

Electronic Supplementary Material (ESI) for New Journal of Chemistry

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A facile intracellular fluorescent probe for hydrazine and its application

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Supplementary information

Materials. CF₃COOH, acenaphthenequinone and malononitrile reagents were purchased from Aladdin (China). Other chemicals and solvents were used as received unless specifically noted. **1** was synthesized and purified according to the reported literature.

Measurements

Absorption and emission spectra were collected by using a Shimadzu 1750 UV-visible spectrometer and a RF-5301 fluorescence spectrometer (Japan), respectively. Cell images were performed on Ziss LSM-510META confocal microscope (Germany). All experiments were performed in compliance with the relevant laws and institutional guidelines, and were approved by Northwest A&F University. Informed consent was obtained for all human subjects.

Sample Preparation and Titration. Stock solutions of hydrazine and relative chemicals were prepared with concentration of 1.0×10⁻² M. Stock solution of compound **1** (2×10⁻³ M) was prepared in CH₃CN and further diluted to 5.0×10⁻⁶ M for titration experiments.

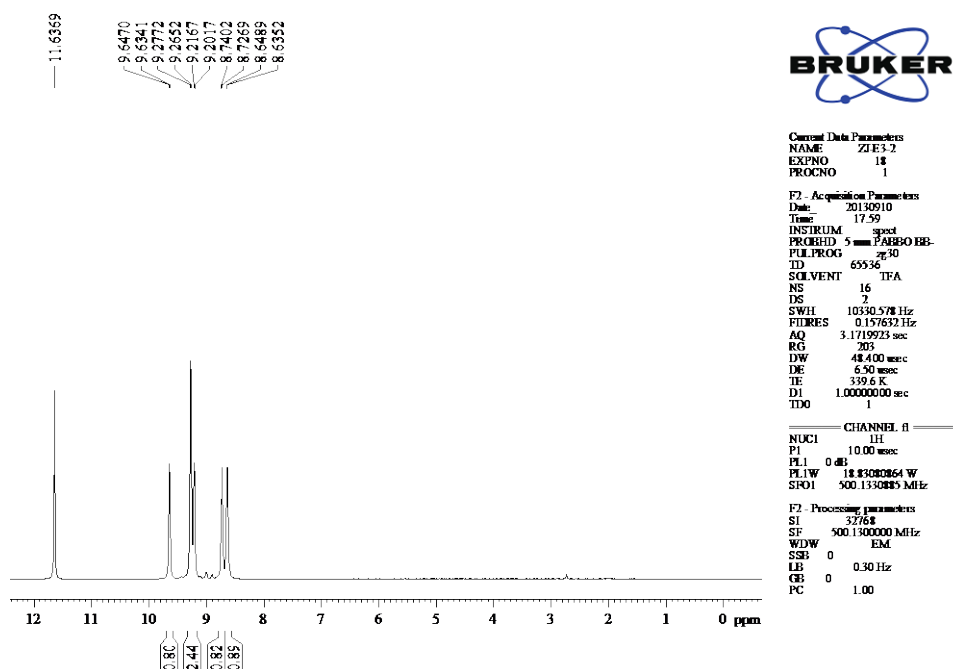


Fig. S1 ¹H NMR spectra of **1** in TFA

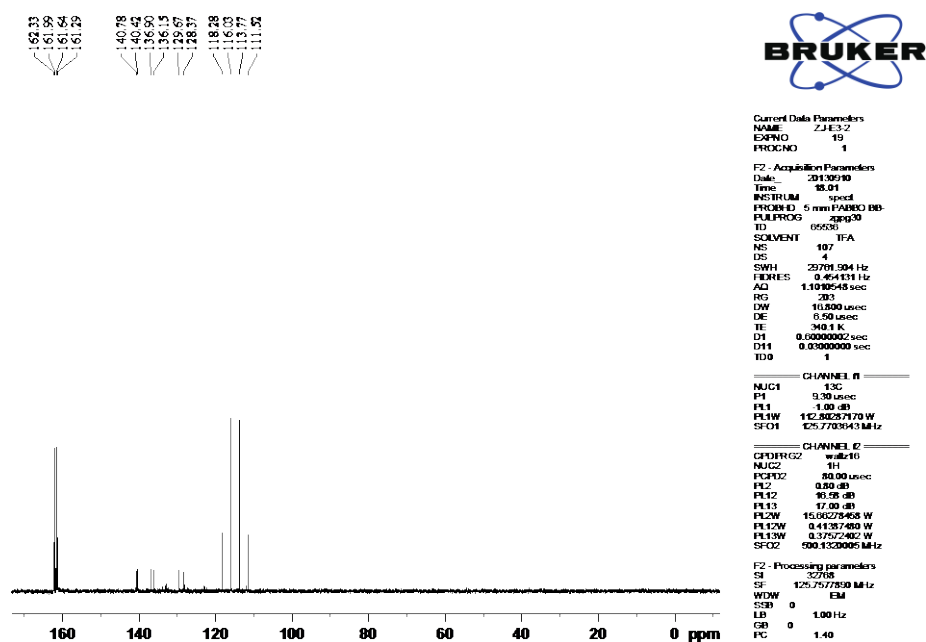


Fig. S2 ¹³C spectra of **1** in TFA.

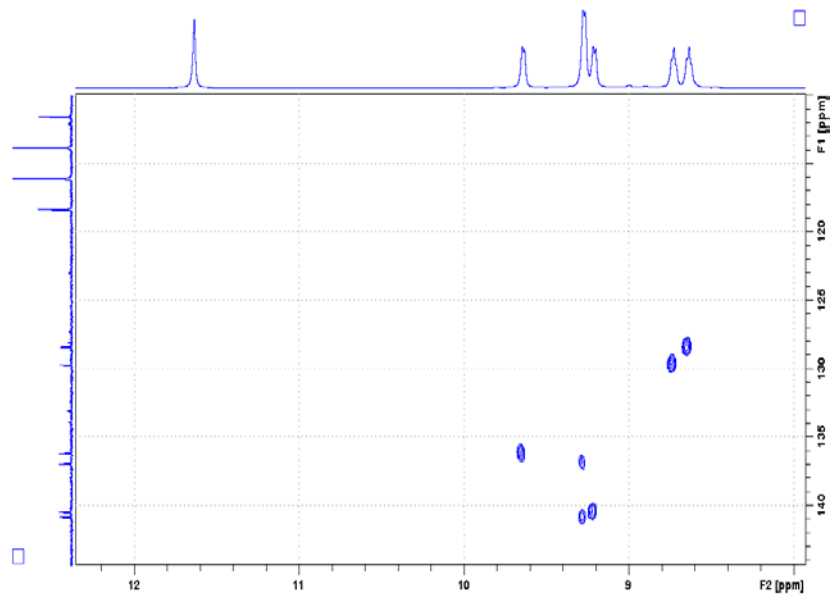


Fig. S3 HSQCGP spectra of **1** in TFA.

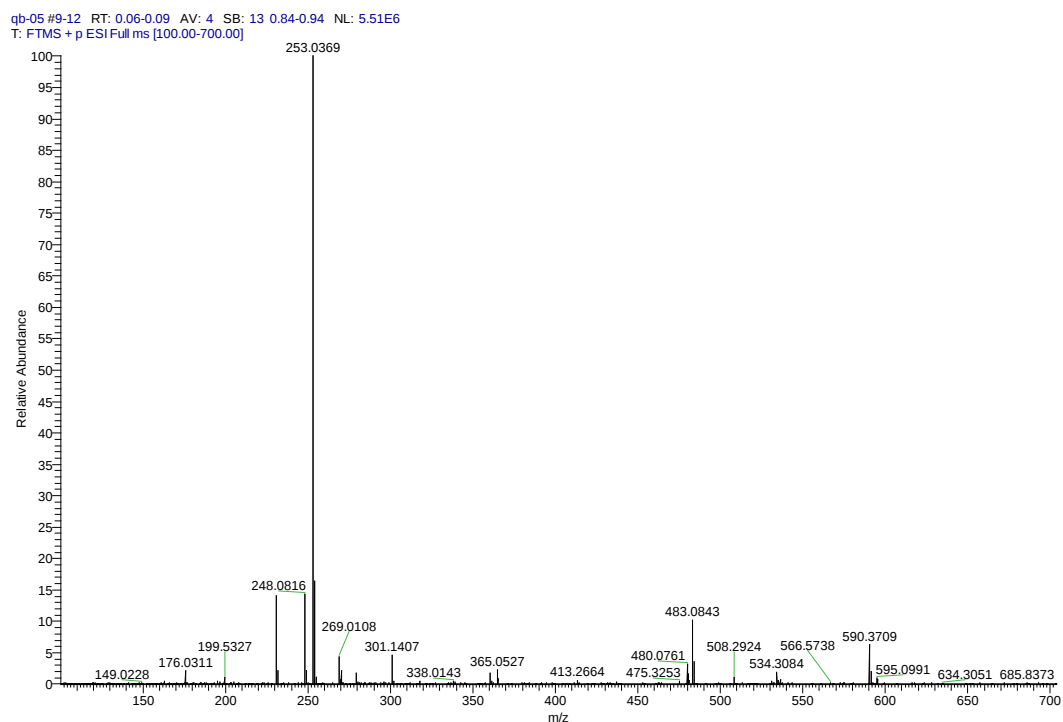


Fig. S4 EI-MS spectrum of compound **1**.

PROTON DMSO {D:\2013} ZHL 18

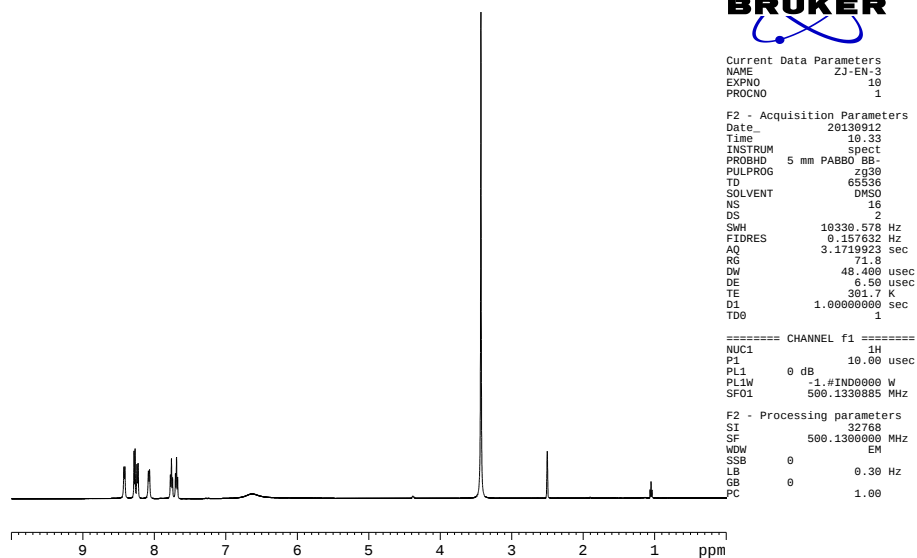
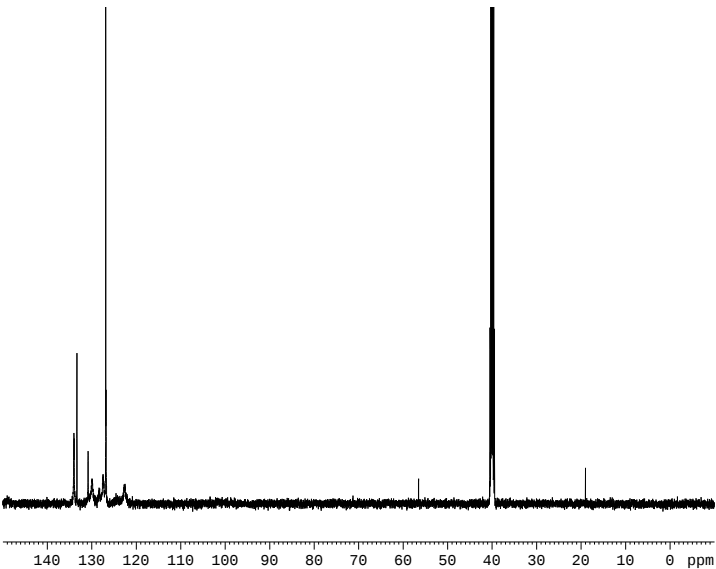


Fig. S5 ^1H NMR spectra of **2** in $\text{DMSO}-d_6$.

C13CPD DMSO {D:\2013} ZHL 18



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EXPNO 11
PROCNO 1

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DE 6.50 usec
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D11 0.03000000 sec
TD0 1

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PL1 -1.00 dB
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F2 - Processing parameters
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GB 0
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Fig. S6 ¹³C spectra of 2 in DMSO-*d*₆.

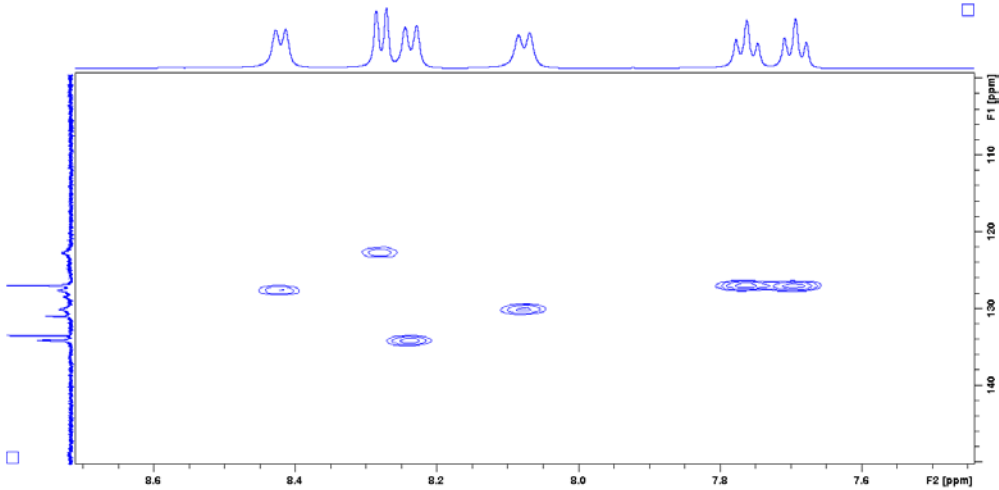


Fig. S7 HSQCGP spectra of 2 in DMSO-*d*₆.

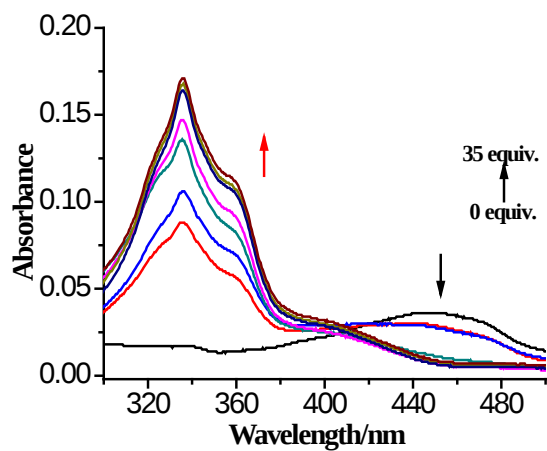


Fig. S8 UV-vis spectra of probe **1** (20 μM) in the presence of different concentrations of hydrazine in a mixture of PBS/CH₃CN solution (9/1, V/V, pH 7.4). Each spectrum was recorded after 40 min of mixing. The arrows indicate the change of the absorption with the increasing of hydrazine.

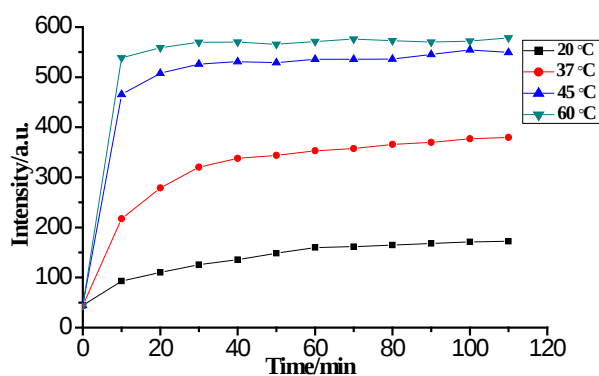


Fig. S9 The probe **1** (5 μM) and 120 μM N₂H₄ were equilibrated in different temperature, the fluorescence intensity was acquired in 0.2 M PBS/CH₃CN (9/1, V/V, pH 7.4) with emission at 565 nm.

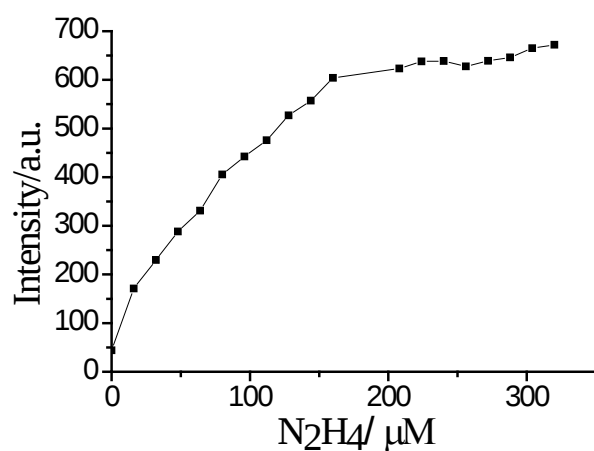


Fig. S10 Emission spectra of probe **1** (5 μM) upon addition of increasing concentrations of hydrazine (0 - 64 equiv.) in PBS/CH₃CN (9/1, V/V, pH 7.4).

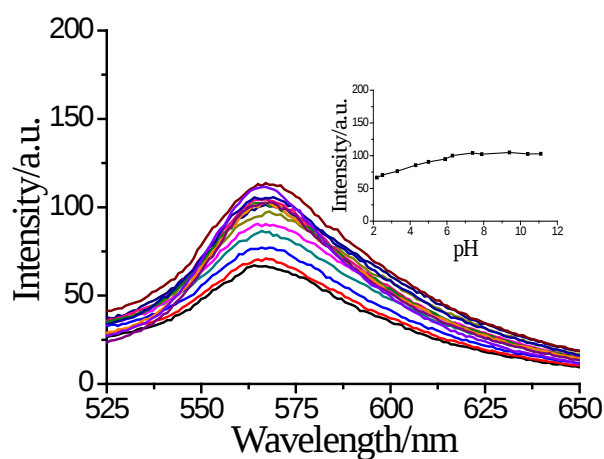


Fig. S11 The effect of pH value on the fluorescence intensity of probe **1** (30 μM) in H₂O/CH₃CN (9/1, V/V). pH values: 2.2, 2.5, 3.3, 4.3, 5.0, 5.9, 6.3, 6.7, 7.4, 7.9, 9.4, 10.4, 11.1.

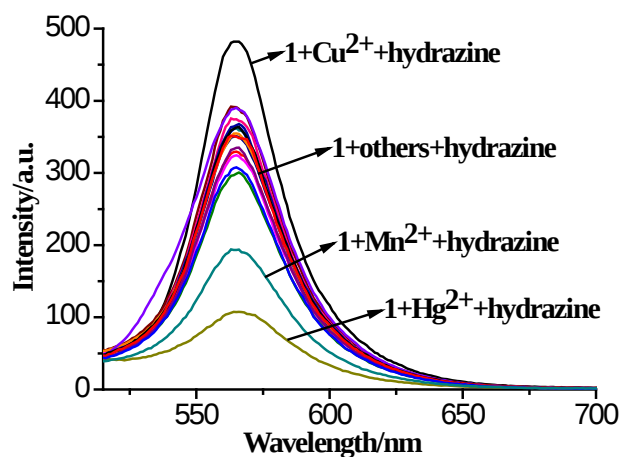


Fig. S12 Fluorescence spectra of **1** in the presence of representative metal ions, anions and hydrazine. Probe **1** = 5 μM , hydrazine = 96 μM , 120 μM for Cu^{2+} , Ba^{2+} , Mg^{2+} , Cd^{2+} , Pb^{2+} , Hg^{2+} , Zn^{2+} , Fe^{3+} , SCN^- , I^- , HCO_3^- , Br^- , Na^+ , SO_3^{2-} , SO_4^{2-} , Cl^- , HPO_4^{2-} , Mn^{2+} . In a mixture of PBS/ CH_3CN (9/1, V/V, pH 7.4), measured after 40 min of mixing.

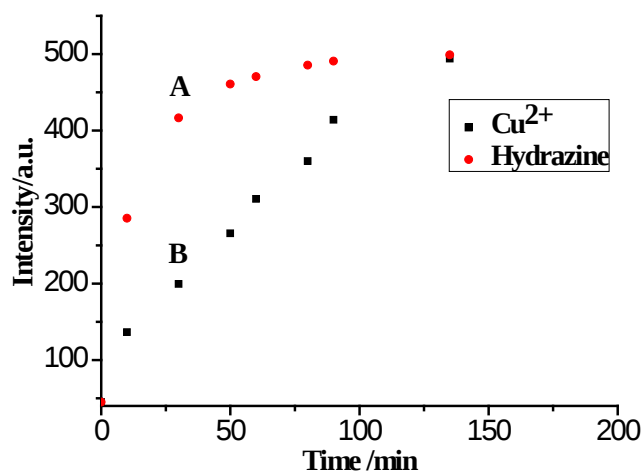


Fig. S13 Response of **1** to addition of (A) 96 μM hydrazine and (B) 120 μM Cu^{2+} , respectively.

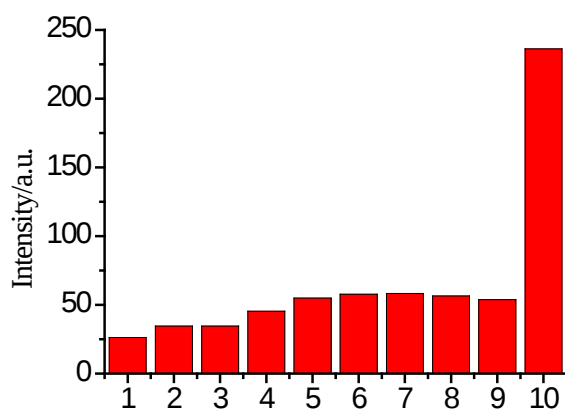


Fig. S14 Fluorescence responses of probe **1** (5 μM) to various amine-containing compounds (120 μM for ethanediamine, triethylamine, ammonia, thiourea, NH_4^+ , Cys, Lys, Glu) and hydrazine (120 μM) at 565 nm. In a mixture of PBS/ CH_3CN (9/1, V/V, pH 7.4), measured after 40 min of mixing. 1. blank, 2. ethanediamine, 3. triethylamine, 4. ammonia, 5. thiourea, 6. NH_4^+ , 7. Cys, 8. Lys, 9. Glu and 10. hydrazine.

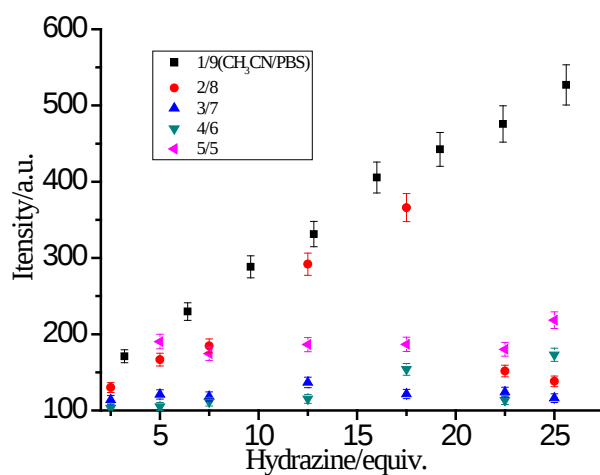


Fig. S15 The effect of different ratio of CH_3CN to PBS on the response of the probe to hydrazine.