

# Supporting Information

## An off-on fluorescent probe based on N, S-GQDs/CoOOH nanocomplexes for in vivo analysis of ascorbic acid

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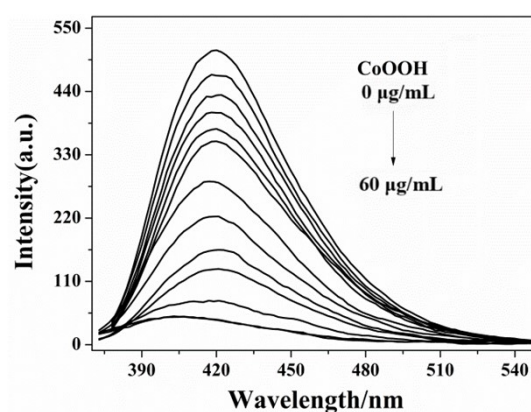
**Table S1** Comparison of different methods for the determination of AA.

**Table S2** Fluorescence lifetime of N, S-GQDs, N, S-GQDs/CoOOH, and N, S-GQDs/CoOOH + AA.

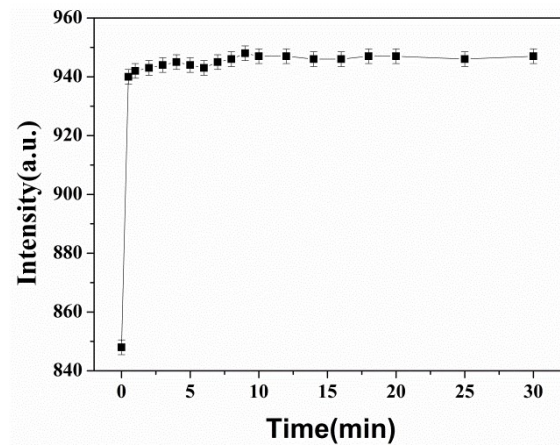
## Materials and Instruments

All starting chemicals and reagents were obtained from commercial suppliers and were used without additional purification. Citric acid, cysteine, sodium hypochlorite, sodium hydroxide (NaOH) were got by Aladdin (Shanghai, China). Cobalt chloride was obtained by Bodhi Chemical Co. (Tianjin, China). 1640 medium (Gibco), penicillin-streptomycin, dimethyl sulfoxide (DMSO, AR) were purchased from Solarbio Technology Co. (Beijing, China). Phytase were got by Yuanye (Shanghai, China). Fetal bovine serum was purchased from Thermo Fisher Scientific, and paraformaldehyde fixative (4%) was obtained through Feiyang Biologicals (Xi'an, China). Human breast cancer cells (MCF-7) were obtained from Shanghai Institutes for Biological Sciences, Chinese Academy of Sciences. Experimental zebrafish were acquired from the National Zebrafish Resource Center. The high-purity water was obtained by the Milli-Q water system (Millipore, Billerica, MA, USA).

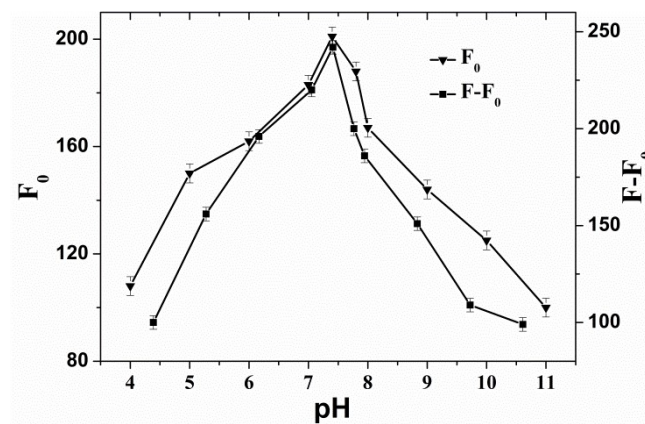
The Fluorescence and UV-Vis absorption spectra were performed on an F-4500 fluorescence spectrophotometer (Hitachi, Japan) and UV-2600 spectrophotometer (Shimadzu, Japan), respectively. The fluorescence decay curves were recorded with FLS 92 (Edinburgh, England) fluorescence spectrometer. The morphology of N, S-GQDs/CoOOH was characterized by transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) on JEM-2100F microscopy. The X-ray photoelectron spectroscopy (XPS) spectra were taken on DMAX UI11MA1V. Fourier transform infrared spectra (FT-IR) was conducted on 6700 FT-IR Microscope (Frontier IR/FIR, UK). The solution pH values were determined using a PHS-320 acidometer. Cell and zebrafish imaging experiments were conducted with FV1000 confocal microscope.



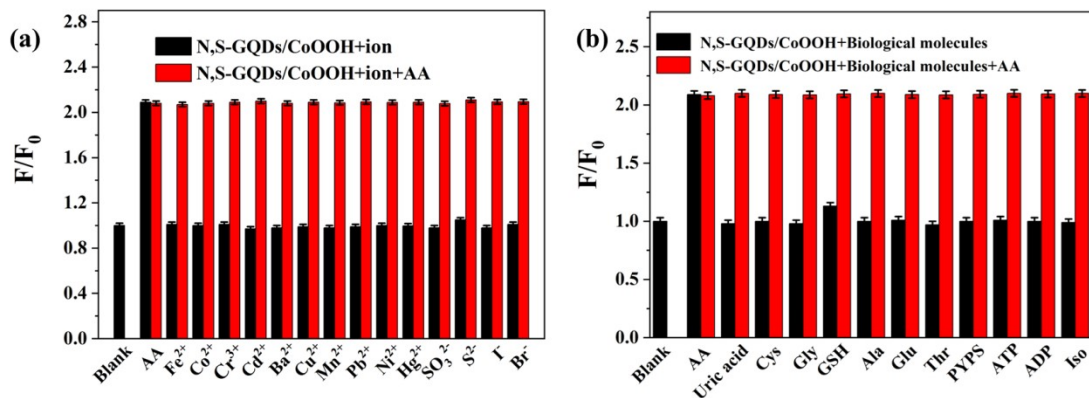
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**Figure S2.** Effect of reaction time on N, S-GQDs/CoOOH fluorescence recovered by AA.



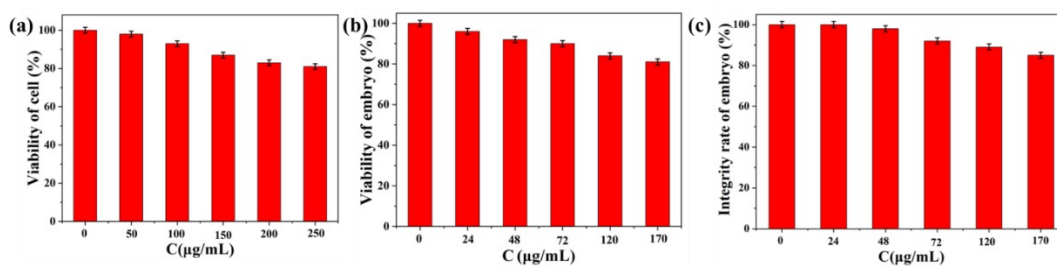
**Figure S3.** Effect of pH value on the fluorescence responses of N, S-GQDs/CoOOH with/without AA.



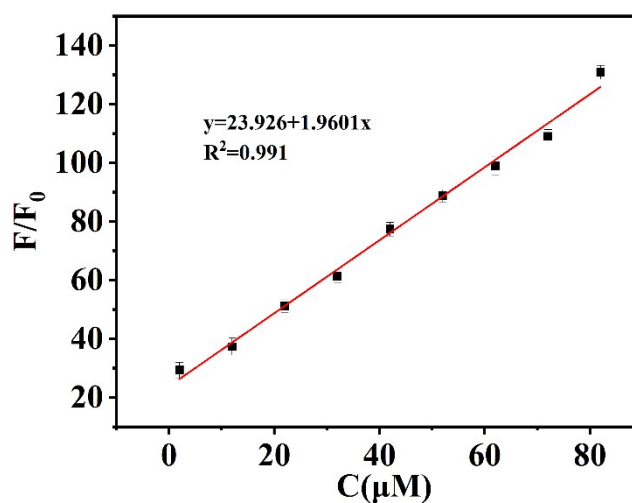
**Figure S4.** (a) Fluorescence response of different ions to N, S-GQDs/CoOOH and N, S-GQDs/CoOOH +AA. (b)

Fluorescent response of common biological disturbance substances to N, S-GQDs/CoOOH and N, S-

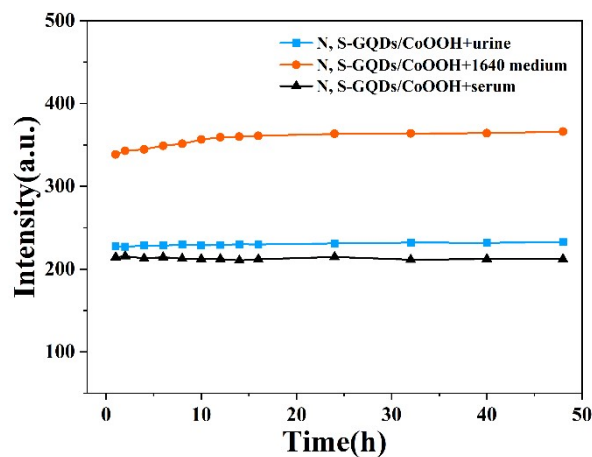
GQDs/CoOOH +AA.



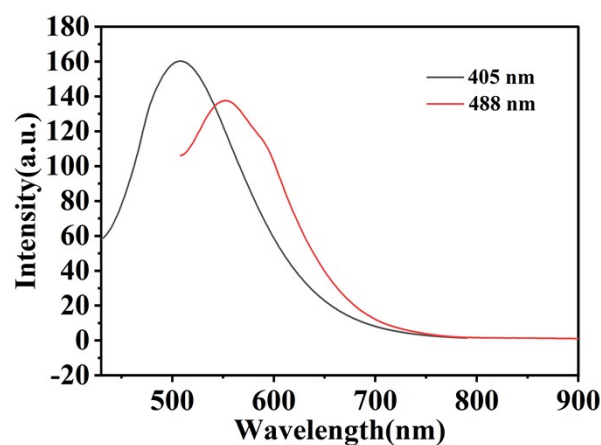
**Figure S5.** (a)Effect of different concentrations of N, S-GQDs/CoOOH on the viability of MCF-7 cells, (b)Effects of different concentrations of N, S-GQDs/CoOOH on zebrafish embryo survival, (c) Effects of different concentrations of N,S-GQDs/CoOOH on zebrafish teratogenesis.



**Figure S6.** Plot of F/F<sub>0</sub> versus the concentration of AA. F and F<sub>0</sub> are the fluorescence intensity of N, S-GQDs/CoOOH (0.7 mg/mL) with or without AA, respectively. The detection limit (LOD) was found to be 48.3 nM, as determined by the equation  $LOD = 3\sigma/k$ . ( $\sigma=0.032$ ,  $k = 1.9601$ ,  $\sigma$  is the standard deviation of background,  $k$  represents the slope of the equation)



**Figure S7.** The fluorescence stability of N, S-GQDs/CoOOH in urine, 1640 medium and serum solution.



**Figure S8.** The fluorescence spectrum of N, S-GQDs/CoOOH at various excitation wavelengths.

**Table S1** Comparison of different methods for the determination of AA.

Method	Materials	Linear range	Limit of Detection	Reference
Fluorimetry	D-CDs	1-400 $\mu\text{M}$	0.14 $\mu\text{M}$	1
Fluorimetry	Au@MnO <sub>2</sub> NPs	0.5-17.5 $\mu\text{M}$	0.47 $\mu\text{M}$	2
Fluorimetry	RhB@MOFs	10-100 $\mu\text{M}$	2.54 $\mu\text{M}$	3
Fluorimetry	N-CDs	0.1-100 $\mu\text{M}$	0.072 $\mu\text{M}$	4
Fluorimetry	AI-FIL	1-1000 $\mu\text{M}$	0.3 $\mu\text{M}$	5
Fluorimetry	Fe-HOF	0.5-8 $\mu\text{M}$	0.14 $\mu\text{M}$	6
Fluorimetry	Ti <sub>3</sub> C <sub>2</sub> QD	0-100 $\mu\text{M}$	0.19 $\mu\text{M}$	7
Electrochemistry	K-CKR	50-1620 $\mu\text{M}$	0.83 $\mu\text{M}$	8
Electrochemistry	Co <sub>2</sub> P	0.1-4.5 mM	12.15 $\mu\text{M}$	9
Titration	DCPI	0.002-0.0012 mg/mL	0.0020 mg/mL	10
spectrophotometry	Cr (VI)	100 $\mu\text{M}$ -100 mM	0.00154 mg/mL	11
Fluorimetry	N, S-GQDs/CoOOH	2-82 $\mu\text{M}$	0.048 $\mu\text{M}$	This work

**Table S2** Fluorescence lifetime of N, S-GQDs, N, S-GQDs/CoOOH, and N, S-GQDs/CoOOH + AA.

Samples	$\tau_1$ (ns)	$\tau_2$ (ns)	$B_1$ (%)	$B_2$ (%)	Average $\tau$ (ns)	$\chi^2$
N, S-GQDs	2.64	10.58	23.33	76.67	8.72	1.076
N, S-GQDs/CoOOH	0.23	4.18	64.68	35.32	1.62	1.120
N, S-GQDs/CoOOH + AA	0.17	3.73	15.31	84.69	3.18	1.196

## References

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