

Table S1. Chemicals and Reagents Used in This Study

Chemical / Reagent	Purity / Grade	Supplier
4-Methyl-7-hydroxycoumarin	99%	Sinopharm Group
2-Methylbenzothiazole	98%	Sinopharm Group
Hexamethylenetetramine (HMTA)	Standard solution	Sinopharm Group
Iodoethane	Standard solution	Sinopharm Group
Glacial acetic acid (CH ₃ COOH)	Analytical grade	Sinopharm Group
Diethyl ether	Analytical grade	Sinopharm Group
Dimethyl sulfoxide (DMSO)	Analytical grade	Sinopharm Group
Piperidine	Analytical grade	Sinopharm Group
Magnesium sulfate (MgSO ₄)	Analytical grade	Sinopharm Group
Potassium carbonate (K ₂ CO ₃)	Analytical grade	Sinopharm Group
Toluene	Analytical grade	Sinopharm Group
Dichloromethane	Analytical grade	Sinopharm Group
Chloroform	Analytical grade	Sinopharm Group
Ethyl acetate	Analytical grade	Sinopharm Group
Petroleum ether	Analytical grade	Sinopharm Group
Sodium hydroxide (NaOH)	Analytical grade	Sinopharm Group
Sodium hypochlorite(NaClO)	Analytical grade	Sinopharm Group
Hypochlorous acid (HClO)	Analytical grade	Sinopharm Group
Sodium bromide (NaBr)	Analytical grade	Sinopharm Group
Sodium acetate	Analytical grade	Sinopharm Group
Sodium chloride (NaCl)	Analytical grade	Sinopharm Group
Sodium iodide (NaI)	Analytical grade	Sinopharm Group
Sodium bisulfite (NaHSO ₃)	Analytical grade	Sinopharm Group
Sodium bisulfate (NaHSO ₄)	Analytical grade	Sinopharm Group

Sodium phosphate (Na ₃ PO ₄)	Analytical grade	Sinopharm Group
Sodium sulfate decahydrate (Na ₂ SO ₄ ·10H ₂ O)	Analytical grade	Sinopharm Group
Sodium nitrite (NaNO ₂)	Analytical grade	Sinopharm Group
Sodium sulfite (Na ₂ SO ₃)	Analytical grade	Sinopharm Group
Sodium bicarbonate (NaHCO ₃)	Analytical grade	Sinopharm Group
Hydrogen peroxide (H ₂ O ₂)	Analytical grade	Sinopharm Group
Ferrous sulfate (FeSO ₄)	Analytical grade	Sinopharm Group
Disodium hydrogen phosphate (Na ₂ HPO ₄)	Analytical grade	Sinopharm Group
Sodium dihydrogen phosphate (NaH ₂ PO ₄)	Analytical grade	Sinopharm Group
Cysteine	≥98.5%	Commercial supplier
Homocysteine	≥95%	Commercial supplier
Glutathione	98%	Commercial supplier
Lipopolysaccharide (LPS)	B.R.	Commercial supplier
Nystatin	USP	Commercial supplier
Monensin	98%	Commercial supplier

Note: All solvents were used as received without further purification. Deionized water (18.2 MΩ·cm) was used for all aqueous solutions. Ronghai Type G high-performance silica gel TLC plates.

Table S2. Instruments Used in This Study

Instrument	Model	Manufacturer
LC–QTOF Mass Spectrometer	Agilent 6530	Agilent Technologies
Nuclear Magnetic Resonance Spectrometer	AVANCE 500 MHz	Bruker
Electronic Balance	AR2140 (0.1 mg precision)	Ohaus
Thermostatic Magnetic Stirrer	DF-101S	Shanghai Yiheng
UV–Vis Spectrophotometer	Lambda 750s	PerkinElmer

Fluorescence Spectrophotometer	F-7100	Hitachi
Portable UV Analyzer	ZF-7A	Shanghai Jingke
pH Meter	pHS-2F	Leici
Biological Incubator	233003-23428	Thermo Scientific
Laser Scanning Confocal Microscope	LSM880	Zeiss
Small Animal In Vivo Imaging System	Ami-13032	Spectral Instruments

Fig S1. ¹H-NMR Spectrum of 4-methyl-7-hydroxy-8-aldehyde coumarin

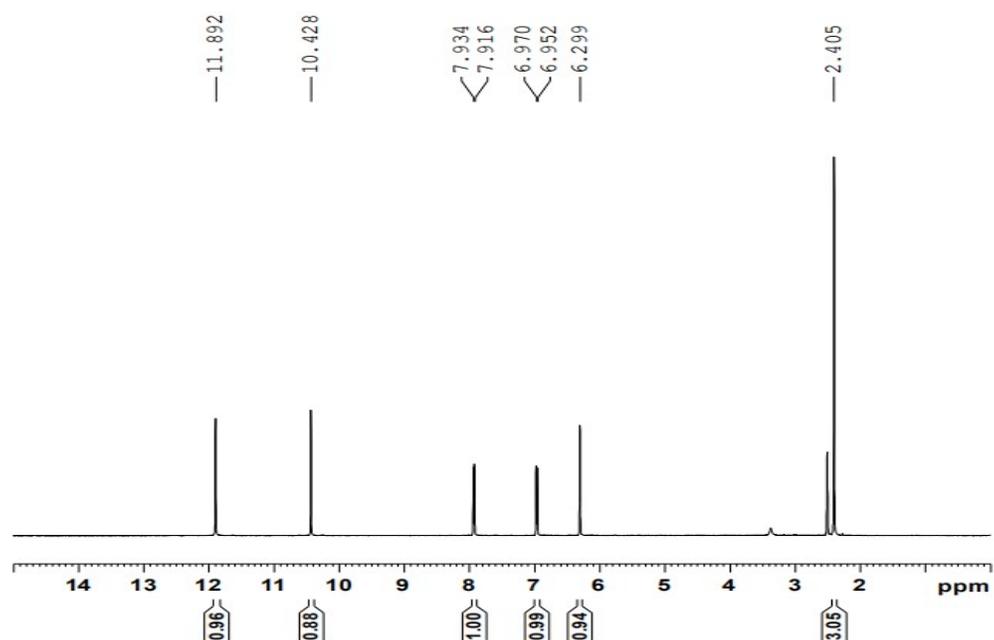


Fig S2. ^{13}C -NMR Spectrum of 4-methyl-7-hydroxy-8-aldehyde coumarin

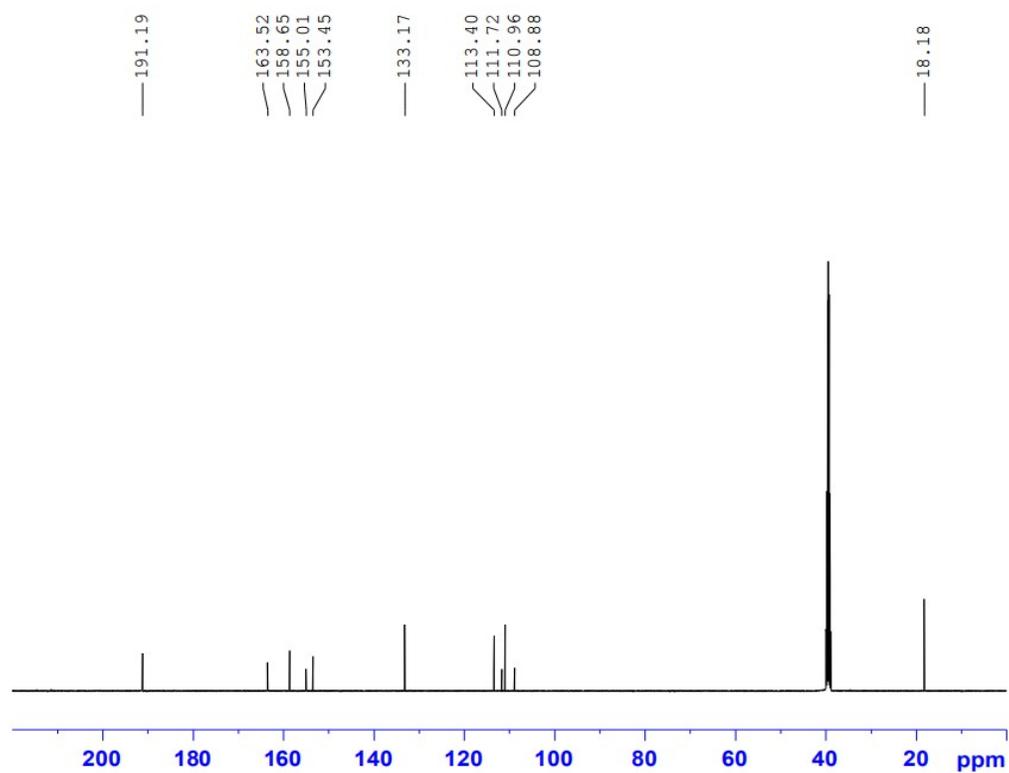


Fig S3. ^1H -NMR Spectrum of 2-methyl-3-ethylBenzothiazole iodized salt

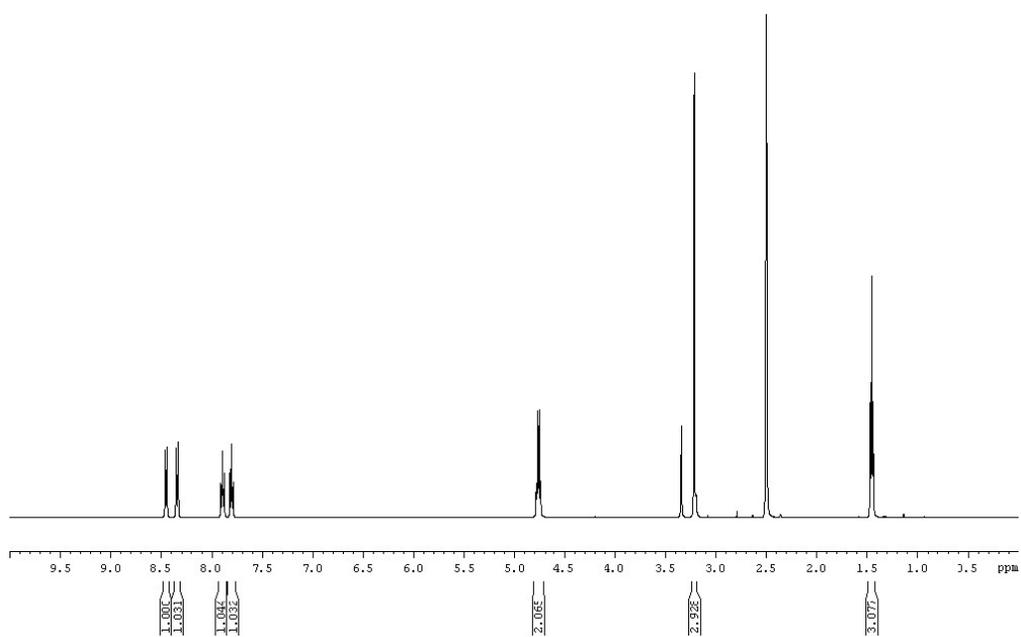


Fig S4. ^{13}C -NMR Spectrum of 2-methyl-3-ethylBenzothiazole iodized salt

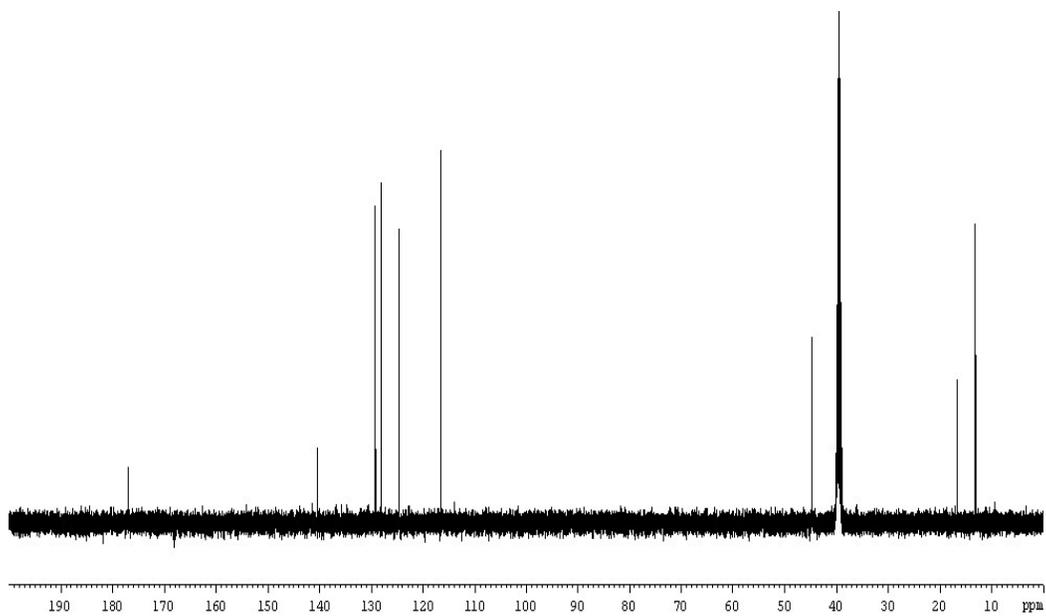


Fig S5. HR-MS Spectrum of 2-methyl-3-ethylBenzothiazole iodized salt

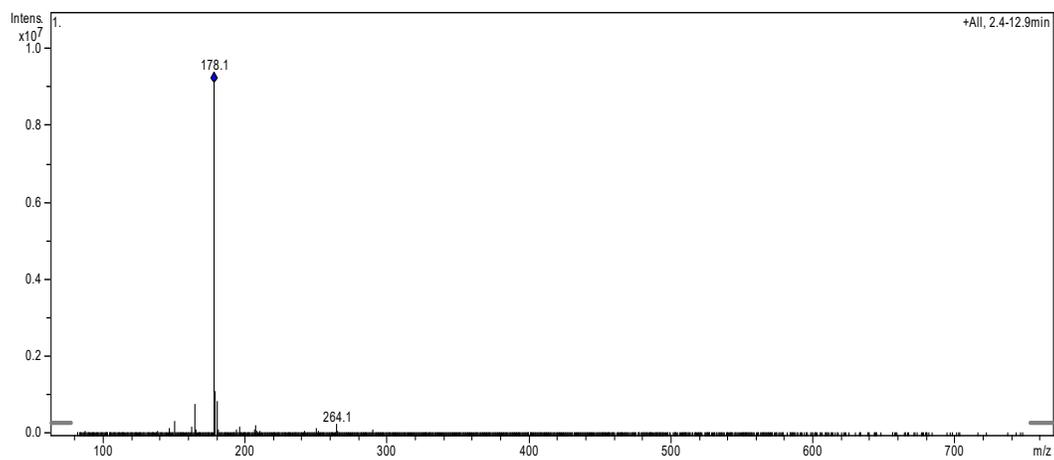


Fig S6. ¹H-NMR Spectrum of Probe XDS

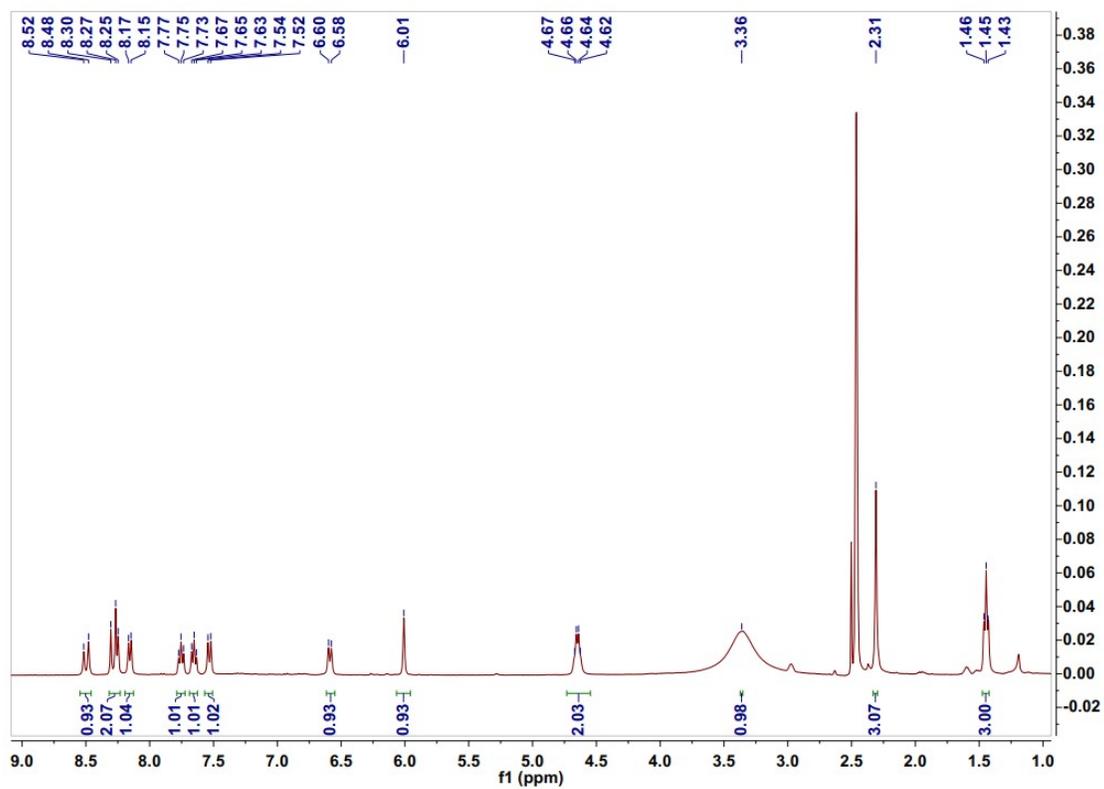


Fig S7. ¹³C-NMR Spectrum of Probe XDS

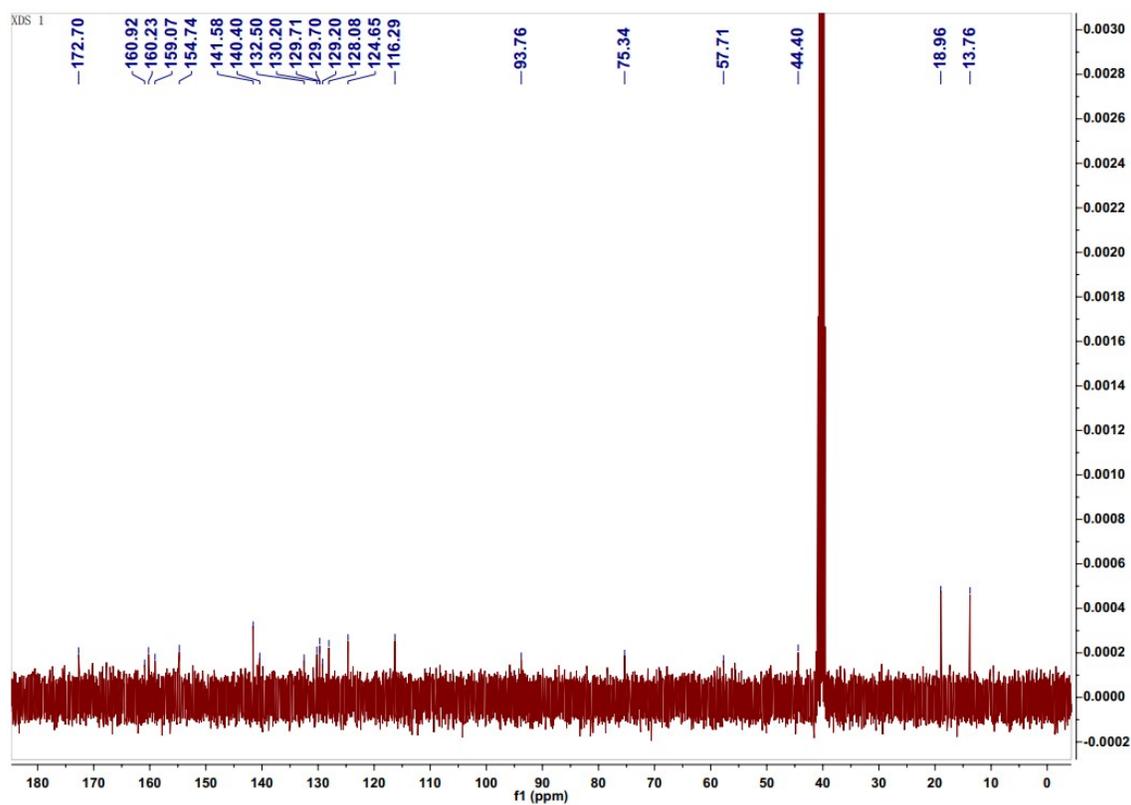


Fig S8. HR-MS Spectrum of Probe XDS

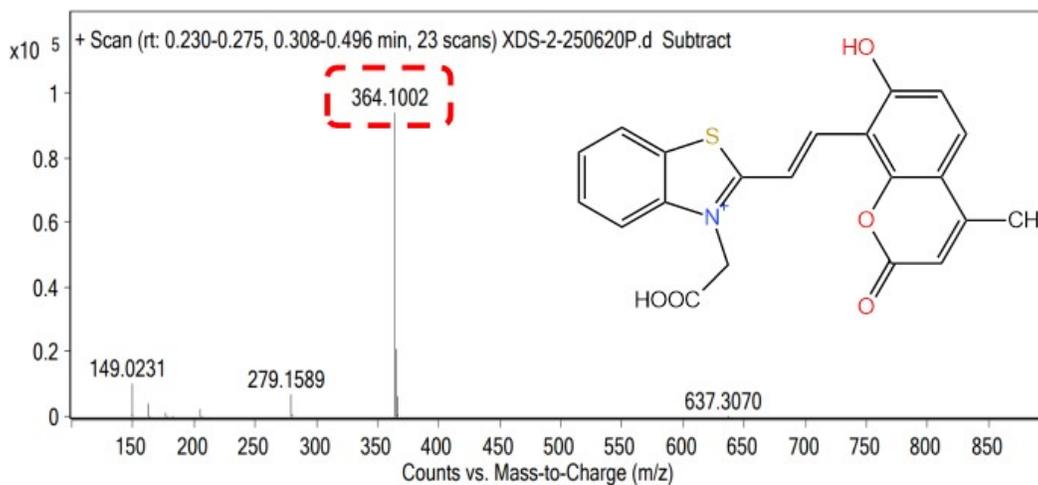


Fig S9. Synthesis of 4-methyl-7-hydroxy-8-aldehyde coumarin

Fig S10. Synthesis of 2-methyl-3-ethylBenzothiazole iodized salt

Fig S11. Synthesis of probe XDS

Fig S12. Solution color development digram.

Fig S13. Fluorescence color development digrams of XDS.

Fluorescence color development diagrams of XDS (10 μM) upon addition of 0.4 mM HClO and various other anions, biothiols and reactive oxygen species in DMSO/PBS(3:7, v/v, pH=7.4, 20 mM) ($\lambda_{\text{ex}}=388$ nm, $\lambda_{\text{em}}=456$ nm). (1.Blank, 2. ClO^- , 3. F^- , 4. Cl^- , 5. Br^- , 6. HSO_4^- , 7. HSO_3^- , 8. S^{2-} , 9. SO_3^{2-} , 10. SO_4^- , 11. HCO_3^- , 12. NO_2^- , 13. CH_3COO^- , 14. $\text{P}_2\text{O}_7^{4-}$, 15. PO_4^{3-} , 16. OH^- , 17.Cys, 18.Hcy, 19.GSH, 20. H_2O_2 , 21. ONOO^- , 22. $\bullet\text{OH}$, 23. 'O_2).

Fig S14. Fluorescence stability line plot of probe XDS in DMSO/PBS(3:7, v/v, pH=7.4, 20 mM) system within 60 hours.

