Supporting Information

A susceptible multifunctional fluorescent probe based on levulinic acid for practical detection of SO_2

Shijun Chen‡, Lin Wang‡, Shunchao Zhou, Xie He, Yuanyuan Wu, Shicong Hou* and Xiaodong Ma*
College of Science, China Agricultural University, Beijing 100193, P.R. China
‡These authors contributed equally.
Corresponding author: * E-mail: houshc@cau.edu.cn
E-mail: dongxm@cau.edu.cn

Table of Contents

1. Supplementary methods

1.1 General procedure for fluorescence detection

UV−Vis absorption spectra were recorded using a PerkinElmer Lambda 650S UV/Vis spectrometer. Fluorescence spectra were recorded using a PerkinElmer LS55 fluorescence spectrometer. ^{1}H NMR and ^{13}C NMR spectra were acquired using a Bruker AvanceAVII-500 MHz spectrometer. Probe stock solution was prepared at the concentration of 1.0×10^{-3} M in DMSO and then diluted to 1.0×10^{-5} M for titration experiments. UV-vis and fluorescence titration experiments were operated in PBS/DMSO=4:1 (10 mM pH 7.4), (λ_{ex} = 415 nm, excitation slit = 5 nm, emission slit = 5 nm).

1.2 Determination of detecting limits

The detecting limits (DL) were calculated according to Eq.

$$DL = 3\sigma/k$$

$$DL = (3 \times 0.0342/5.0625) \times 10^{-6} = 2.0 \times 10^{-8}\ M$$

Where σ is the standard derivation of blank solution and k is the slope of calibration curve.

1.3 Cytotoxicity test

HeLa cells were planted in 96-well plates and incubated for 24 h. The culture medium was removed after cell adherence, then the culture medium was washed with PBS for three times, and the probe with different concentrations was added to the culture medium. After incubation for 24 h, 100 μL MTT was added to each well and then incubated for 4 h. After the medium was discarded and 110 μL DMSO was added, the cell survival rate was calculated by measuring the absorbance at 570 nm after shaking for 10 min.

1.4 Determination of the fluorescence quantum yield

In our system, the fluorescence quantum yields of SO-2 were determined to be $\Phi = 0.37$ in PBS/DMSO=4:1 (10 mM pH 7.4) at 25℃, using quinine sulfate ($\Phi_f = 0.58$ in 1N H_2SO_4) as standard. The quantum yield was calculated using the following equation:

$$\Phi_x = \Phi_s(A_sF_x / A_xF_s)$$

where, Ax and As are the absorbance of the sample and the reference, respectively, at the same excitation wavelength, Fx and Fs are the corresponding relative integrated fluorescence intensities. Absorbance of sample and reference at their respective excitation wavelengths was controlled to be lower than 0.05.

2. Study on spectral properties

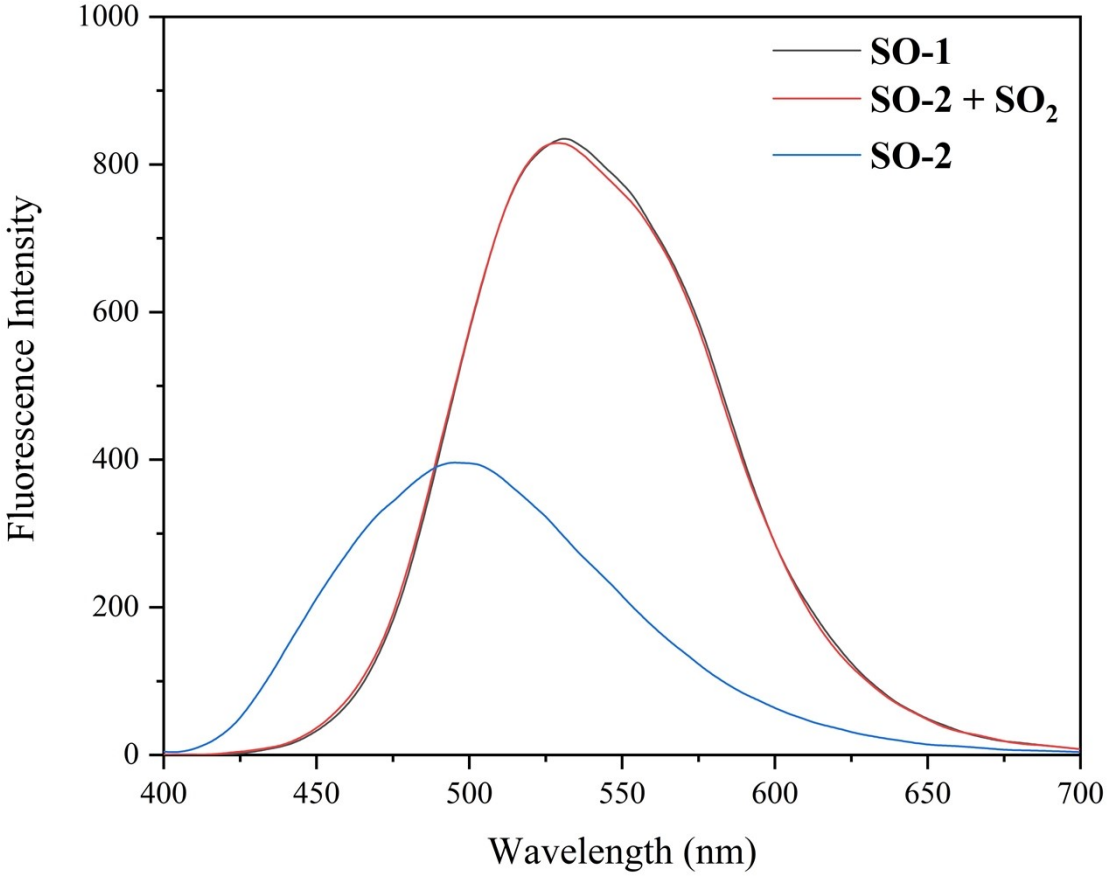

Fig. S1. Fluorescence spectrum of probe SO-2 (10 μM) and fluorophore SO-1 with and without SO_2 in the PBS/DMSO mixtures (4:1, v/v, 10 mM, pH = 7.4, λex = 417 nm).

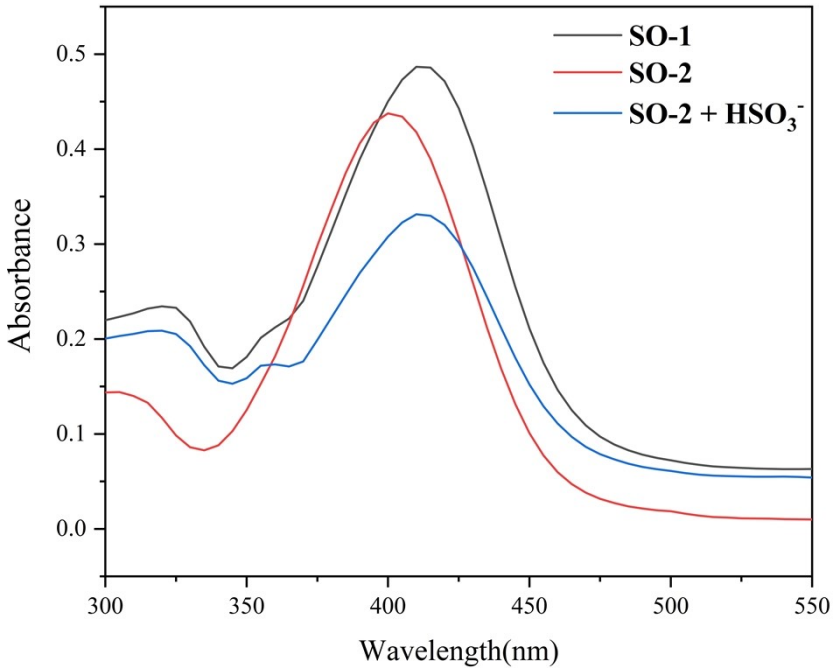

Fig. S2. Ultraviolet spectrum of probe **SO-2** (10 μM) and fluorophore SO-1 with and without SO_2 in the PBS/DMSO mixtures (4:1, v/v, 10 mM, pH = 7.4).

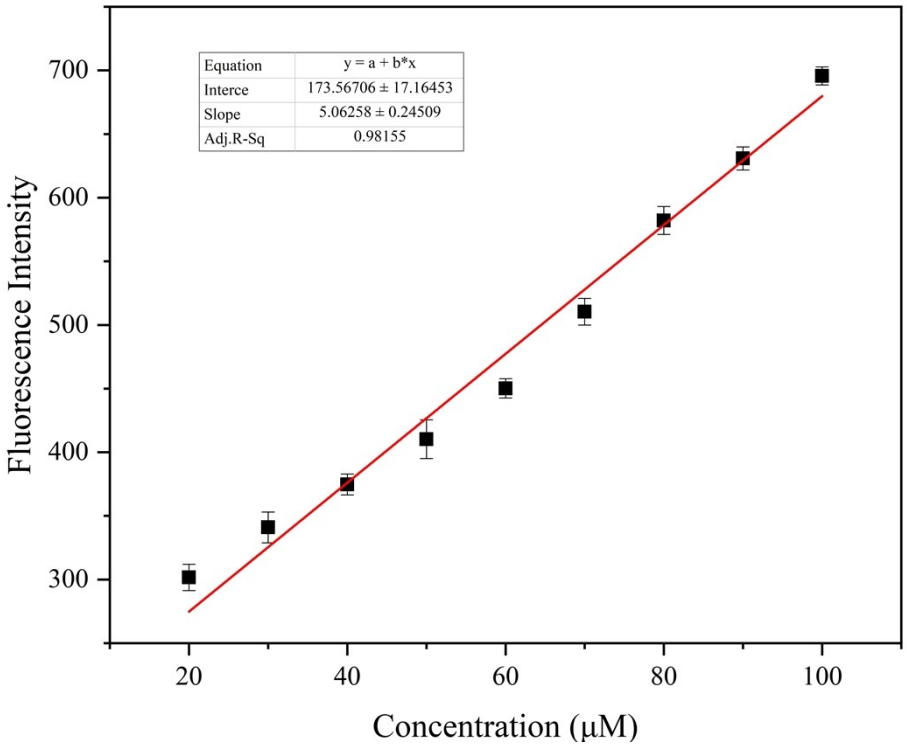

Fig. S4. The sensitivity evaluation by fitting the fluorescence intensity with the concentration of SO_2.

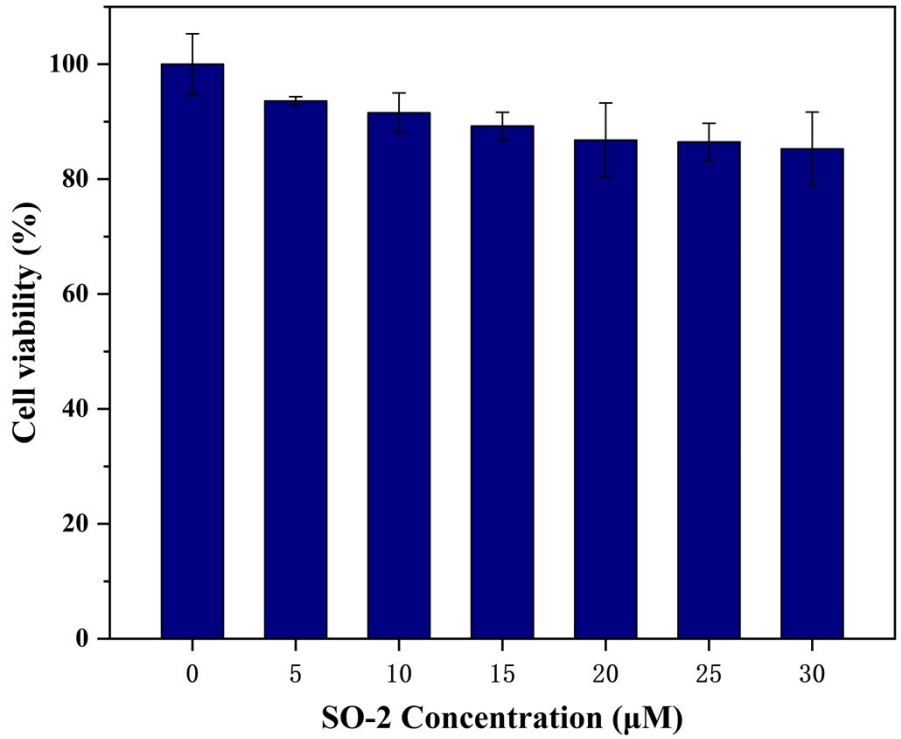

Fig. S5. MTT assay for the survival rate of HeLa cells treated with various concentrations of **SO-2** (from 0 to 30 μM) for 24 h. Error bars represent the standard deviation (n = 3).

3. ^{1}H NMR, and ^{13}C NMR analyses

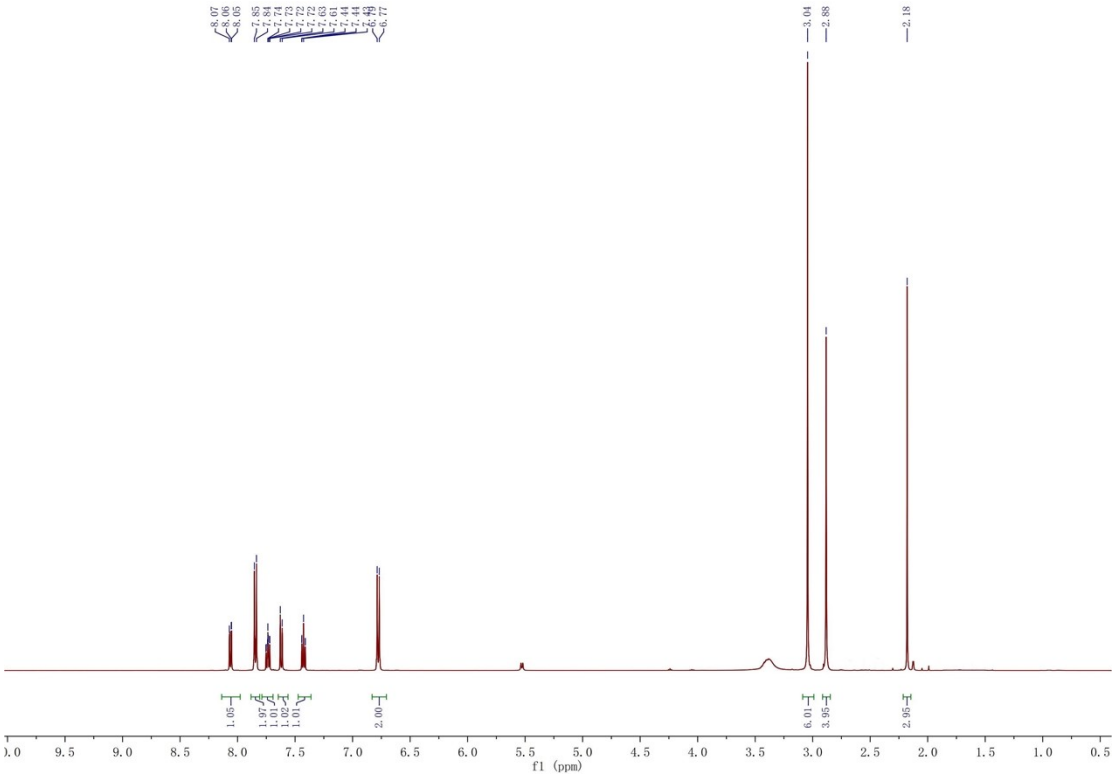

Fig. S6. ^{1}H NMR spectrum of **SO-2** in DMSO-d_6.

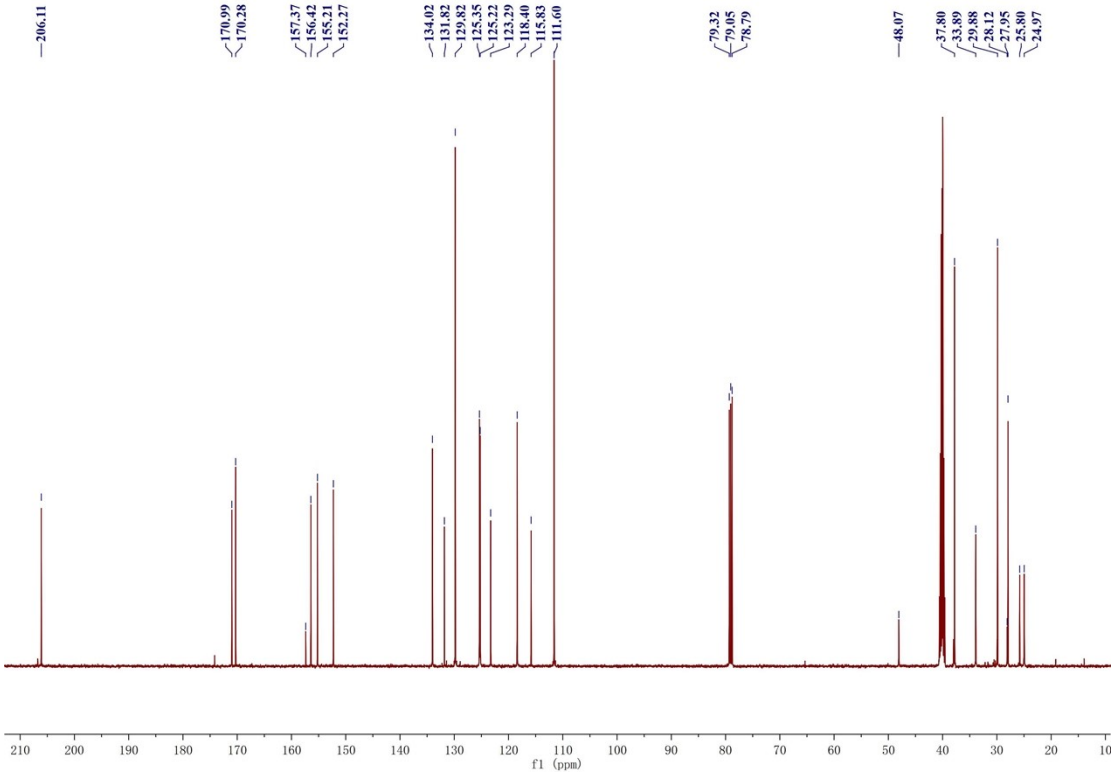

Fig. S7. ^{13}C NMR spectrum of **SO-2** in DMSO-d_6.

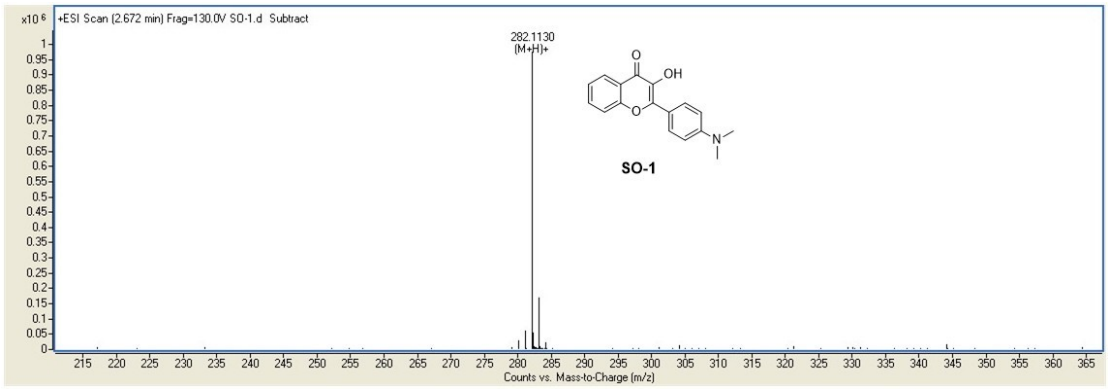

Fig. S8. High resolution mass spectrometry of **SO-2**.

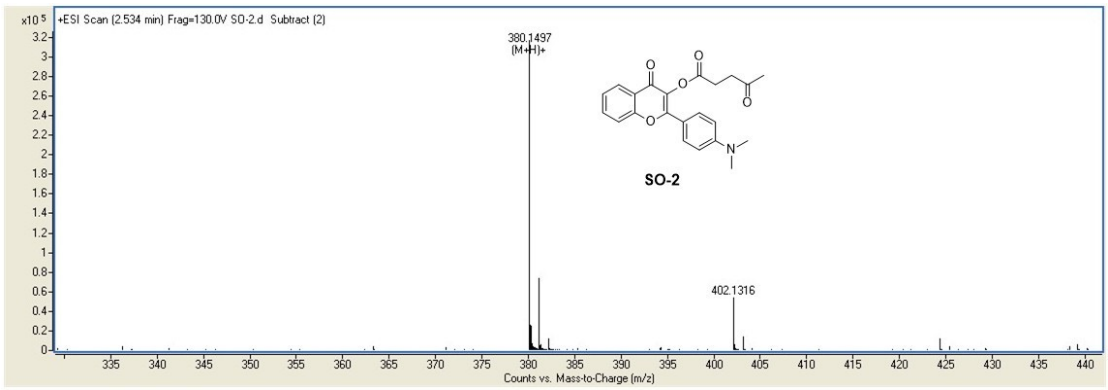

Fig. S9. High resolution mass spectrometry of **SO-2**.