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## **Electronic Supplementary Information**

## Reaction-based fluorometric analysis of *N*-bromosuccinimide by oxidative deprotection of dithiane

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**Fig. S1.** Fluorescence spectra of probe **1** in the presence of NCS, NBS, and NIS. [**1**] = 5.0  $\times$  10<sup>-6</sup> M, [*N*-halosuccinimide] = 5.0  $\times$  10<sup>-5</sup> M, [EDTA] = 1.0  $\times$  10<sup>-4</sup> M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex}$  = 340 nm.



**Fig. S2.** UV–vis spectra of probe **1** in the absence and presence of NBS.  $[1] = 1.0 \times 10^{-5}$  M, [NBS] =  $1.0 \times 10^{-4}$  M, [EDTA] =  $2.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).



Fig. S3. Ratiometric NBS-selective signaling of probe 1 expressed by the fluorescence intensity ratio ( $I_{460}/I_{376}$ ) at 460 nm and 376 nm. [1] =  $5.0 \times 10^{-6}$  M, [NBS] = [M<sup>n+</sup>] =  $5.0 \times 10^{-5}$  M, [EDTA] =  $1.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex} = 340$  nm.



**Fig. S4.** NBS-selective signaling of probe 1 expressed by the fluorescence intensity ratio  $(I/I_0)$  at 460 nm. [1] =  $5.0 \times 10^{-6}$  M, [NBS] =  $[A^{n-}] = 5.0 \times 10^{-5}$  M, [EDTA] =  $1.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex} = 340$  nm.



Fig. S5. Effect of the presence of background anions on the NBS signaling of probe 1 as expressed by the fluorescence intensity ratio ( $I_{(Anion+NBS)}/I_{NBS}$ ) at 460 nm. [1] =  $5.0 \times 10^{-6}$  M, [NBS] = [ $A^{n-}$ ] =  $5.0 \times 10^{-5}$  M, [EDTA] =  $1.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex}$  = 340 nm. Significantly reduced responses for iodide (highlighted in blue) and N<sub>3</sub><sup>-</sup> (highlighted in green) were due to the consumption of NBS by the relevant redox reactions.



Fig. S6. NBS signaling of probe 1 in the presence of background aromatic halides as expressed by the fluorescence intensity at 460 nm.  $[1] = 5.0 \times 10^{-6}$  M,  $[NBS] = [aromatic halides] = 5.0 \times 10^{-5}$  M,  $[EDTA] = 1.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex} = 340$  nm.



Fig. S7. Mass spectrum of the NBS-signaling product of probe 1.



**Fig. S8.** Time-course plot of NBS signaling by probe **2** expressed by the fluorescence intensity change at 436 nm. [**2**] =  $5.0 \times 10^{-6}$  M, [NBS] =  $5.0 \times 10^{-5}$  M, [EDTA] =  $1.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex} = 323$  nm.



Fig. S9. Effect of pH on the NBS signaling of probe 1 monitored by the changes in fluorescence intensity at 460 nm.  $[1] = 5.0 \times 10^{-6}$  M,  $[NBS] = 5.0 \times 10^{-5}$  M,  $[EDTA] = 1.0 \times 10^{-4}$  M in a mixture of acetate buffer (pH 4.76, 20 mM) containing varying amounts of 0.1 M NaOH and acetonitrile (1:1, v/v).  $\lambda_{ex} = 340$  nm.



**Fig. S10.** Changes in fluorescence intensity at 460 nm of probe **1** as a function of NBS concentration.  $[\mathbf{1}] = 5.0 \times 10^{-6} \text{ M}$ ,  $[\text{NBS}] = 0 - 1.0 \times 10^{-5} \text{ M}$ ,  $[\text{EDTA}] = 1.0 \times 10^{-4} \text{ M}$  in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{\text{ex}} = 340 \text{ nm}$ .



Fig. S11. Plots of the red, green, and blue channel levels of signal images obtained using a smartphone under 365 nm UV LED illumination as a function of NBS concentration.  $[1] = 5.0 \times 10^{-6} \text{ M}, [\text{NBS}] = 0-5.0 \times 10^{-6} \text{ M}, [\text{EDTA}] = 1.0 \times 10^{-4} \text{ M}$  in a mixture of acetate buffer (pH 4.76, 20 mM) and acetonitrile (1:1, v/v).  $\lambda_{ex} = 340$  nm.





Fig. S12. <sup>1</sup>H NMR spectrum of probe 1 in CDCl<sub>3</sub> (600 MHz).

Fig. S13. <sup>13</sup>C NMR spectrum of probe 1 in CDCl<sub>3</sub> (150 MHz).







Fig. S15. <sup>1</sup>H NMR spectrum of probe 2 in CDCl<sub>3</sub> (600 MHz).



Fig. S16. <sup>13</sup>C NMR spectrum of probe 2 in CDCl<sub>3</sub> (150 MHz).



Fig. S17. Electron ionization mass spectrum of probe 2.