Electronic Supplementary Material (ESI) for Journal of Materials Chemistry B. This journal is © The Royal Society of Chemistry 2016

Supporting Information (SI)

Yutao Yang,^{a,b†} Fangjun Huo,^{b†} Caixia Yin,^{a*} Ming Xu,^c Ying Hu,^d Jianbin Chao,^b Yongbin Zhang,^b Timothy E. Glass*^c and Juyoung Yoon*^d

^aInstitute of Molecular Science, Shanxi University, Taiyuan 030006, China; ^bResearch Institute of Applied Chemistry, Shanxi University, Taiyuan 030006, China; ^cDepartment of Chemistry, University of Missouri 601 South college Avenue, Columbia, Missouri 65211, United States; ^dDepartment of Chemistry and Nano Science, Ewha Womans University, Seoul 120-750, Korea

*Correspondence - yincx@sxu.edu.cn; glasst@missouri.edu; jyoon@ewha.ac.kr

Figure S1: The characterization data, ¹H NMR, ¹³C NMR, ESI-MS of the probe and probe-ClO⁻

Figure S2: The characterization data, ¹H NMR, ¹³C NMR, ESI-MS of probe-ClO⁻

- Figure S3: UV–vis spectra of 1 (25 μ M) in HEPES (10 mM, pH 7.4) upon addition of various concentrations of ClO⁻
- Figure S4: The corresponding fluorescence intensity of 1 upon addition of sodium hypochlorite disinfectant.
- Figure S5: Choice of pH range for the measurements

Experimental Section..

Synthesis of probe 1: The heterocyclic compound was obtained as shown in Scheme 1. Salicylhydroxamic acid (compound 1, 1 mmol,) was dissolved in methanol (20 mL), then excess sodium hypochlorite was poured into the solution. The mixture was placed at ambient temperature for 30 s to complete the reaction. The final mixture was diluted by water (20 ml) and extracted with ethyl acetate (3×30 mL). The organic layer was concentrated under reduced pressure to give the desired product as a pale-yellow solid (compound 2, 110 mg, 82% yield) ¹H NMR (300 MHz, 25 °C, DMSO-*d*₆): δ 7.58 (d, 1H, J=2.7), 7.23 (d, 1H, J=2.8), 7.08 (m, 2H, J=23.7), 6.61 (d, 1H, J = 8.7 Hz); ¹³C NMR (75 MHz, DMSO-*d*₆): δ 168.9, 159.8 131.3, 128.9, 123.7, 121.7, 117.6, 113.8 ESI-MS m/z [probe+Na] 158.33; Elemental analysis (calcd. %) for C₇H₅NO₂: C, 62.22; H, 3.73; N, 10.37; Found: C, 62.35; H, 3.68; N, 10.24.



Methods. All spectroscopic measurements were performed in HEPES (10 mM, pH 7.4) buffer. HEPES buffer solutions were obtained by adding 1 M NaOH solution into 10 mM aqueous HEPES using a pH meter. The probe was dissolved in absolute CH_3OH to prepare the stock solutions with concentrations of 2.0 mM. The UV-Visible spectra and fluorescence spectra were recorded at 25 °C.

Measurement procedure The UV-Vis procedures were shown as follows: into a HEPES (10 mM, pH 7.4) buffer solution containing 25 μ M probe, ClO⁻ sample was gradually titrated. All UV-Vis spectra data were recorded at 30 s after the ClO⁻ addition.

The fluorescence procedures were as follows: into a HEPES (10 mM, pH 7.4) buffer solution containing 2.5 μ M probe 1, ClO⁻ sample was gradually titrated. All fluorescence spectra data were recorded at 30 s after the ClO⁻ addition.

The HepG2 cells were grown in 1×SPP medium (1% proteose peptone, 0.2% glucose, 0.1% yeast extract, 0.003% EDTA ferric sodium salt) at 30 °C. The HepG2 were treated with 2.5 μ M of probe **1** in culture media for 30 min at 37 °C and washed three times with phosphate-buffered saline (PBS).



The ¹³CNMR (75MHz) spectra of the probe in DMSO- d_6 .



Figure S1: ¹H NMR (300 MHz, 25 °C, DMSO-*d*₆): δ 12.21 (s, 1H), 11.41 (s, 1H), 9.31(s, 1H), 7.68 (d, 1H, *J* = 9.9 Hz), 7.39 (m, 1H, *J* = 15.5 Hz), 6.91 (m, 2H, *J* = 25.2 Hz), ¹³C NMR (75 MHz, DMSO-*d*₆): δ 166.1, 159.2, 133.1, 126.8, 145.7, 118.5, 117.1, 113.8 ESI-MS m/z 152.50; Elemental analysis (calcd. %) for C₇H₇NO₃: C, 54.91; H, 4.61; N, 9.15; Found: C, 54.68; H, 4.39; N, 9.21.

Figure S2: The characterization data, ¹H NMR, ¹³C NMR, ESI-MS of probe-ClO⁻



The ¹³CNMR (75MHz) spectra of the probe-ClO⁻ in DMSO- d_6



Figure S2: ¹H NMR (300 MHz, 25 °C, DMSO-*d*₆): δ 7.58 (d, 1H, J=2.7), 7.23 (d, 1H, J=2.8), 7.08(m, 2H, J=23.7), 6.61 (d, 1H, *J* = 8.7 Hz) ¹³C NMR (75 MHz, DMSO-*d*₆): δ 168.9, 159.8 131.3, 128.9, 123.7, 121.7, 117.6, 113.8 ESI-MS m/z [probe+Na] 158.33; Elemental analysis (calcd. %) for C₇H₅NO₂: C, 62.22; H, 3.73; N, 10.37; Found: C, 62.35; H, 3.68; N, 10.24.



Figure S3:UV-Vis spectra of probe (25 μ M) in HEPES (10 mM, pH = 7.4) upon addition of various concentrations of ClO⁻ (0 – 250 μ M).



Figure S4: The corresponding fluorescence intensity of 1 upon addition of sodium hypochlorite disinfectant with different volumes: 0, 7, 12, 29 μ L, respectively.

Table S1.^a

	Added (µM) ^b	ClO-	Found ClO ⁻ $(\mu M)^{c}$	% Recovery
1	.2		1.23±0.01	98.4
2	.5		1.50±0.03	96.8
3	8		1.86±0.02	100.5

^aInitial solution contained 100,000 fold diluted disinfectant, 2.5 µM probe in 10 mM HEPES, pH 7.4. ^bConcentration of pure NaOCl solution added. ^cCalculated by fluorescence.

Figure S5: Choice of pH range for the measurements





Figure S5: (a) Fluorescence intensity of free probe (2.5 μ M) under different pH conditions. b) Fluorescence intensity of free probe+ClO⁻ under different pH conditions. (c) The fluorescence intensity of probe at 415 nm in the absence and presence of Cys under different pH (λ ex = 300 nm; Slit: 5nm/5 nm).