Electronic Supplementary Material (ESI) for Materials Chemistry Frontiers.

Electronic Supplementary Information

A highly selective fluorescent nanoprobe based on AIE and ESIPT for imaging hydrogen sulfide in live cells and in vivo

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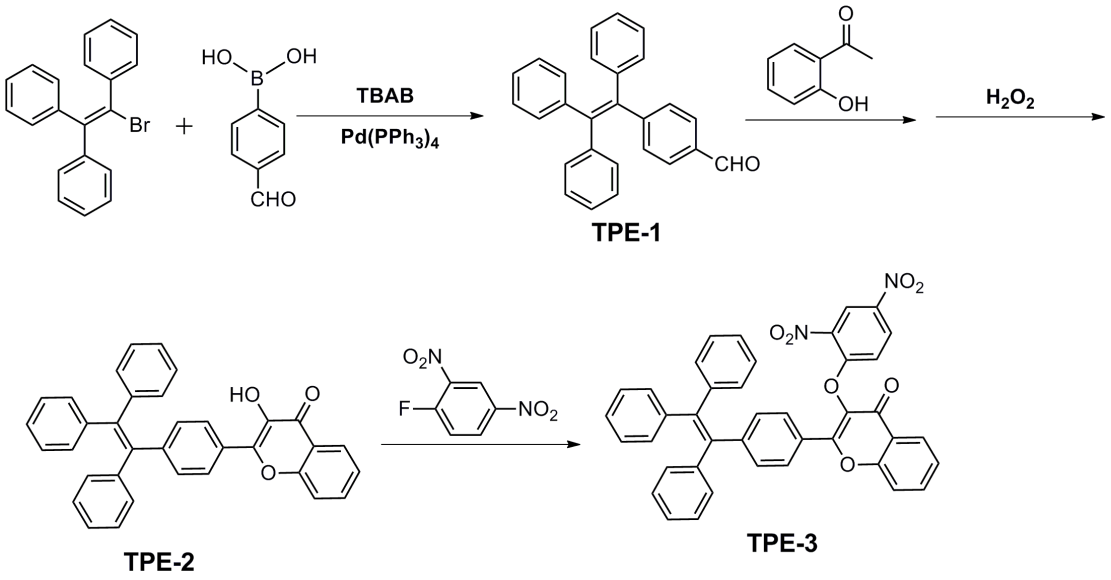
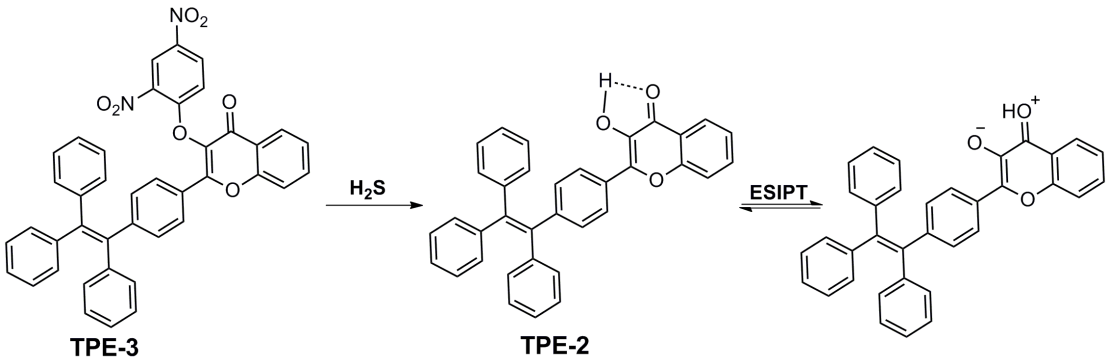


Scheme S1. Synthetic route of the probe (**TPE-3**).



Scheme S2. Possible reaction mechanism of TPE-3 with H_2S

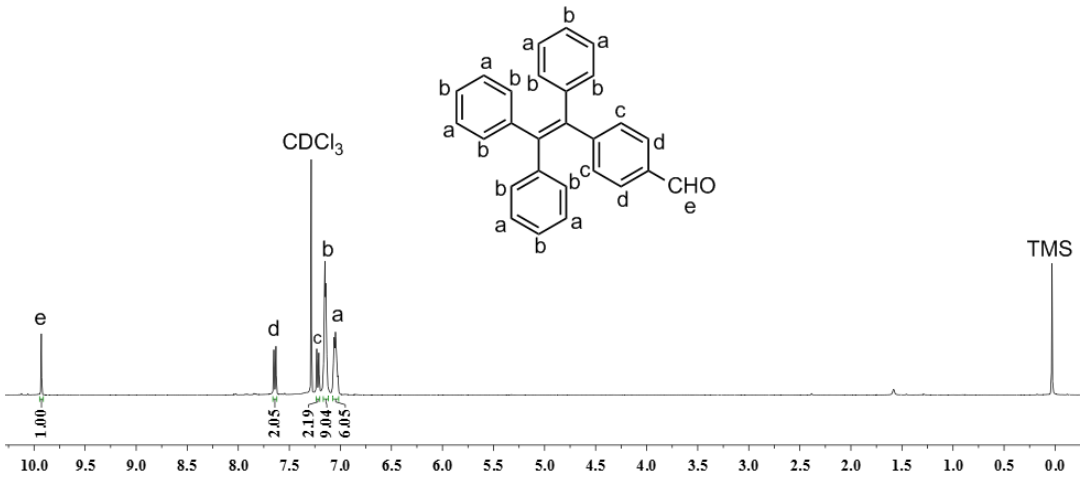
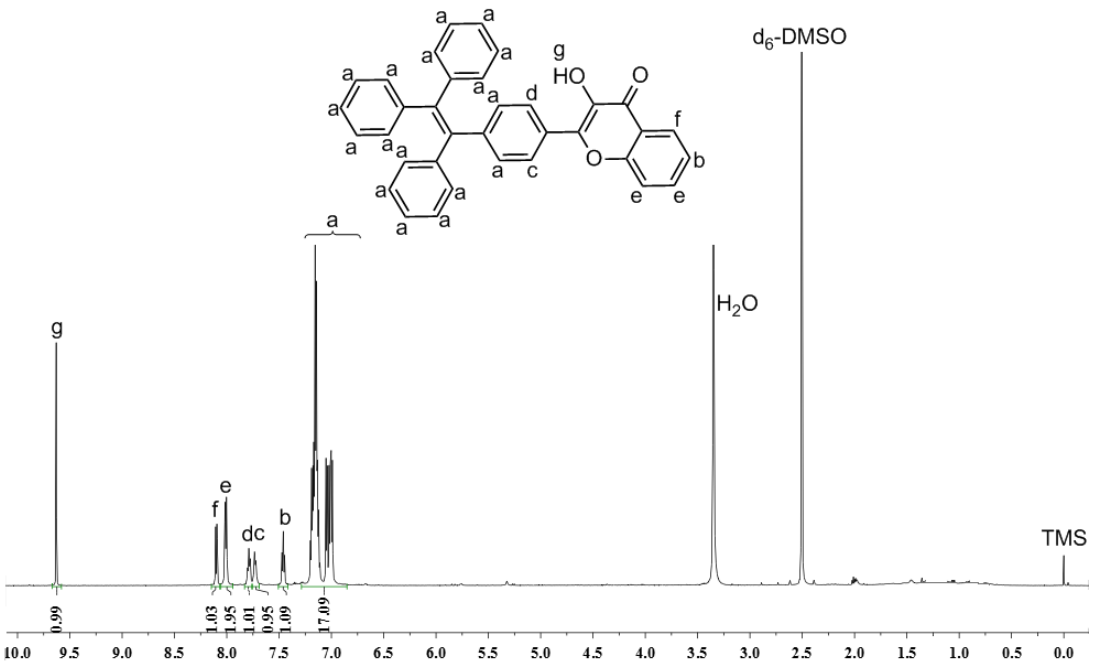


Figure S1. ^{1}H-NMR spectra ($CDCl_3$) of **TPE-1**.



(A)

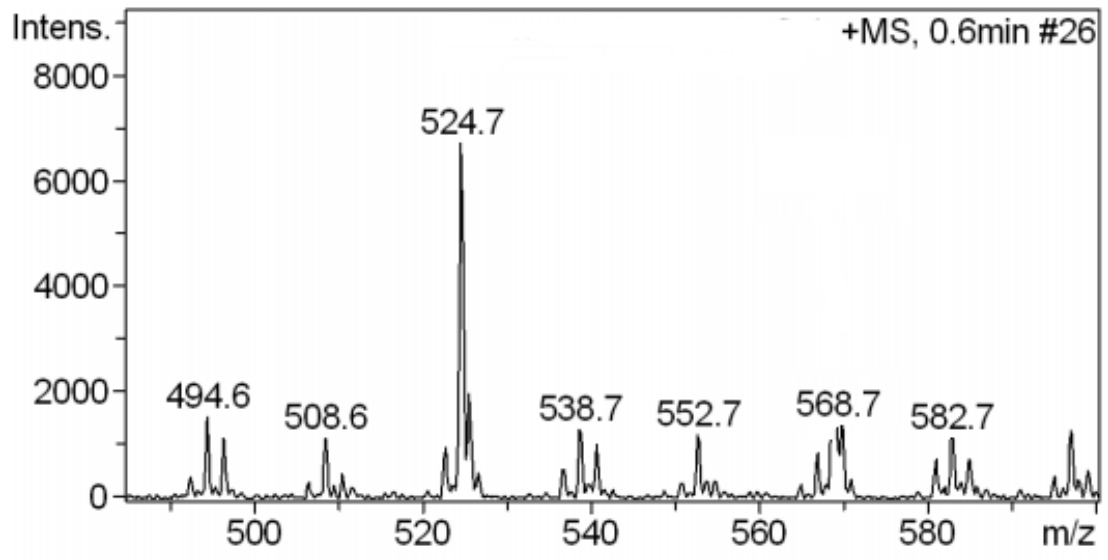
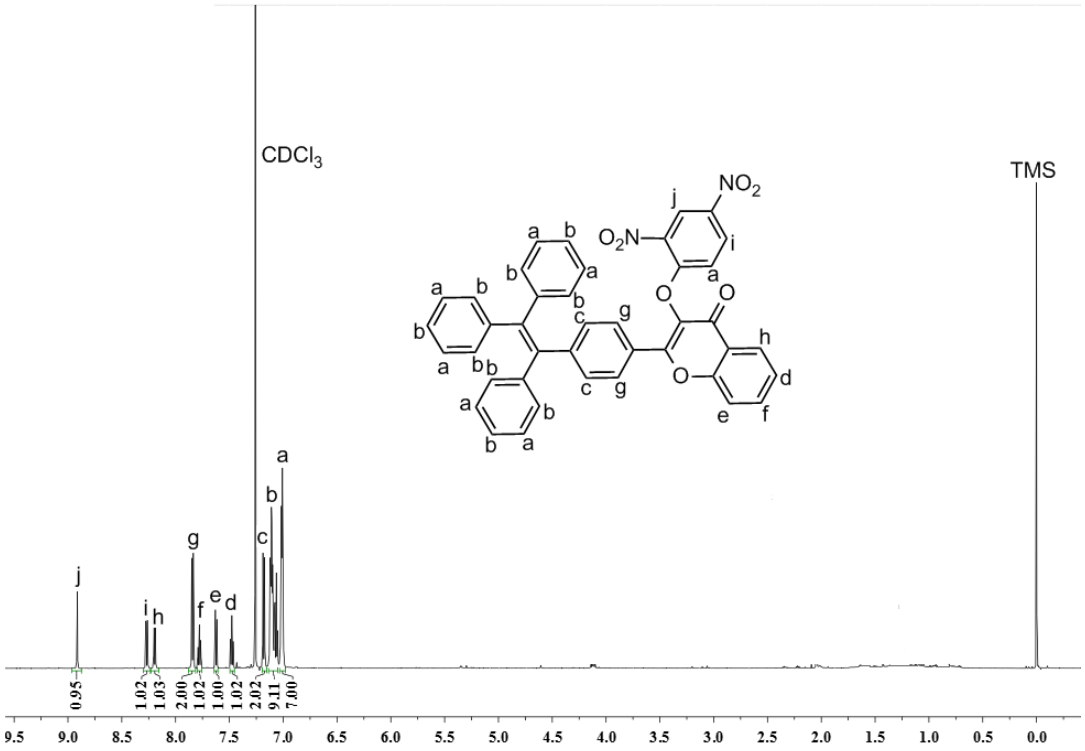
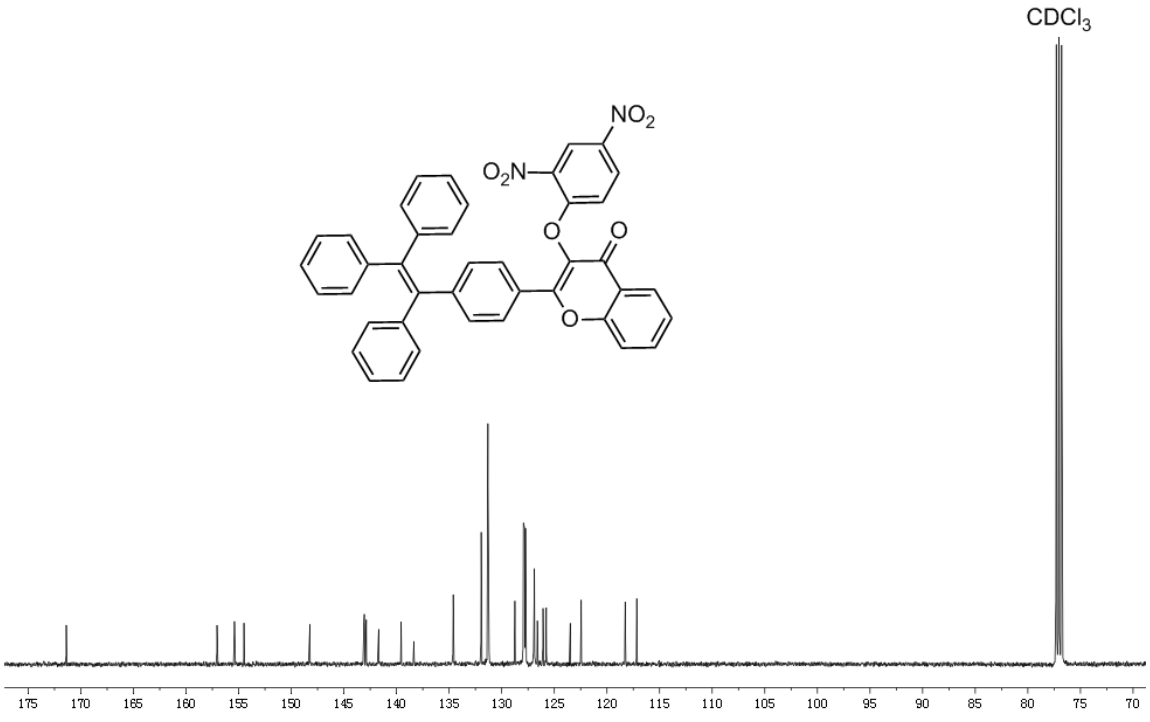


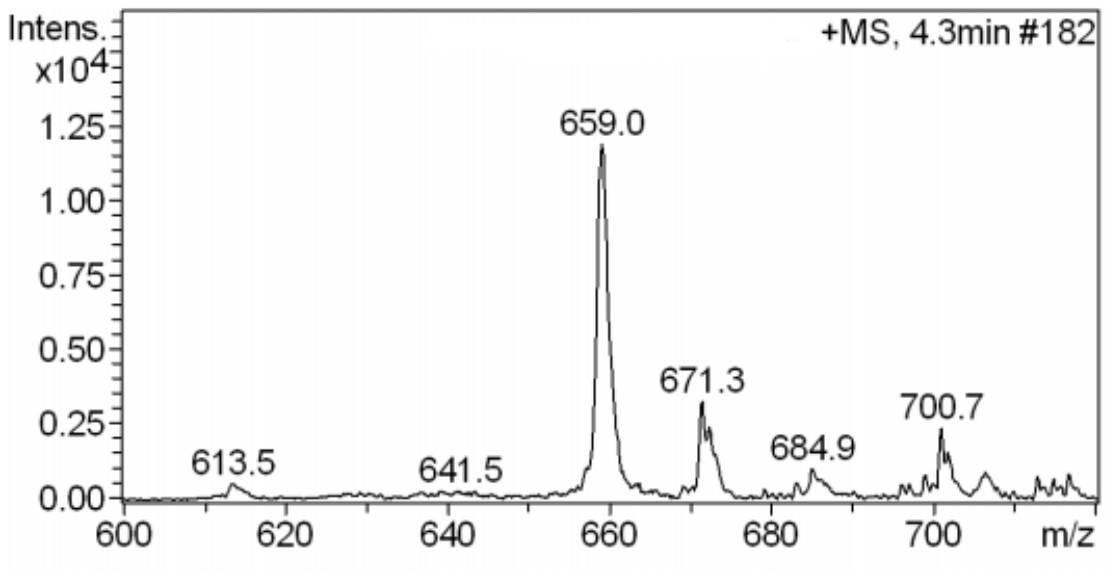
Figure S2. (A) ^{1}H-NMR spectrum (in d_6-DMSO) and (B) mass spectrum of **TPE-2** (m/z 524.7 $[M+CH_3OH]^+$).


CDCl3
TMS
NO2
O2N
O
O
j
i
h
g
f
e
d
c
b
a
0.95
1.02
1.03
2.00
1.02
1.00
1.02
2.02
9.11
7.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

(A)


CDCl3
NO2
O2N
O
O
O
175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70

(B)



(C)

Figure S3. (A) ^{1}H-NMR spectra ($CDCl_3$), (B) ^{13}C-NMR spectra ($CDCl_3$) and (B) mass spectrum of **TPE-3** (m/z 659.0 $[M+H]^+$).

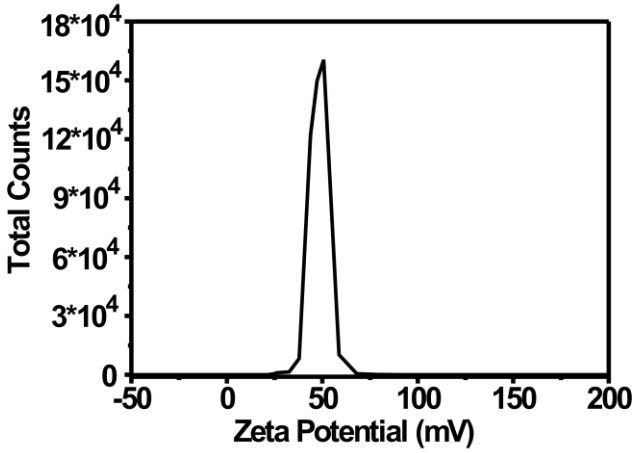


Figure S4. The zeta potential of the self-assembled nanoprobe.

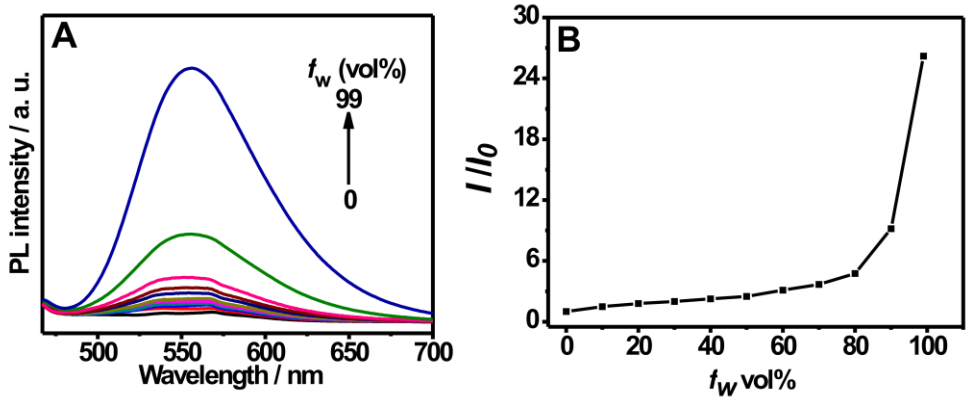


Figure S5. (A) Fluorescence emission spectra of TPE-2 in water/DMSO mixtures with varied water content. (B) Plot of I/I_0 at 550 nm versus water content (f_w), where I_0 is the fluorescence intensity in pure DMSO solution. λ_{ex} = 452 nm

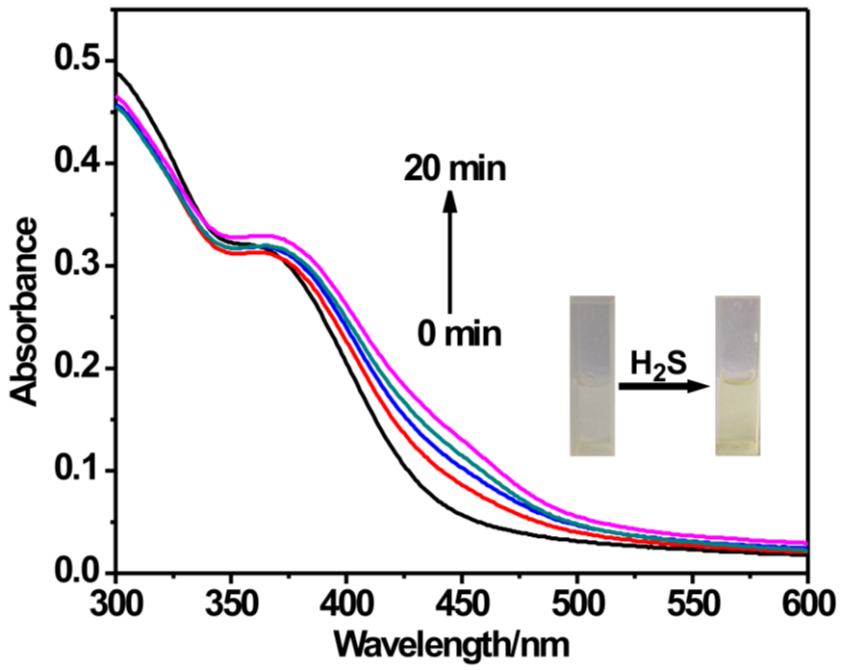


Figure S6. Time-dependent absorption spectrum of the nanoprobe (0.01 mg/mL) in the presence of H_2S (200 μM) in PBS buffer (10 mM, pH=7.4); The inset displays the photographs under ambient light.

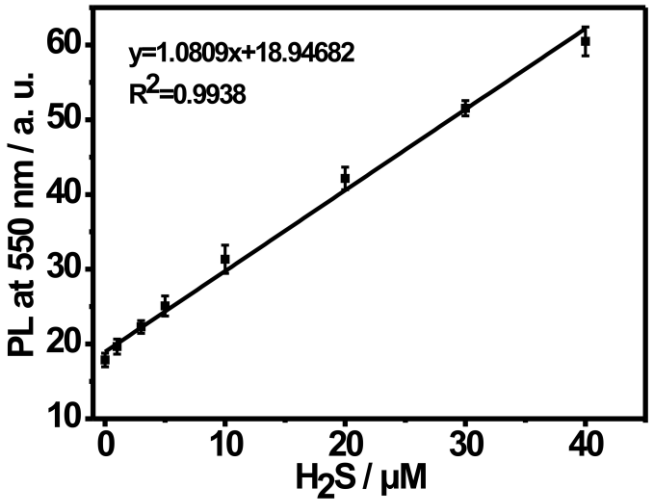


Figure S7. Plot of fluorescence intensity at 550 nm (I_{550}) versus H_2S concentrations.

Determination of the detection limit:

First the calibration curve was obtained from the plot of fluorescence intensity at 550 nm (I_{550}) versus H_2S concentrations. The regression curve equation was then obtained for the lower concentration part.

The detection limit = 3 × S.D. / k

where k is the slope of the curve equation, and S.D. represents the standard deviation for the fluorescence intensity at 550 nm (I_{550}) of nanoprobe in the absence of H_2S.

I_{550} = 18.94682 + 1.0809 × [H_2S](R^2=0.9938)

LOD = 3 × 0.0323 / 1.0809 = 0.09 μM

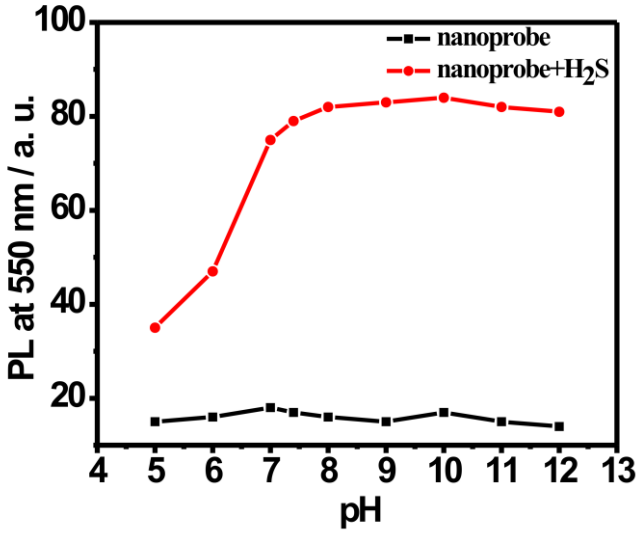


Figure S8. The effect of pH on the fluorescence intensity at 550 nm (I_{550}) of the nanoprobe (0.01 mg/mL) in the absence or presence of H_2S (200 μM). λ_{ex} = 452 nm.

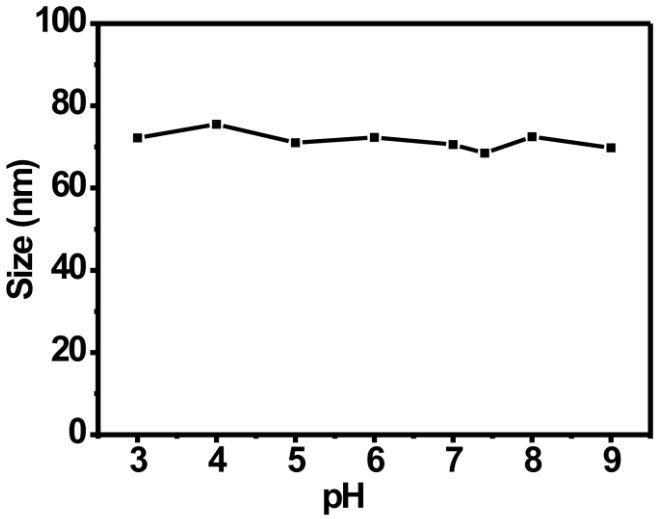
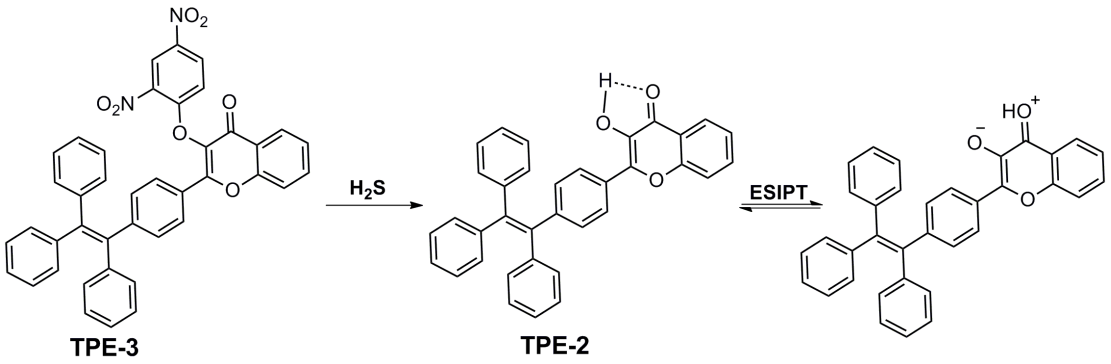
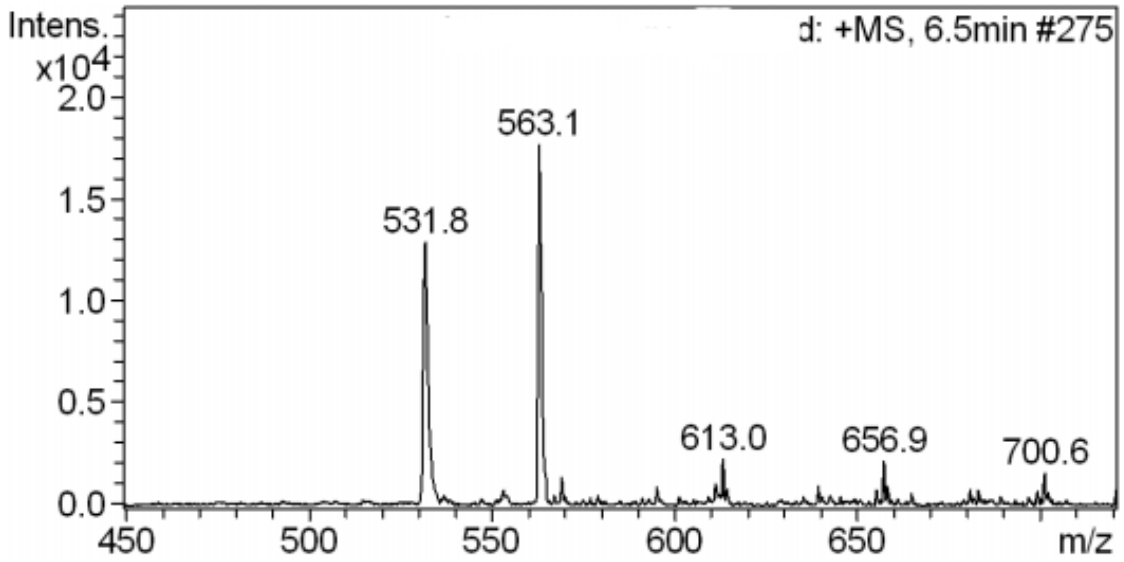


Figure S9. The effect of pH on the size of the self-assembled nanoprobe.



(A)



(B)

Figure S10. (A) Possible reaction mechanism of TPE-3 with H_2S; (B) Mass spectra of TPE-3 upon reaction with H_2S. The signals at m/z 531.8 are $[(TPE\text{-}2)+K]^+$, and the signals at m/z 563.1 are $[(TPE\text{-}2)+K+MeOH]^+$.

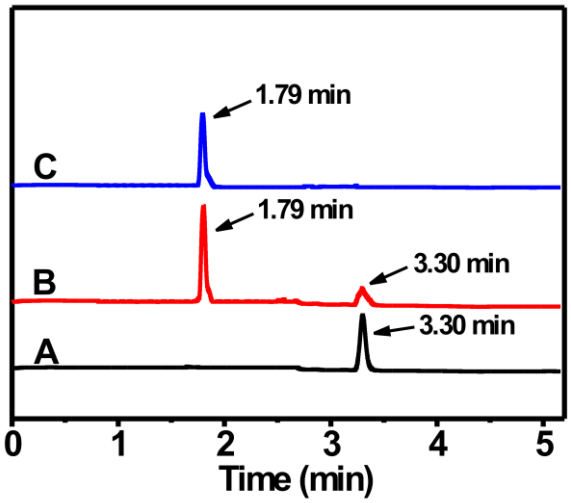


Figure S11. Typical HPLC chromatogram for the TPE-3 (A), TPE-3 incubated with H_2S for 30 min (B) and TPE-2 (C). Peaks in the chromatograms were detected by monitoring the absorption at both 365 nm and 450 nm. The mobile phase was 50/50 methanol/acetonitrile at a flow rate of 1.0 mL/min.

TPE-3 and the reaction product (TPE-2) give rise to a peak at 3.30 and 1.79 min in HPLC chromatogram, respectively. For the TPE-3, upon treatment with H_2S, the peak intensity at 3.30 min decreases and a new strong peak emerges at 1.79 min which well matches that for TPE-2.

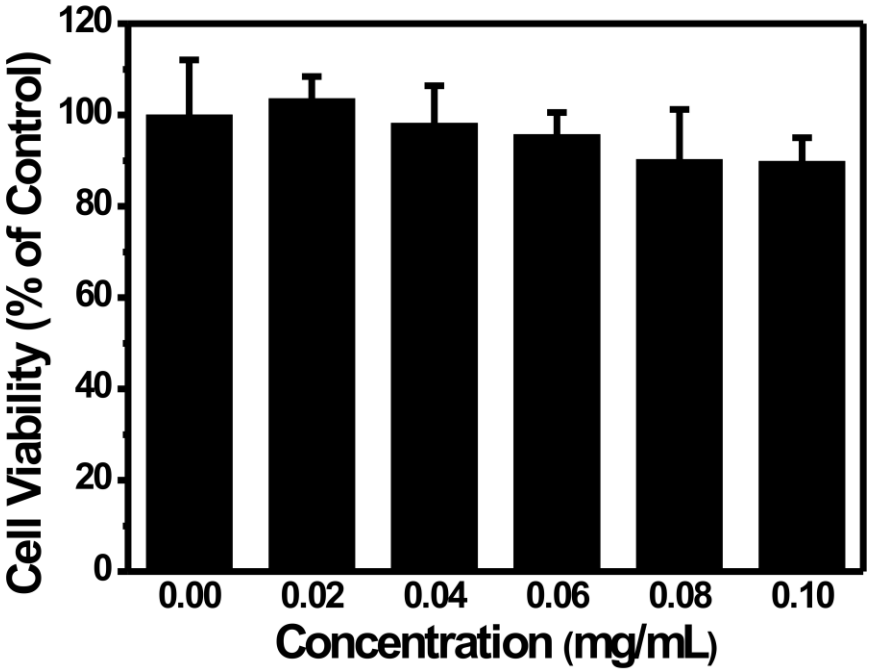


Figure S12. Viability for HeLa cells treated with the nanoprobe of varied concentrations for 24 h.