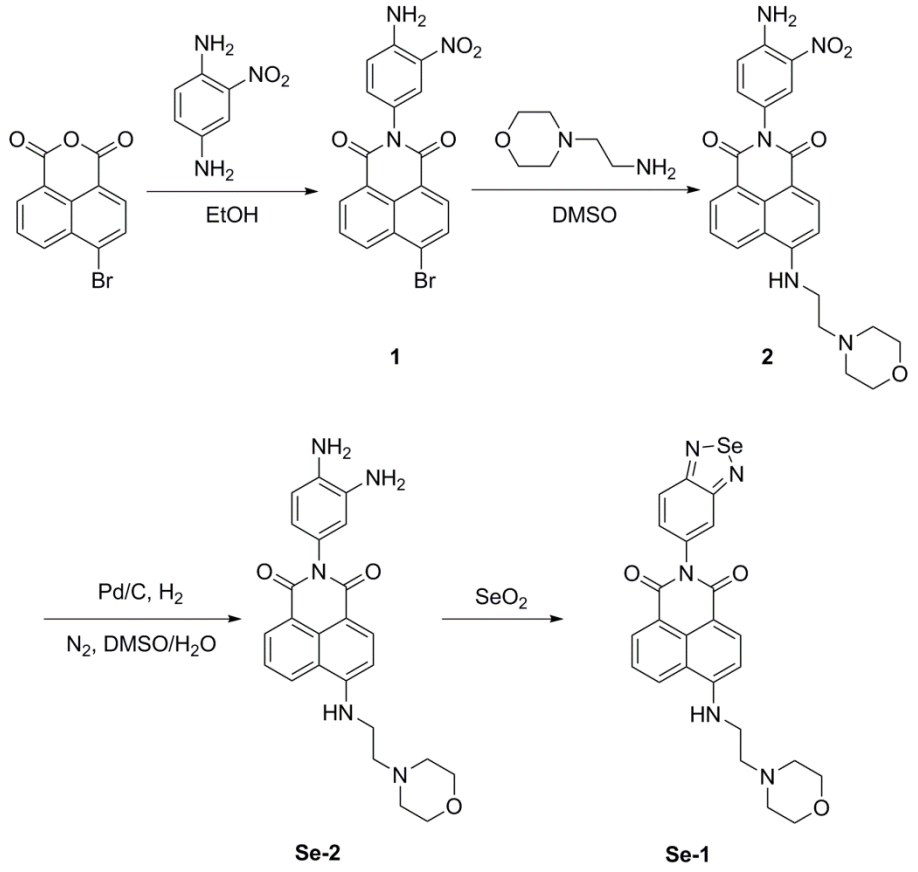
Compound 1 (1 g, 2.4 mmol) and 4-(2-aminoethyl)morpholine (1.3 mL, 9.6 mmol) were dissolved in dry DMSO and stirred at 90 °C overnight. After completion of the reaction, the resulting solution was poured into ice water and a yellow precipitate was isolated by filtration. The resulting solid was purified by silica gel column chromatography using eluent CH_2Cl_2/MeOH (v/v, 10/1). Compound 2 was obtained as a yellow solid (940 mg, 85%).

Synthesis of compound Se-2

Compound 2 (500 mg, 1.08 mmol) and Pd/C (10% Pd, 100 mg) were stirred in THF (100 mL) under hydrogen (2.5 atm) at room temperature for 48 h. The product was purified by silica gel column chromatography with eluent CH_2Cl_2/MeOH (v/v, 8/1) to give **Se-2** (414 mg, 89%). ^{1}H NMR (400 MHz, DMSO, ppm) δ: 2.52 (s, 2H), 3.71-3.83 (m, 6H), 6.34 (d, 1H), 6.47 (s, 1H), 6.67 (d, 1H), 6.88 (d, 1H), 7.50-7.61 (m, 1H), 7.70 (t, 1H), 8.27 (d, 1H), 8.42 (d, 1H), 8.76 (d, 1H). ^{13}C NMR (176 MHz, DMSO, ppm) δ: 164.61, 163.87, 150.43, 135.19, 134.41, 131.19, 130.06, 129.39, 126.74, 124.95, 122.92, 120.91, 118.36, 115.88, 115.11, 109.47, 104.59, 72.98, 64.55, 63.52, 55.08, 52.23, 40.89, 38.39. ESI-MS calcd. for C24H25N5O3 [M+H+]: 432.1957; found: 432.2044.

Synthesis of compound Se-1

To a porcelain mortar was added compound **Se-2** (90 mg, 0.21 mmol) and SeO_2 (28 mg, 0.25 mmol). The mixture was fully grinded for 30 min, and TLC showed full conversion of compound **Se-2** to the probe. The resulting mixture was extracted by MeOH (20 mL) and the solvent was removed under reduced pressure, then the crude product was purified by silica gel chromatography with eluent CH_2Cl_2/MeOH (v/v, 3/1) to give **Se-1** (76 mg, 82%). ^{1}H NMR (400 MHz, DMSO, ppm) δ: 3.03-3.24 (m, 2H), 3.38-3.62 (m, 3H), 3.74-4.01 (m, 7H), 6.97 (d, 1H), 7.53 (d, 1H), 7.78 (t, 1H), 7.89-7.96 (m, 2H), 8.08 (s, 1H), 8.34 (d, 1H), 8.51 (d, 1H), 8.90 (d, 1H). ESI-MS calcd. for C24H21N5O3Se [M+H+]: 508.0810; found: 508.0871.



Scheme S1 Synthetic route of Se-1

2. Characterization of Se-1

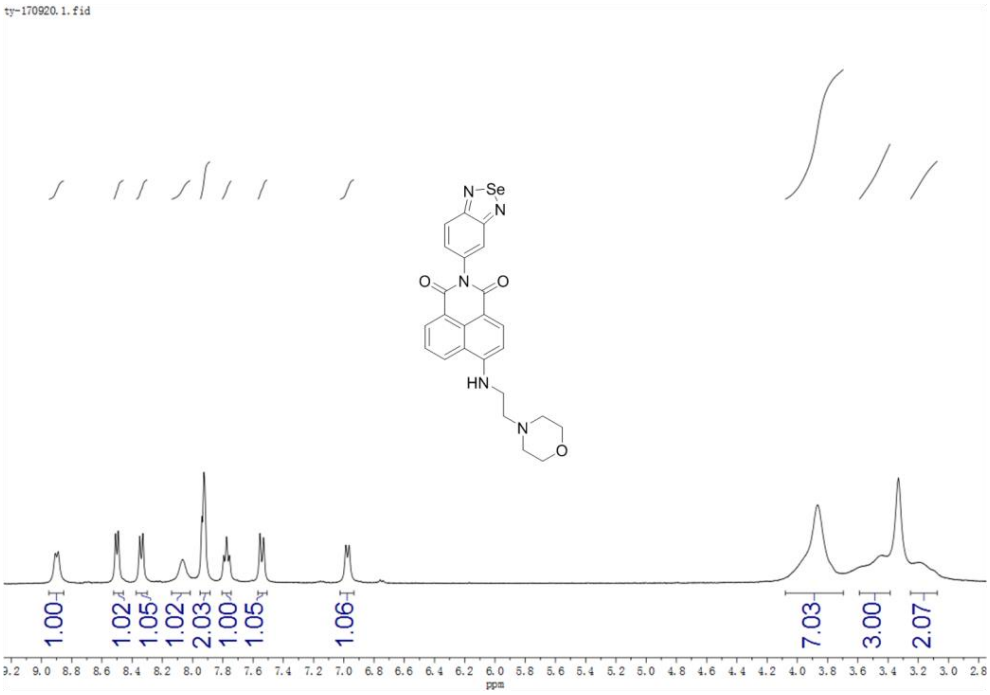


Figure S1. 1H NMR of Se-1

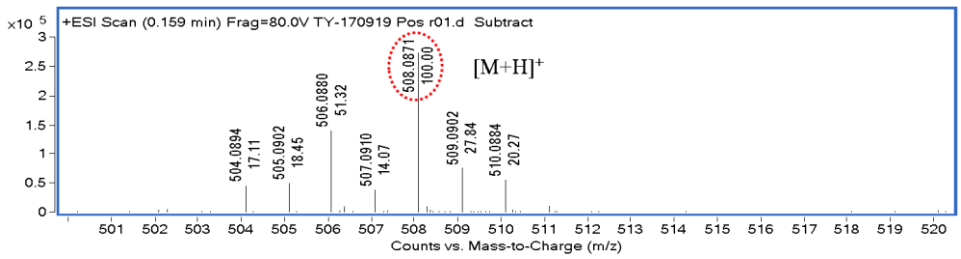


Figure S2. HR-MS spectrum of Se-1

3. Time-dependent fluorescence responses of Se-1 toward H_2Se

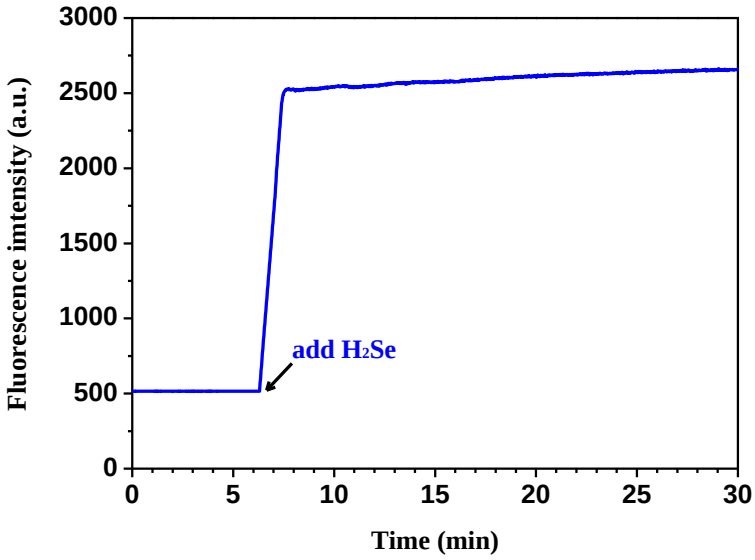


Figure S3. Time-dependent fluorescence responses of Se-1 (10 μM) incubated with 60 μM H_2Se at 535 nm, in acetic acid/sodium acetate buffer (20 mM, pH 5).

4. Characterization of Se-1 reacting with H_2Se

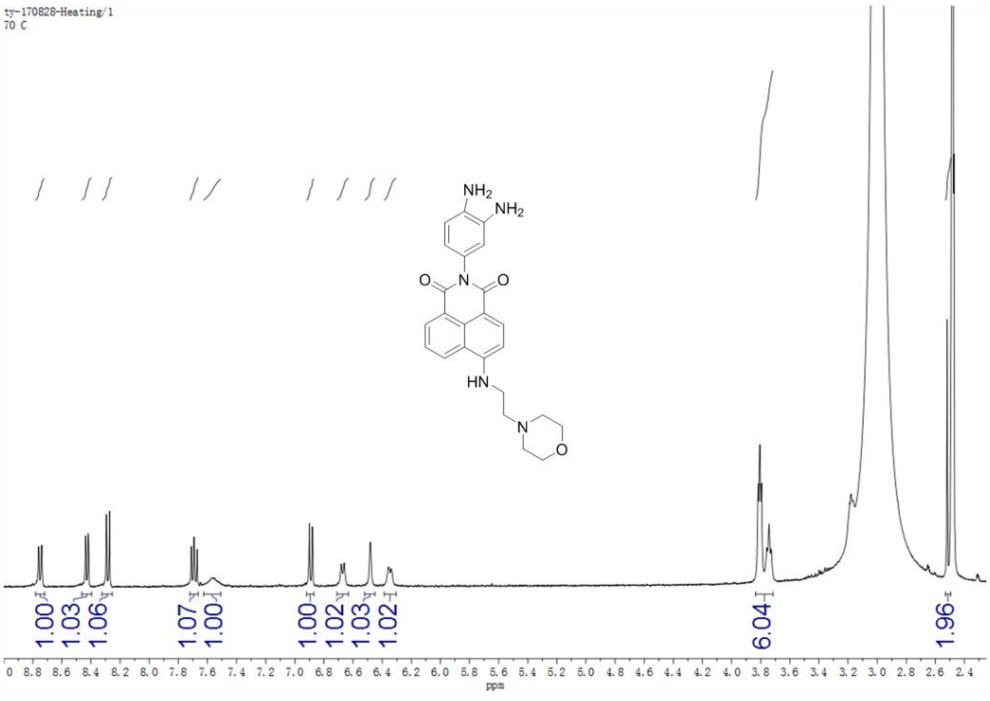


Figure S4. ^1H NMR spectrum of the product extracted from the mixture of Se-1 and H_2Se.

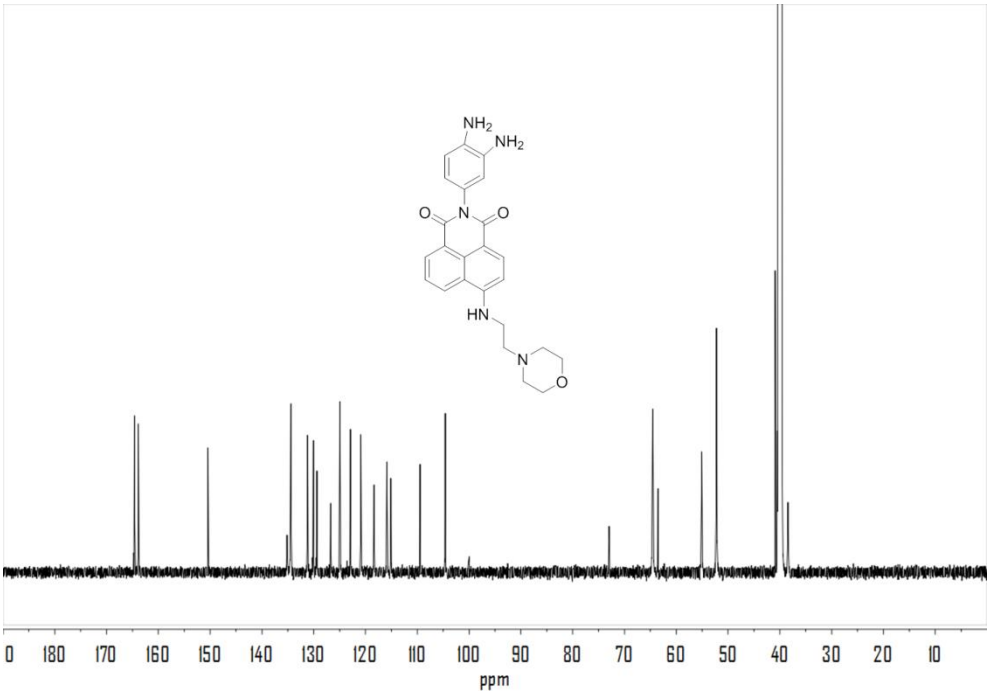


Figure S5. ^{13}C NMR spectrum of the product extracted from the mixture of Se-1 and H_2Se.

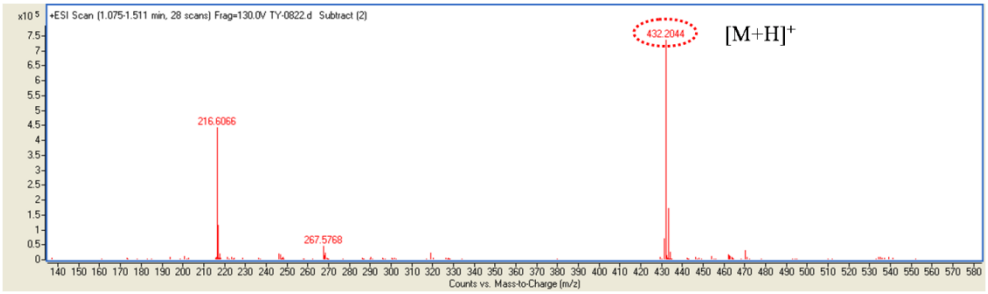


Figure S6. HR-MS spectrum of the product extracted from the mixture of Se-1 and H_2Se.

5. The pH effects

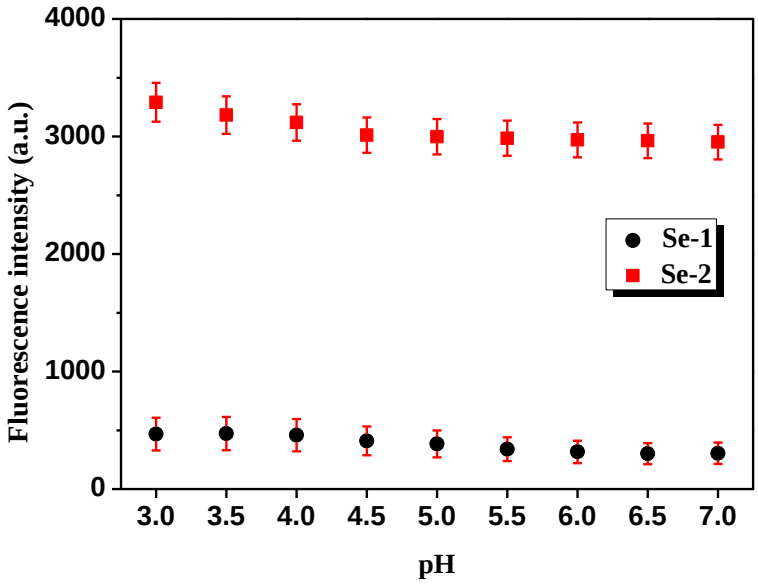


Figure S7. Effects of pH on relative fluorescence of Se-1 (10 μM) and Se-2 (10 μM) in acetic acid/sodium acetate buffer (20 mM), λ_{ex} = 435 nm.