Electronic supplementary Information

Quantification and visual inspection of adipic dihydrazide in textile using a xanthonium-based ratiometric fluorescent probe

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CONTENT

1. Structural characterizations of probe SH-Py

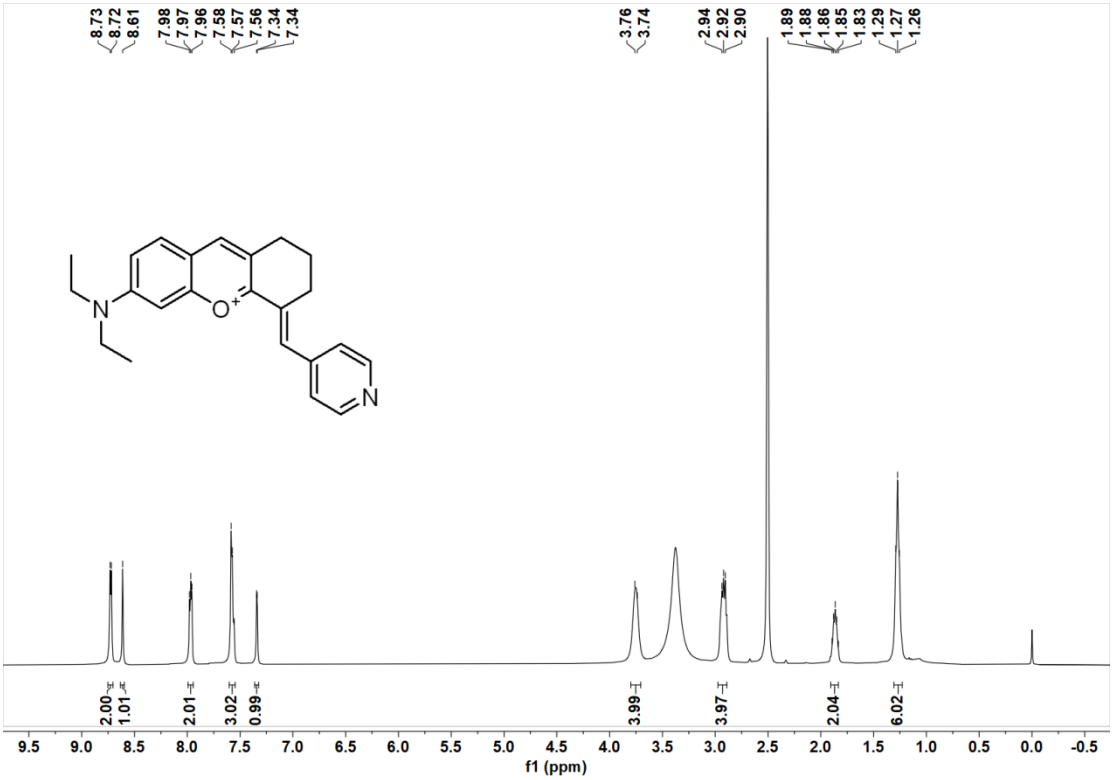

Fig. S1 ^{1}H NMR (400 MHz) spectrum of **SH-Py** in DMSO-d_6.

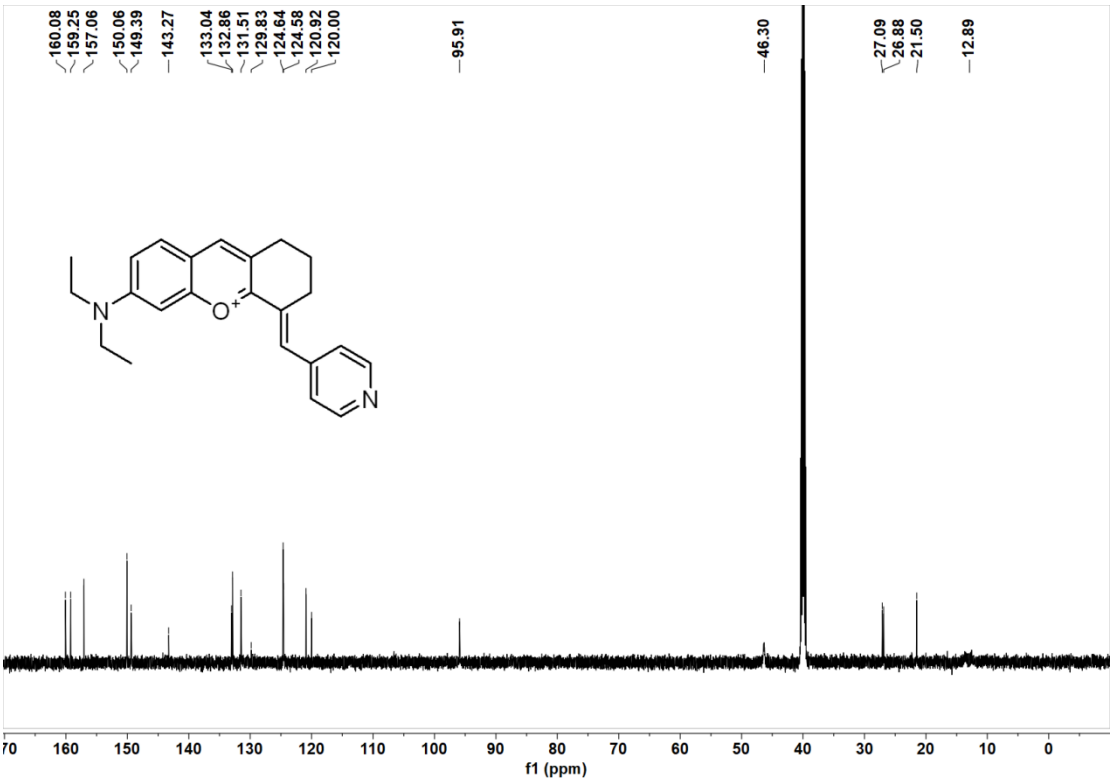

Fig. S2 ^{13}C NMR (100 MHz) spectrum of **SH-Py** in DMSO-d_6.

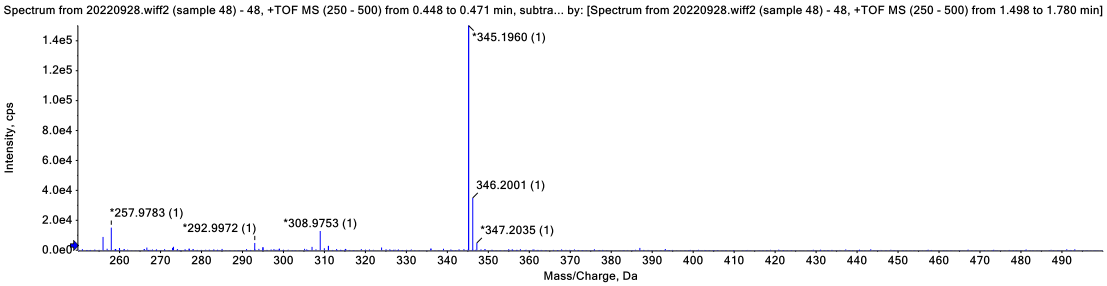

Fig. S3 High resolution mass spectrometry of **SH-Py**.

2. Color changes of SH-Py upon the addition of ADH in different solutions

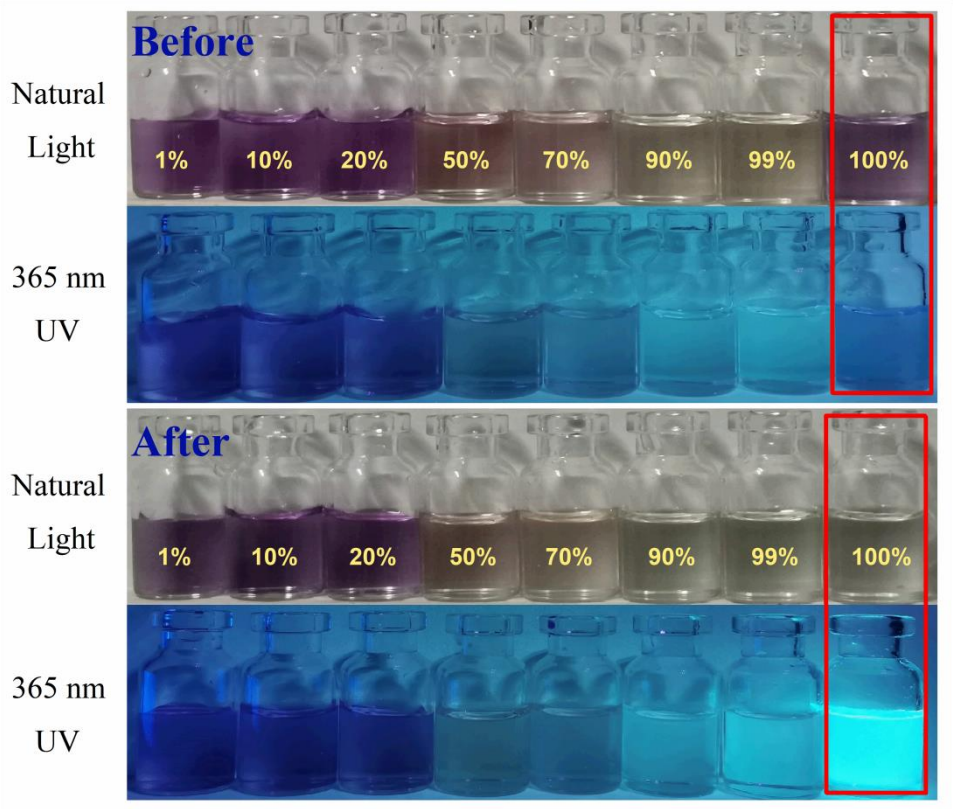

Fig. S4 The color changes of **SH-Py** (10 μM) in solutions with increasing volume percentage (1%, 10%, 20%, 50%, 70%, 90%, 99%, 100%) of DMSO before and after the addition of ADH (500 μM).

3. Spectroscopic properties of SH-Py

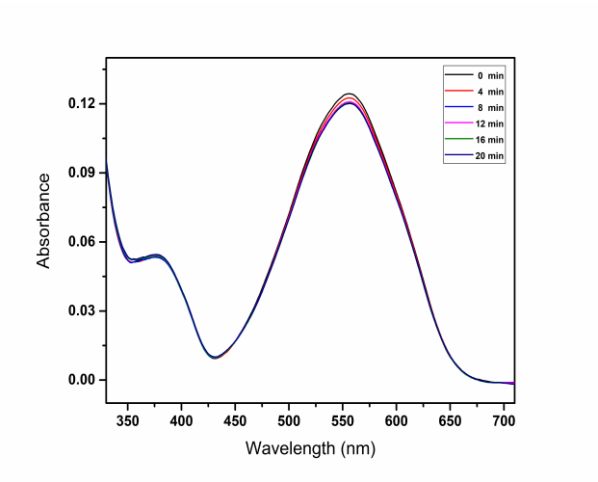

Fig. S5 Time-dependent UV-vis absorption spectra of probe **SH-Py**.

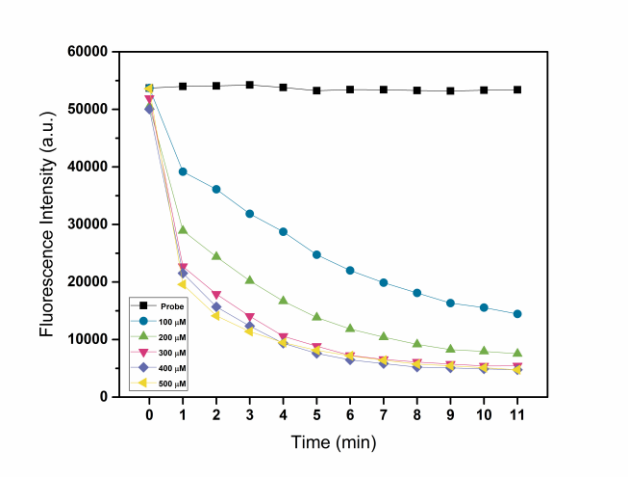

Fig. S6 Time-dependent fluorescence intensities at 680 nm (λ_{ex} = 560 nm, slit width (ex/em) = 20/20 nm) before and after the response of **SH-Py** (10 μM) to different concentrations (100 μM, 200 μM, 300 μM, 400 μM, 500 μM) of ADH.

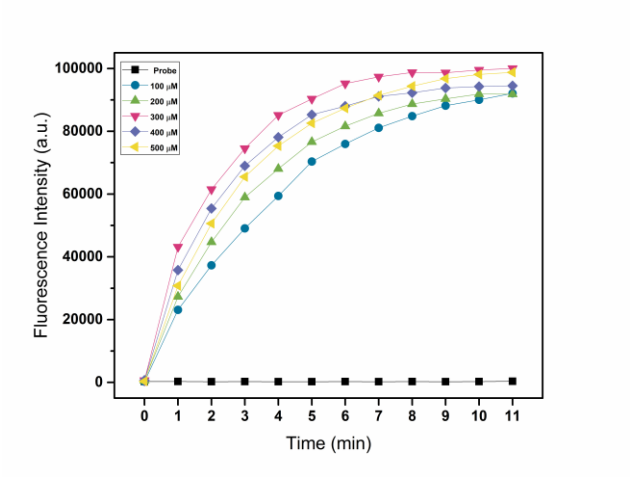

Fig. S7 Time-dependent fluorescence intensities at 463 nm (λ_{ex} = 560 nm, slit width (ex/em) = 20/20 nm) before and after the response of **SH-Py** (10 μM) to different concentrations (100 μM, 200 μM, 300 μM, 400 μM, 500 μM) of ADH.

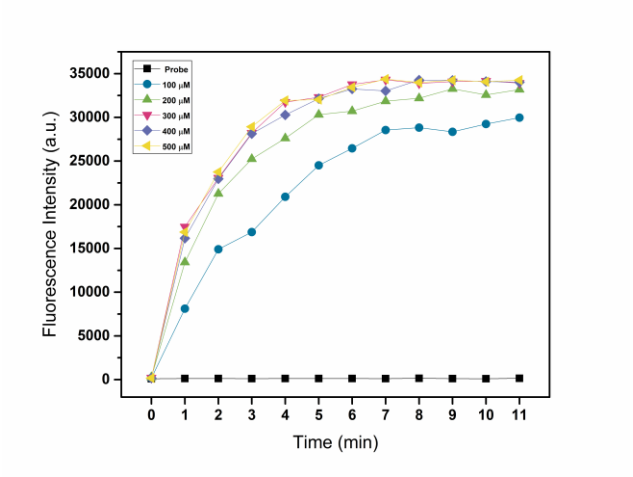

Fig. S8 Time-dependent fluorescence intensities at 460 nm (λ_{ex} = 380 nm, slit width (ex/em) = 3/3 nm) before and after the response of the probe **SH-Py** (10 μM) to different concentrations (100 μM, 200 μM, 300 μM, 400 μM, 500 μM) of ADH.

4. Verification of sensing mechanism

To the solution of probe **SH-Py** (0.89 mg, 1 mM) in DMSO (2 mL) was added the solution of ADH in DMSO (20 μL) and the final concentration of ADH was 20 mM. Then, the mixture was subjected to HR-MS analysis after incubation at room temperature for 15 min.

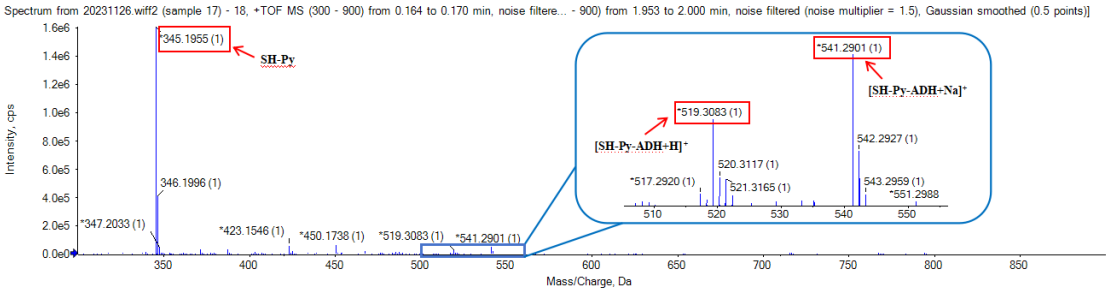

Fig. S9 High resolution mass spectrometry of **SH-Py** after the addition of excess ADH.

Preparation of addition product SH-Py-ADH

ADH (1044 mg, 6 mmol) was dissolved in DMSO (7 mL) with the aid of ultrasound treatment, then, **SH-Py** (134 mg, 0.3 mmol) was added in four portions. After the mixture was stirred at 30℃ for 30 min, ethyl acetate (30 mL) and brine (7 mL) were added and the mixture was stirred at room temperature for 5 min. The organic layer separated was further washed with water, dried with sodium sulfate, and concentrated under reduced pressure to yield yellow crude product (110 mg). Yellow brown solid (35 mg, 22.5%) was finally obtained by chromatograph on aluminium oxide (neutral, 100 - 200 mesh) with ethyl acetate/ methanol (30/1, v/v) as the eluent. ^{1}H NMR (400 MHz, DMSO-d_6): δ (ppm) 9.16 (d, J = 27.6 Hz, 1H), 8.47 (d, J = 4.6 Hz, 2H), 7.42 – 7.36 (m, 2H), 6.80 (d, J = 8.3 Hz, 1H), 6.19 (s, 2H), 6.10 (s, 1H), 5.38 (s, 1H), 5.20 (s, 1H), 3.31 – 3.18 (m, 4H), 2.31 (d, J = 11.0 Hz, 4H), 1.99 (d, J = 1.9 Hz, 2H), 1.84 (d, J = 5.6 Hz, 2H), 1.59 (s, 2H), 1.40 (s, 2H), 1.30 – 1.13 (m, 4H), 1.07 (t, J = 5.8 Hz, 6H).

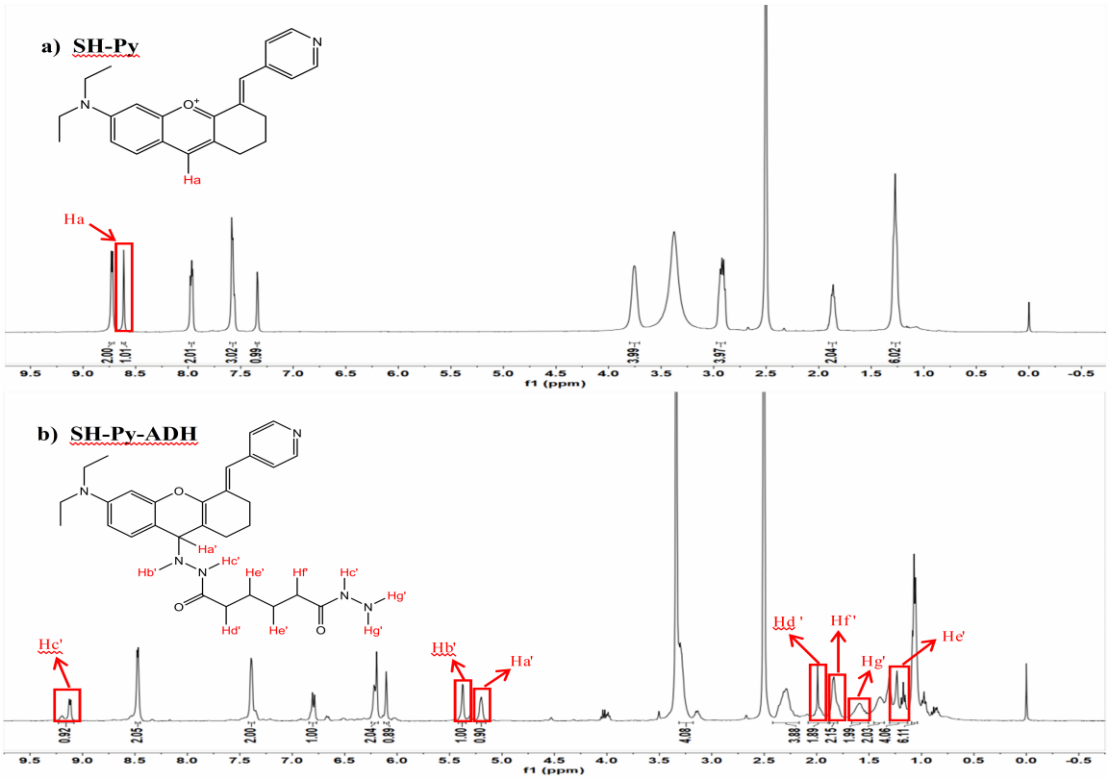

Fig. S10 ^{1}H-NMR spectrum of probe **SH-Py** before (a) and after (b) the reaction with ADH.

5. Comparison of experimental results of different methods for the determination of ADH in real textile samples

Table S1 Comparison of experimental results of different methods for the determination of ADH in real textile samples.

Method	Solvent	Analysis time (min)	Linearity range	Recovery (%)	RSD (%)	Reference
HPLC-MS/MS	Water	5	0.05-2 mg/L	85-100%	＜10%	[1]
Fluorescent probe	DMSO	10	0-300 μM	97.7-101.1%	＜1.1%	This work

Reference

[1] J. Tao, Z. Lin, H. Zhang, Z. Wu and H. Cao, *RSC Adv.*, 2018, **8**, 2915-2921.

6. AGREE assessment of the fluorescent method

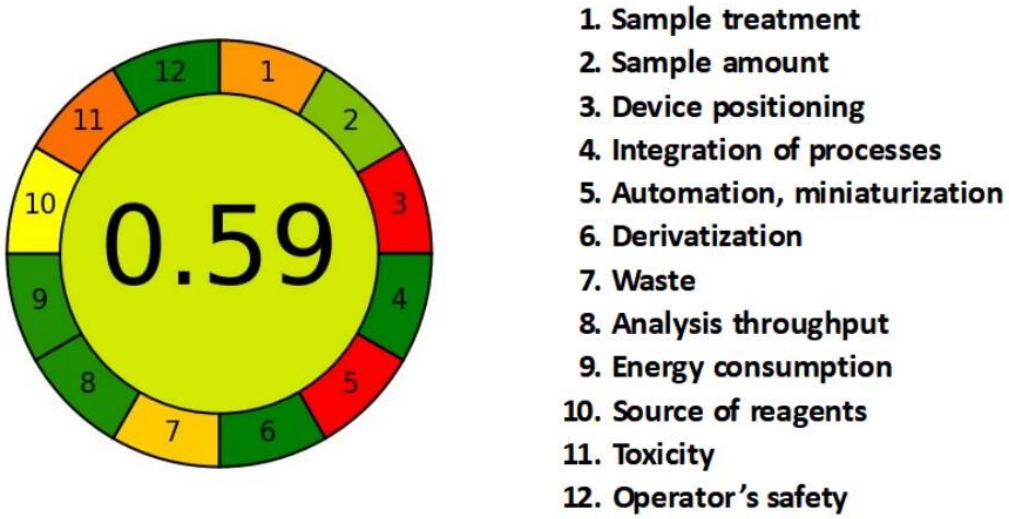

Fig. S11 AGREE assessment of the fluorescent method for the quantification of ADH in textile sample.

7. Determination of LOD for acetyl hydrazine

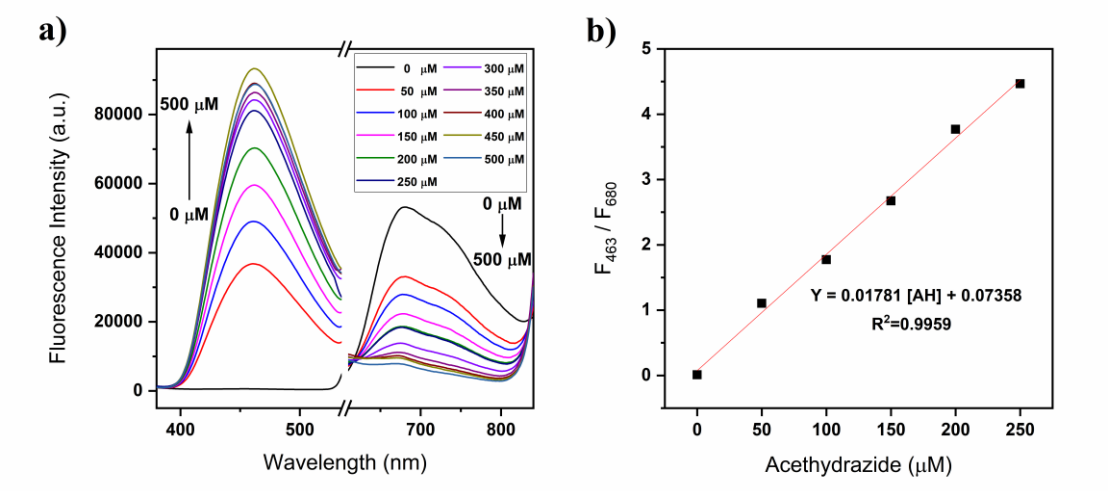

Fig. S12 (a) Changes of fluorescence spectra of **SH-Py** (10 μM) upon the addition of increasing concentrations of acetyl hydrazine (0-500 μM). (b) Plot of the linear relationship between F_{463}/F_{680} of **SH-Py** and the concentration of acetyl hydrazine (0-250 μM). λ_{ex} = 560 nm, slit width (ex/em) = 20/20 nm.

8. Verification of sensing mechanism of SH-Py towards acetyl hydrazine

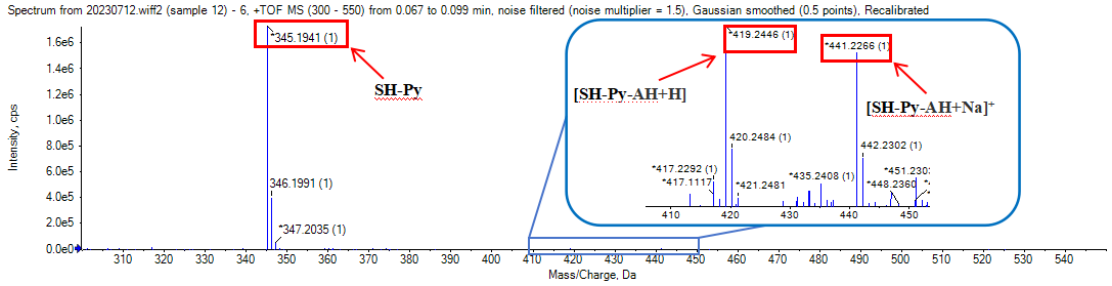

Fig. S13 High resolution mass spectrometry of **SH-Py** after the addition of excess acetyl hydrazine.

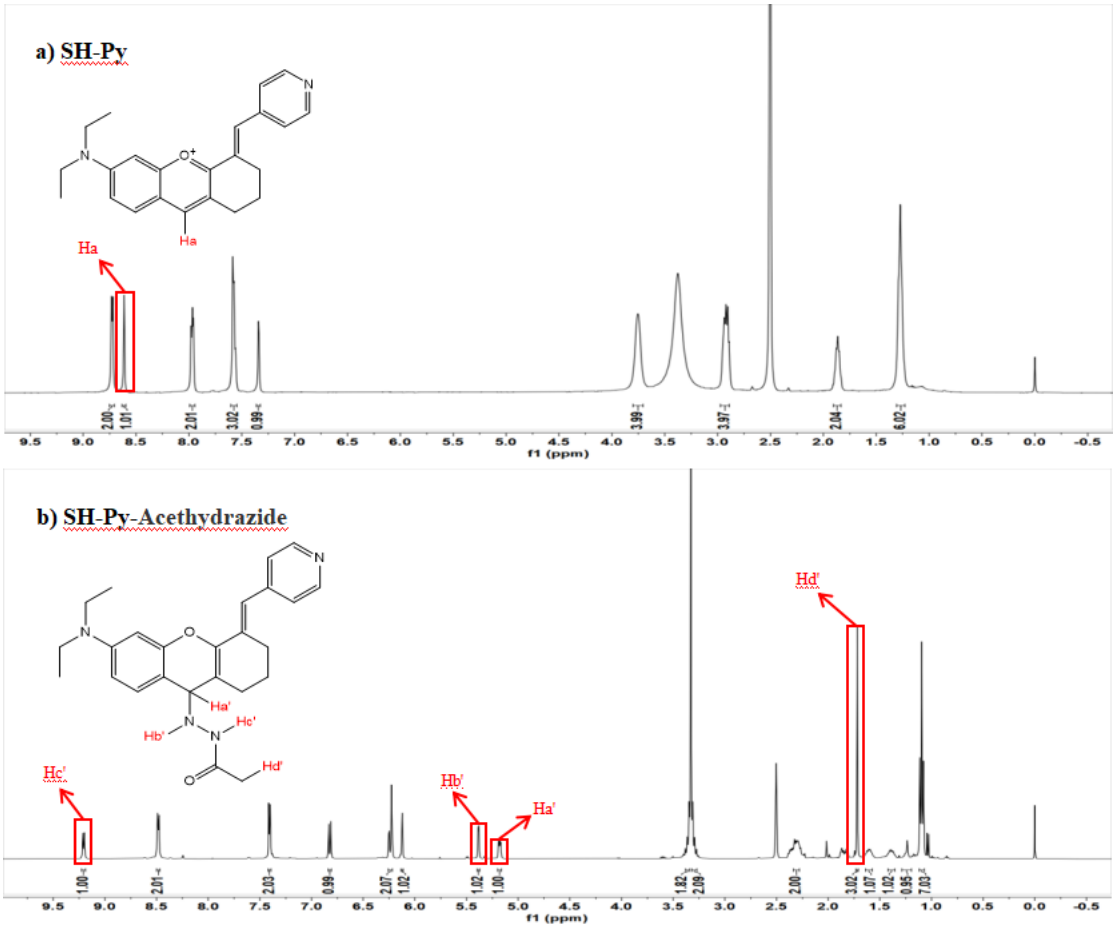

Fig. S14 ^{1}H-NMR spectrum of probe **SH-Py** before (a) and after (b) the reaction with acetyl hydrazine.