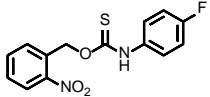
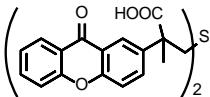
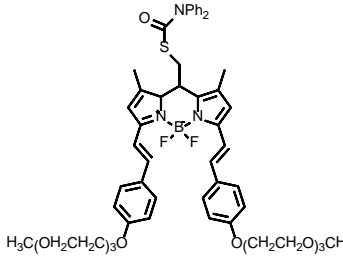
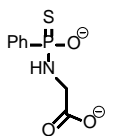
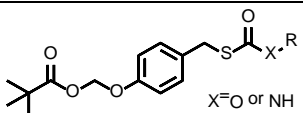
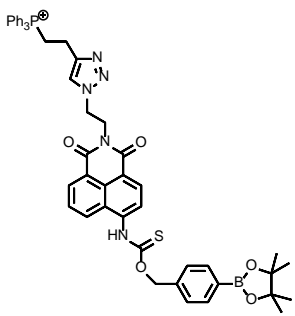
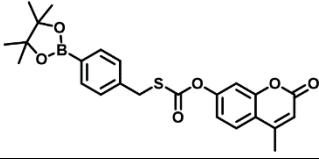
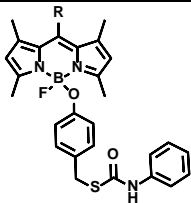
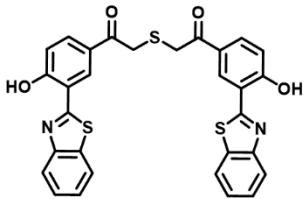
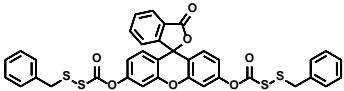
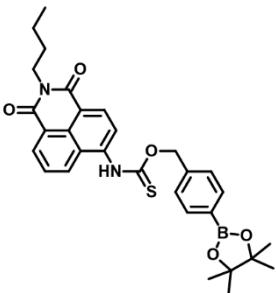
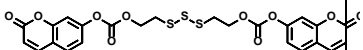
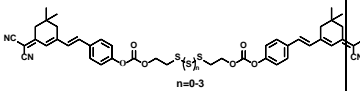
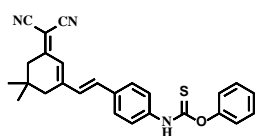
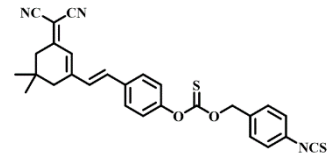


Table S1. Summary of activatable H₂S donors.

Fluorescent H ₂ S donors	Activation type	Optical parameters	H ₂ S detecting method	H ₂ S releasing efficiency	Ref
	UV Light (365 nm)	-	Methylene blue assay	^a N.F.	<i>Org. Lett.</i> 2017 , 19, 2278–2281
	UV Light (325–385 nm)	-	Methylene blue and HSip-1 assay	N.F.	<i>Bioorg. Med. Chem. Lett.</i> 2015 , 25, 175–178
	VIS-NIR (700 nm)	$\lambda_{\text{ex/em}} = 688/779 \text{ nm}$	Methylene blue and SF7-AM assay	48%	<i>Org. Lett.</i> 2018 , 20, 4907–4911
	pH	-	Methylene blue assay	N.F.	<i>J. Am. Chem. Soc.</i> 2016 , 138, 6336–6339
	Esterase	-	Dn-N3-based assay	N.F.	<i>Org. Lett.</i> 2017 , 19, 62–65
	ROS	$\lambda_{\text{ex/em}} = 405/540 \text{ nm}$	Methylene blue assay	N.F.	<i>ACS Sens.</i> 2020 , 5, 319–326
	ROS	$\lambda_{\text{ex/em}} = 365/448 \text{ nm}$	Methylene blue assay	N.F.	<i>Tetrahedron Lett.</i> 2021 , 69, 152944
	Visible-Light ($\lambda = 470 \text{ nm}$)	$\lambda_{\text{ex/em}} = 470/540 \text{ nm}$	Methylene blue and H ₂ S-sensitive electrode assay	30%~40%	<i>Org. Lett.</i> 2017 , 19, 4822–4825

	UV-Vis light ($\lambda > 410$ nm)	From green ($\lambda_{\text{ex/em}} = 350/517$ nm) to blue ($\lambda_{\text{ex/em}} = 350/450$ nm)	Coumarin-hemicyanine fluorescence dye and methylene blue assay	N.F.	<i>Chem. Commun.</i> 2018 , 54, 3106
	Cys	$\lambda_{\text{ex/em}} = 490/520$ nm	Methylene blue assay	75%	<i>Chem. Sci.</i> 2019 , 10, 1873–1878
	ROS	$\lambda_{\text{ex/em}} = 405/577$ nm	Methylene blue assay	N.F.	<i>Chem. Sci.</i> 2019 , 10, 7690–7694
	Biothiol	$\lambda_{\text{ex/em}} = 332/446$ nm	Fluorescent probe NAP-1 and methylene blue assay	N.F.	<i>Chem. Commun.</i> 2020 , 56, 7769–7772
	Thioredoxin reductase	$\lambda_{\text{ex/em}} = 580/670$ nm	Fluorescent probe WSP-2 and methylene blue assay	N.F.	<i>J. Mater. Chem. B</i> 2022 , 10, 2183–2193
	Cys	$\lambda_{\text{ex/em}} = 470/660$ nm	H ₂ S fluorescent probe C-7AZ	62%	<i>Anal. Chem.</i> 2021 , 93, 4894–4901
	Biothiols	$\lambda_{\text{ex/em}} = 530/666$ nm	H ₂ S fluorescent probe C-7AZ and methylene blue assay	72.9%	This work

^aN.F. means “Not Find”.

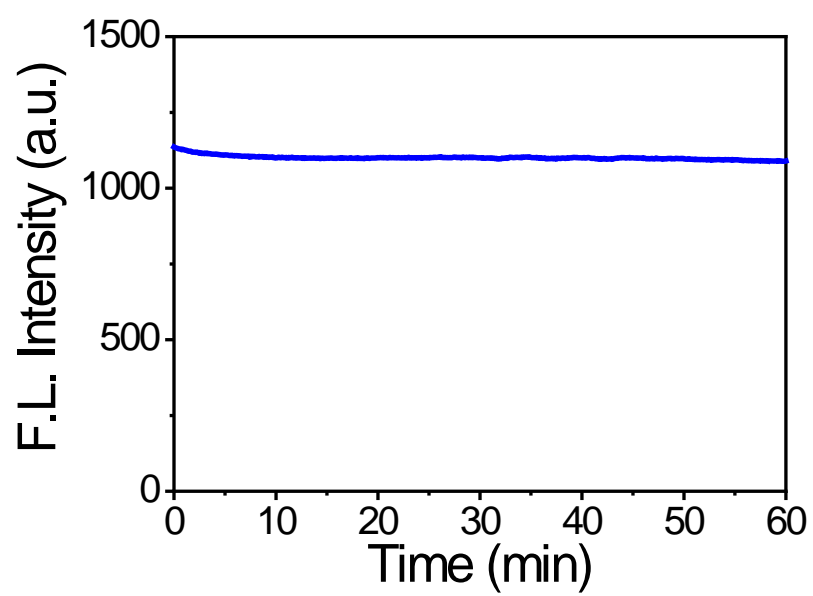


Figure R1. Fluorescence intensity of TCOO (10 μM) as a function of time.

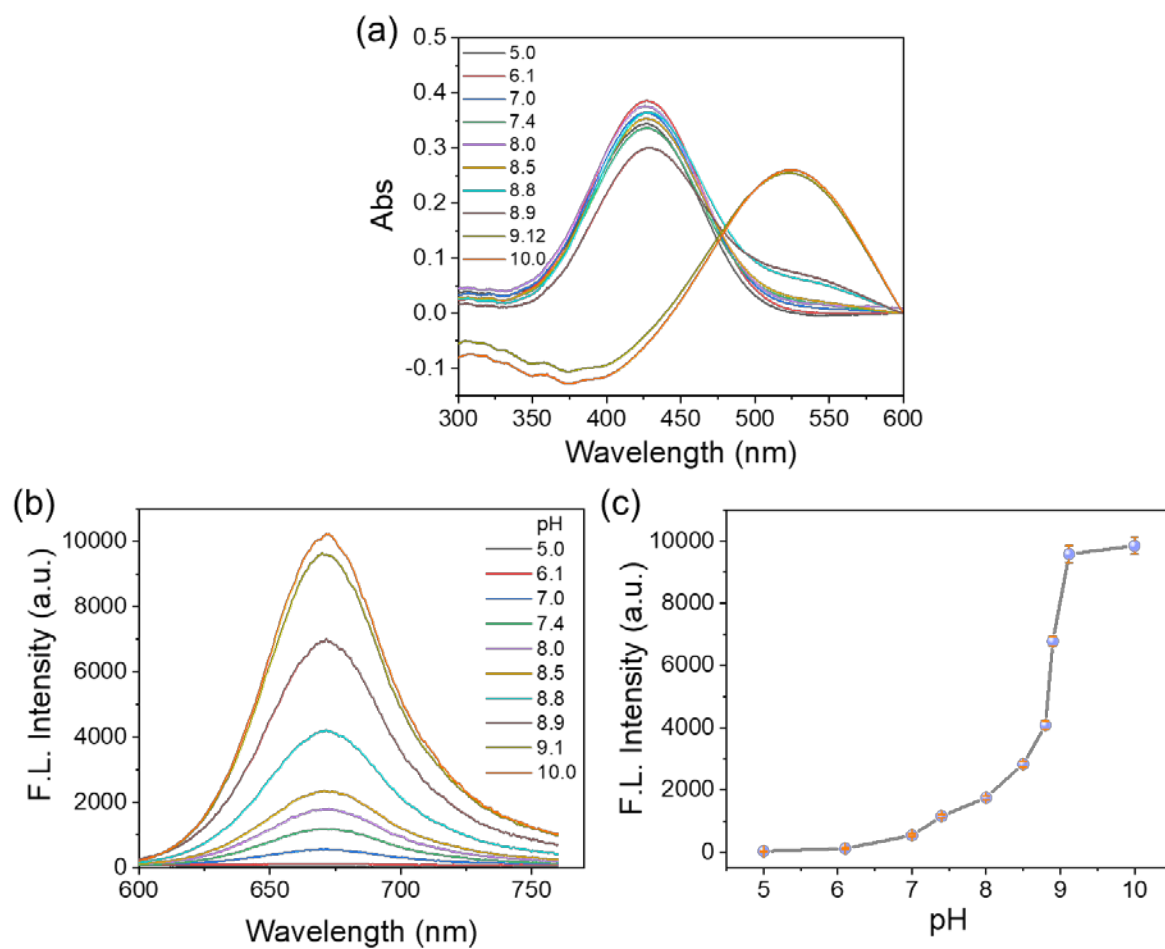


Figure R2. Absorption spectrum (a) and fluorescence spectrum (b) of TCOO (10 μ M) at various pH values. (c) Fluorescence intensity of **TCOO** at 666 nm as a function of pH. Conditions: DMF/PBS (20 mM, v/v, 3/7) at 37 $^{\circ}$ C for 30 min.

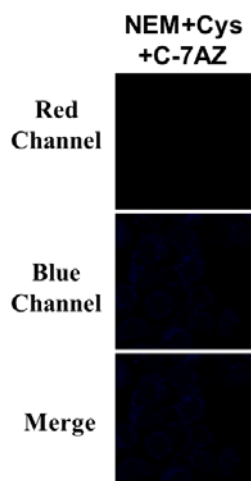
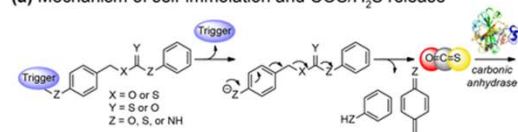
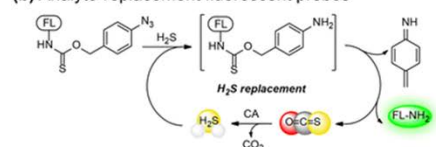


Figure R3. Confocal fluorescence images in RAW 246.7 cells. NEM-pretreated Cells were incubated with Cys (200 μ M) for 30 min and then with C-7AZ (3 μ M) for 30 min. Red channel: λ_{ex} =514 nm, λ_{em} =660 \pm 30 nm; blue channel: λ_{ex} =405 nm, λ_{em} =450 \pm 20 nm.

(a) Mechanism of self-immolation and COS/H₂S release



(b) Analyte-replacement fluorescent probes



(c) Representative self-immolative COS donors and triggers

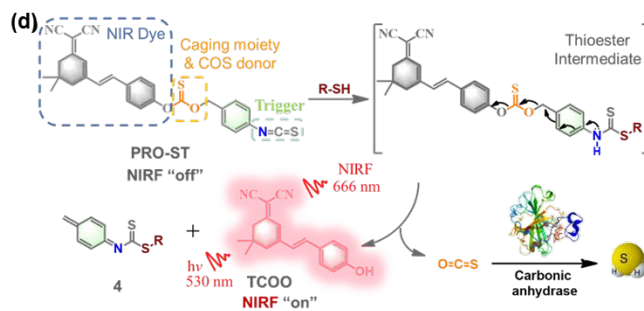
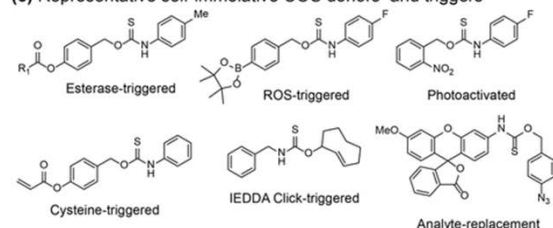


Figure R4. (a) Mechanism of self-immolation and subsequent conversion of COS to H₂S by CA. (b) Development of analyte-replacement fluorescent probes. (c) Current examples of self-immolation-based COS/H₂S donors. (d) This work.