

## Electronic Supplementary Information

### **Reversible Detection of Hypochlorite Using Deprotonation-Protonation Strategy: A**

#### **Search for New Building Blocks**

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#### **Contents**

- S4 Synthetic scheme
- S6 Comparison table of ESIPT-AIE active probe **1** in present manuscript over other literature reports
- S8 UV-vis spectra of probe **1** in different fractions of H<sub>2</sub>O/DMSO mixture
- S8 Fluorescence spectra of probe **1** in different solvents
- S9 Fluorescence emission spectrum of probe **1** in DMSO
- S9 Fluorescence emission spectra of derivative **2** in different fractions of H<sub>2</sub>O/DMSO mixture
- S10 Fluorescence emission spectra of probe **1** in different fractions of H<sub>2</sub>O/DMSO mixture
- S10 Fluorescence emission spectra of probe **1** in solid state
- S11 FTIR Spectrum of probe **1** in solid state
- S11 FTIR Spectrum of probe **1** in DMSO
- S12 FTIR Spectrum of probe **1** in DMSO: H<sub>2</sub>O
- S12 <sup>1</sup>H NMR of probe **1** in DMSO-*d*<sub>6</sub> and DMSO-*d*<sub>6</sub>: D<sub>2</sub>O (2:8) mixture
- S13 <sup>13</sup>C NMR of probe **1** in DMSO-*d*<sub>6</sub>: D<sub>2</sub>O (2:8) mixture
- S13 Fluorescence emission spectra of probe **1** in different fractions of glycerol: DMSO
- S14 TEM image of probe **1** in H<sub>2</sub>O/DMSO (8/2, v/v) mixture

- S14 Fluorescence spectrum of probe **1** in PBS/DMSO (8/2, v/v) mixture
- S15 Detection limit of probe **1**
- S16 Absorption spectra of probe **1** in presence of NaOCl in water
- S16 Fluorescence spectra of probe **1** in presence of NaOCl in water
- S17 Fluorescence lifetime decay profiles of probe **1** in presence hypochlorite in PBS/DMSO (8/2, v/v)
- S17 The reversibility of fluorescence emission intensity at 510 nm by alternate addition of ClO<sup>-</sup> and H<sup>+</sup> to solution of **1** in PBS/DMSO (8/2, v/v)
- S18 Fluorescence spectra of probe **1** in PBS/DMSO (8/2, v/v) mixture at different pH
- S18 UV-vis spectra of probe **1** upon addition of different ROS and anions
- S19 <sup>1</sup>H-NMR titration of probe **1** in presence of sodium hypochlorite in D<sub>2</sub>O:DMSO-*d*<sub>6</sub> (8:2)
- S19 Absorbance intensity at 350 nm of probe **1** in DMSO:PBS (2:8) solutions with different pH values
- S20 Fluorescence emission spectra of derivative **3** in different fractions of H<sub>2</sub>O/DMSO mixture
- S20 Fluorescence emission spectra of derivative **4** in different fractions of H<sub>2</sub>O/DMSO mixture
- S21 Fluorescence emission spectra of derivative **3** in presence of hypochlorite in PBS/DMSO
- S21 Fluorescence emission spectra of derivative **4** in presence of hypochlorite in PBS/DMSO
- S22 UV-vis spectra of probe **5** in DMSO and DMSO: PBS (2/8, v/v) solvent mixture
- S22 Fluorescence spectra of probe **5** in DMSO and DMSO: PBS (2/8, v/v) solvent mixture
- S23 Fluorescence intensity at 520 nm of probe **5** under excitation at 450 nm in DMSO:PBS (2:8) solutions with different pH values
- S23 UV-vis spectra of probe **5** upon increasing concentration of hypochlorite in PBS/DMSO (8/2, v/v)
- S24 Fluorescence spectra of probe **5** upon increasing concentration of hypochlorite in PBS/DMSO (8/2, v/v)
- S24 Portable kit for detecting hypochlorite
- S25 Inhibition of homocoupling reaction of benzylamine
- S25 <sup>1</sup>H NMR spectrum of **7** in CDCl<sub>3</sub>

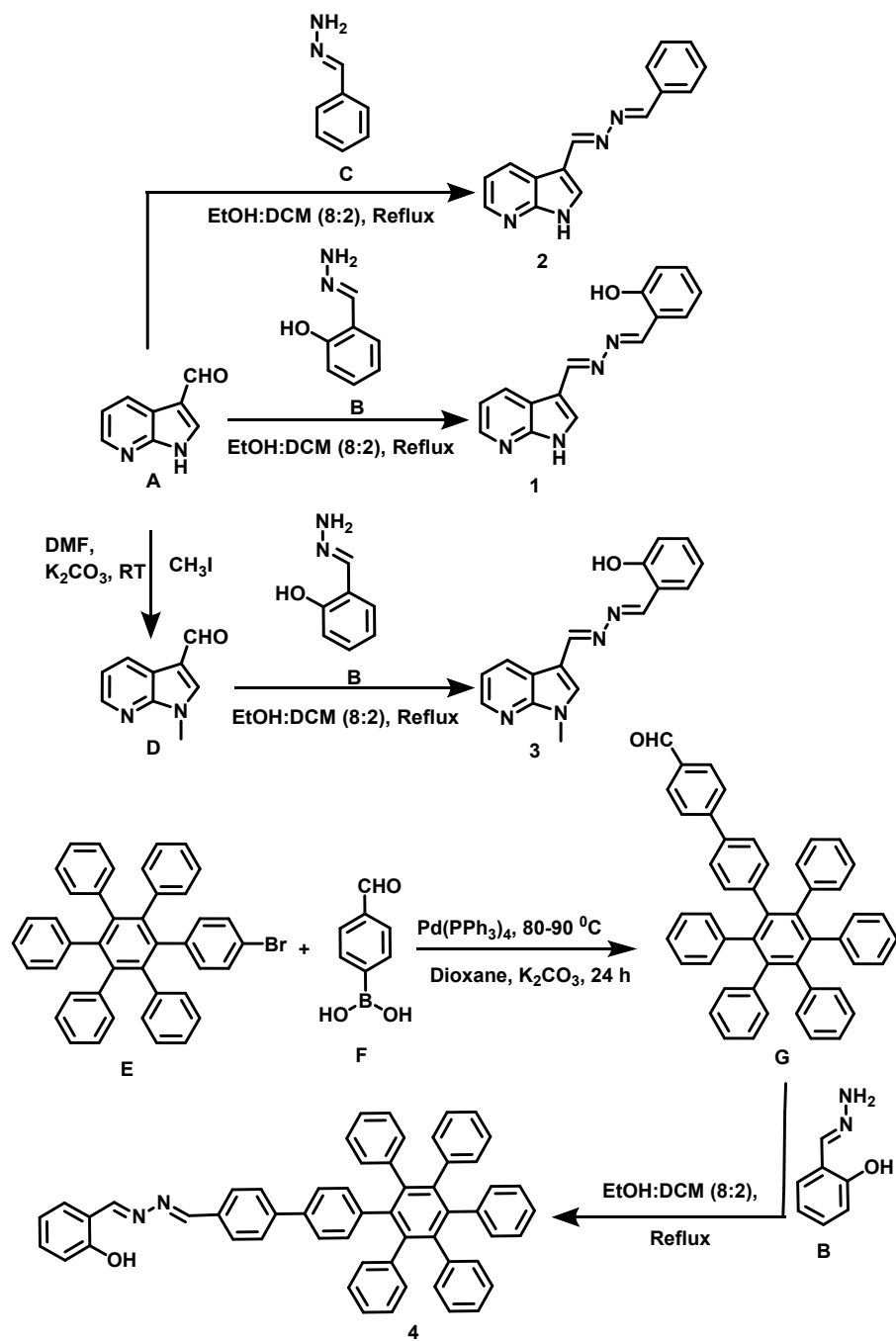
- S26  $^1\text{H}$  NMR spectrum of **6** in  $\text{CDCl}_3$
- S27 Cell cytotoxicity of probe **1** in L929 cell line at different concentrations
- S27 Cell cytotoxicity of sodium hypochlorite in L-929 cell line at different concentrations
- S28 Cytoprotection against sodium hypochlorite using probe **1** in L-929 cell at different concentrations
- S29  $^1\text{H}$  NMR spectrum of **1** in  $\text{DMSO-}d_6$
- S30  $^{13}\text{C}$  NMR spectrum of **1** in  $\text{DMSO-}d_6$
- S31 Mass spectrum of compound **1**
- S32  $^1\text{H}$  NMR spectrum of **2** in  $\text{DMSO-}d_6$
- S33  $^{13}\text{C}$  NMR spectrum of **2** in  $\text{CDCl}_3$
- S34 Mass spectrum of compound **2**
- S35  $^1\text{H}$  NMR spectrum of **D** in  $\text{CDCl}_3$
- S36  $^1\text{H}$  NMR spectrum of **3** in  $\text{CDCl}_3$
- S37  $^{13}\text{C}$  NMR spectrum of **3** in  $\text{DMSO-}d_6$
- S38 Mass spectrum of compound **3**
- S39  $^1\text{H}$  NMR spectrum of compound **G** in  $\text{CDCl}_3$
- S40  $^{13}\text{C}$  NMR spectrum of compound **G** in  $\text{CDCl}_3$
- S41 Mass spectrum of compound **G**
- S42  $^1\text{H}$  NMR spectrum of compound **4**
- S43  $^{13}\text{C}$  NMR spectrum of compound **4**
- S44 Mass spectrum of compound **4**

**Synthesis of compound C.** Compound C has been synthesized according to the reported procedure.<sup>1</sup>

**Synthesis of compound D<sup>2</sup>.** A clear solution of 7-Azaindole-3-carboxaldehyde (**A**) (0.09g, 0.68 mmol) and methyl iodide (0.21g, 1.50 mmol) in DMF was stirred at room temperature in presence of K<sub>2</sub>CO<sub>3</sub> (0.187g, 1.36 mmol) for 24 h. After the completion of the reaction (monitored by TLC), the reaction mixture was filtered. The filtrate was distilled under reduced pressure to give a solid residue and organic part was extracted with ethyl acetate and organic layer was dried over anhydrous sodium sulphate and evaporated under reduced pressure to give a solid residue. The product was purified by column chromatography on silica gel using EtOAc: hexane (2:8) as eluent to afford product D as white solid in 80% yield; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, ppm) δ = 9.97 (s, 1H, CHO), 8.56 (d, *J* = 4 Hz, 1H, Ar-H), 8.45 (d, *J* = 4 Hz, 1H, Ar-H), 7.85 (s, 1H, Ar-H), 7.30-7.28 (m, 1H, Ar-H), 3.98 (s, 3H, CH<sub>3</sub>) (Fig. S45).

**Synthesis of compound G.** To a solution mixture of **E**<sup>3</sup> (0.4 g, 0.65 mmol) and **F** (0.12 g, 0.78 mmol) in 1,4-dioxane (20 mL) were added K<sub>2</sub>CO<sub>3</sub> (0.71 g, 5.19 mmol), distilled H<sub>2</sub>O (1 mL), and [Pd(PPh<sub>3</sub>)<sub>4</sub>] (0.37 g, 0.32 mmol) under N<sub>2</sub> atmosphere. The reaction mixture was degassed with nitrogen for three times and was refluxed at 80-90 °C under the inert atmosphere for 24 h. The THF was then removed under vacuum, and the residue so obtained was treated with water, extracted with chloroform and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The organic layer was evaporated and compound was purified by column chromatography using (10:90) (hexane/chloroform) as an eluent to give compound G, and recrystallized from methanol to afford pale yellow solid in 70% yield; mp: > 250 °C; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>, ppm). δ = 9.99 (s, 1H, CHO), 7.85 (d, *J* = 9 Hz, 2H, Ar-H), 7.60 (d, *J* = 9 Hz, 2H, Ar-H), 7.18 (d, *J* = 6 Hz, 2H, Ar-H), 6.94 (d, *J* = 6 Hz, 4H, Ar-H), 6.86 (m, 25H, Ar-H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 125 MHz, ppm) 191.74, 153.32, 145.98, 139.59, 135.47, 133.26, 130.55, 130.28, 130.19, 128.19, 127.75, 127.49, 126.90, 126.03; ESI/MS *m/z* calculated 639.2688, found 639.1964 [M + H]<sup>+</sup> (Fig. S49-S51).

## Synthetic Schemes

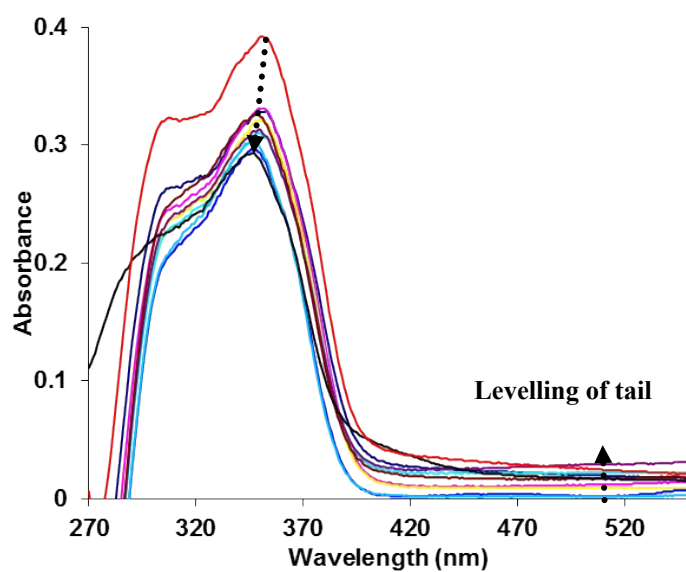


**Scheme S1.** Synthesis of derivatives 1, 2, 3 and 4.

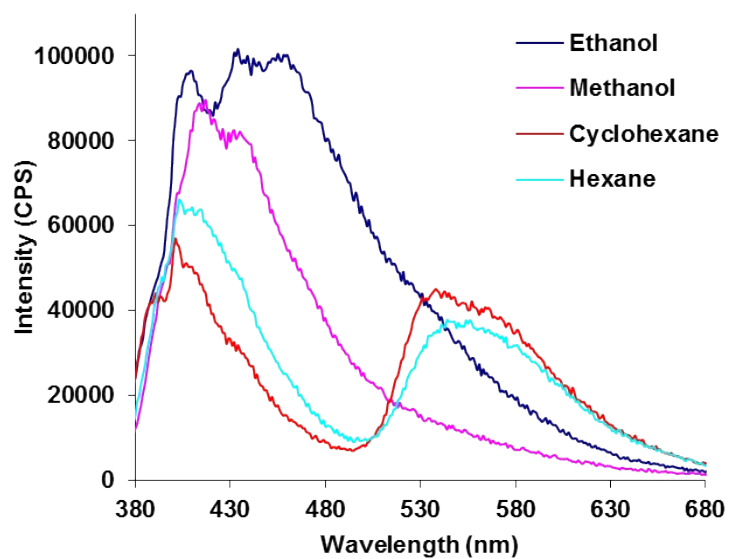
**Table S1.** Comparison of ESIPT-AIE active probe **1** in present manuscript over other literature reports.

S. No	Journal	Solvent media	Single molecule/Self-assembled probe	Reaction based	Reversibility	Colorimetric	Cell/Tissue/Animal imaging	Anti-oxidant	Detection limit
<b>1</b>	<b>Present work</b>	<b>PBS:DMSO (8:2)</b>	<b>Self-assembled</b>	<b>Reversible Deprotonation-protonation</b>	<b>Yes</b>	<b>Yes</b>	<b>Yes</b>	<b>Yes</b>	<b>50.2 nM</b>
2	<i>Anal. Chem.</i> <b>2020</b> , 92, 6072	buffer / methanol (4 :1, v/v)	Single molecule	Cleavage of thioether bond to form alkene	No	No	Yes	No	0.053 $\mu$ M
3	<i>Chem. Commun.</i> , <b>2020</b> , 56, 1219	PBS	Single molecule	Cleavage of C=C bond to form aldehyde group	No	Yes	Yes	No	49.1 nM
4	<i>J. Mater. Chem. B</i> , <b>2020</b> , 8, 125	CH <sub>3</sub> CN: H <sub>2</sub> O (1 : 1, v/v)	Self-assembled	Cleavage of C=N linkage to form aldehyde group	No	Yes	Yes	No	1 nM
5	<i>Anal. Chem.</i> <b>2020</b> , 92, 3262	PBS	Single molecule	Cleavage of C-N bond results in open form of rhodamine dye	No	Yes	Yes	No	13 nM
6	<i>Chem. Commun.</i> , <b>2019</b> , 55, 10768	PBS/DMF (7/3, v/v)	Self-assembled	Oxidation of sulphur	No	No	No	No	-
7	<i>Chem. Commun.</i> , <b>2019</b> , 55, 2533	PBS: DMF (5/5)	Single molecule	Cleavage of C=N linkage to form carboxylic group	No	Yes	Yes	No	0.59 $\mu$ M
8	<i>ACS Appl. Mater. Interfaces</i> <b>2019</b> , 11, 32489	H <sub>2</sub> O	Self-assembled	Oxidation of hydroxyl group to positively charged radical	No	No	No	No	1.9 nM
9	<i>Chem. Commun.</i> , <b>2018</b> , 54, 8522	PBS buffer (containing 1% DMSO, 1 mM CTAB)	Single molecule	Cleavage of dimethylthiocarbamate and form hydroxyl group	No	No	Yes	No	.016nM
10	<i>J. Mater. Chem. B</i> , <b>2019</b> , 7, 7332	(DMSO/PBS 1 : 4, v/v)	Single molecule	Addition of Cl around C=C bond	No	Yes	Yes	No	0.012 $\mu$ M
11	<i>Chem. Commun.</i> ,	PBS/DMF	Single molecule	Oxidation of	No	No	Yes	No	16 nM

	<b>2019</b> , 55, 12912	(1/1, v/v)		sulphur					
12	<i>J. Mater. Chem. B</i> , <b>2019</b> , 7, 6861	PBS (0.1% CH <sub>3</sub> CN)	Single molecule	Oxidation of selenium	No	No	Yes	No	0.3 nM
13	<i>Chem. Commun.</i> , <b>2018</b> , 54, 7967	PBS	Single molecular	Cleavage of dimethylthiocarba mate and form hydroxyl group	No	No	Yes	No	2.37 nM
14	<i>Chem. Sci.</i> , <b>2018</b> , 9, 8207	PBS	Single molecule	Addition on C=C bond to form epoxide ring	No	No	Yes	No	0.1 μM
15	<i>Chem. Commun.</i> , <b>2018</b> , 54, 9238	PBS/DMF (8/2, v/v)	Single molecule	Open form of fluorescein on cleavage of C-N bond	No	No	Yes	No	6.2×10 <sup>-8</sup> M

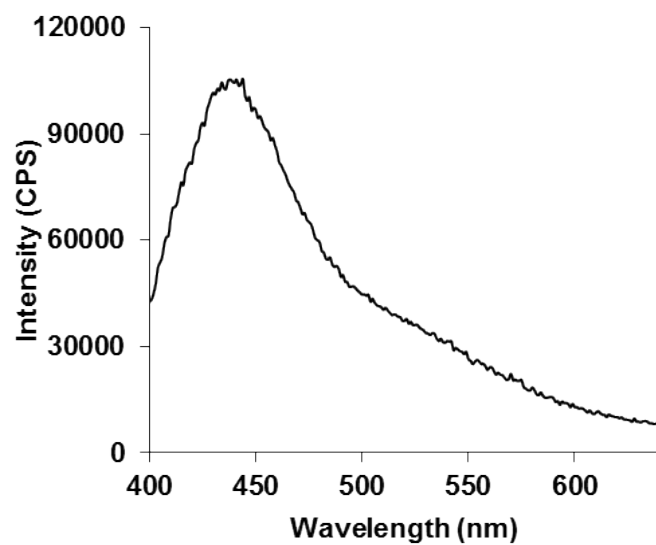


**Fig. S1** UV-vis spectra of probe **1** in different fractions of H<sub>2</sub>O/DMSO mixture.

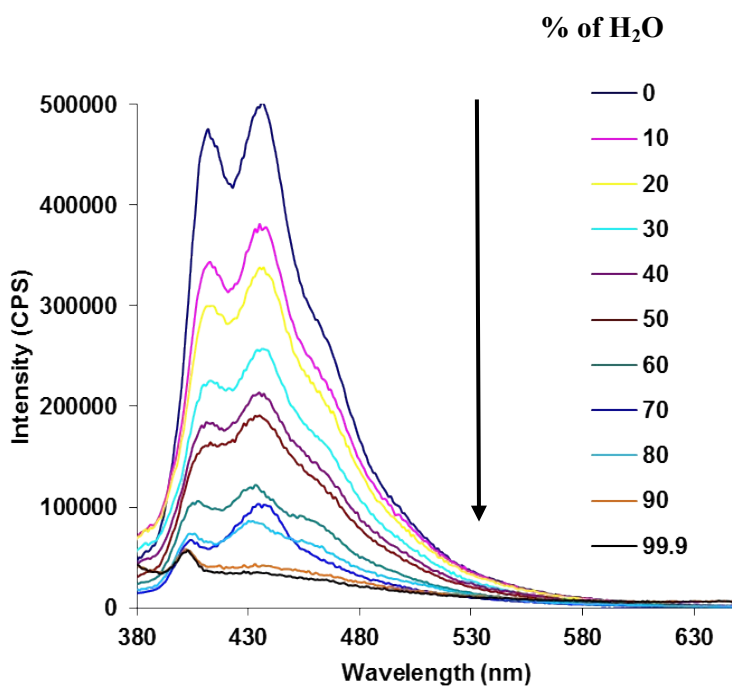


**Fig. S2** Fluorescence spectra of probe **1** (5.0 μM) in different solvents;  $\lambda_{\text{ex}} = 350\text{nm}$ .

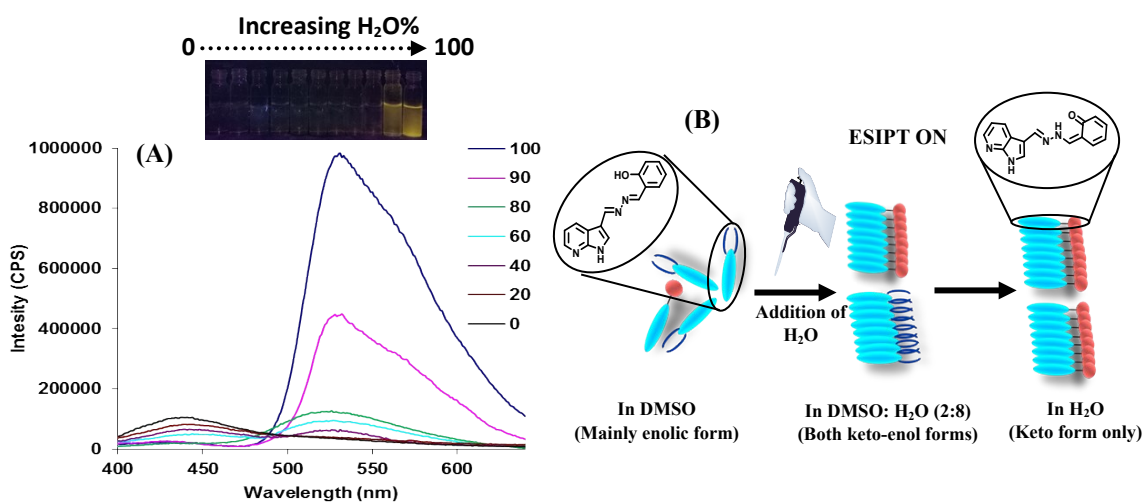




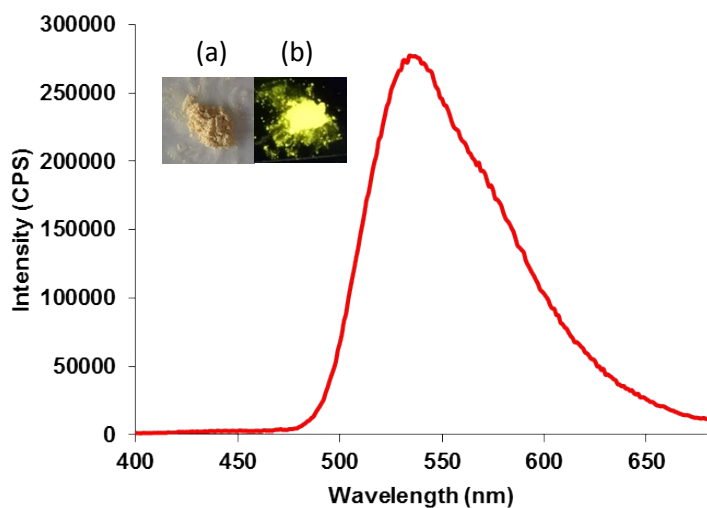
**Fig. S3** Fluorescence emission spectrum of probe 1 in DMSO.



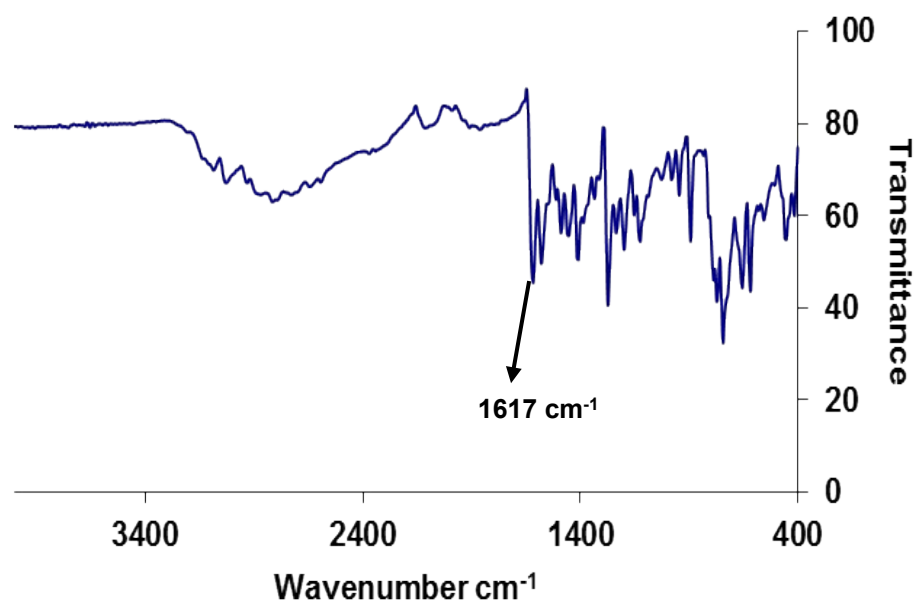
**Fig. S4** Fluorescence emission spectra of derivative 2 (5.0  $\mu$ M) in different fractions of DMSO/H<sub>2</sub>O mixture;  $\lambda_{\text{ex}} = 340$  nm.



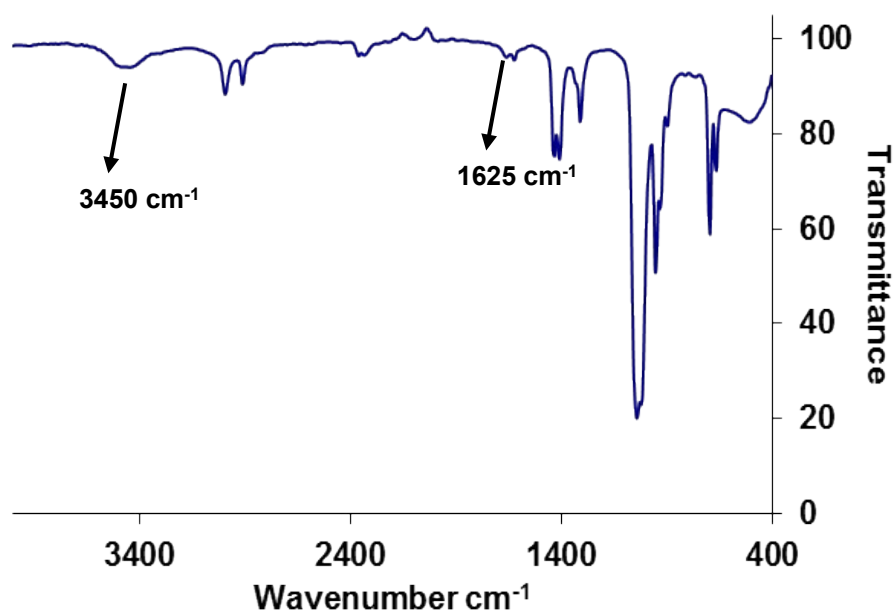
**Fig. S5** (A) Fluorescence emission spectra of probe **1** in different fractions of H<sub>2</sub>O/DMSO mixture,  $\lambda_{\text{ex}} = 350$  nm. (B) Schematic representation of ESIP and AIE phenomenon of probe **1**. Inset showing the photograph with different water fractions under 365 nm UV lamp illumination.



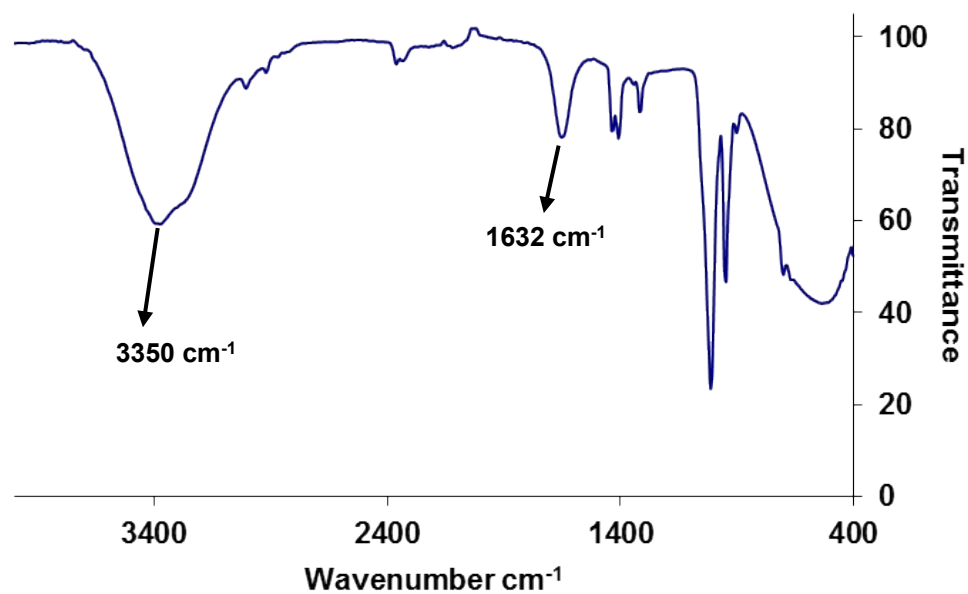
**Fig. S6** Fluorescence emission spectra of probe **1** in solid state (slit width 1:1),  $\lambda_{\text{ex}} = 350$  nm. Inset showing the photographs (a) in day light and (b) under 365 nm UV lamp illumination.



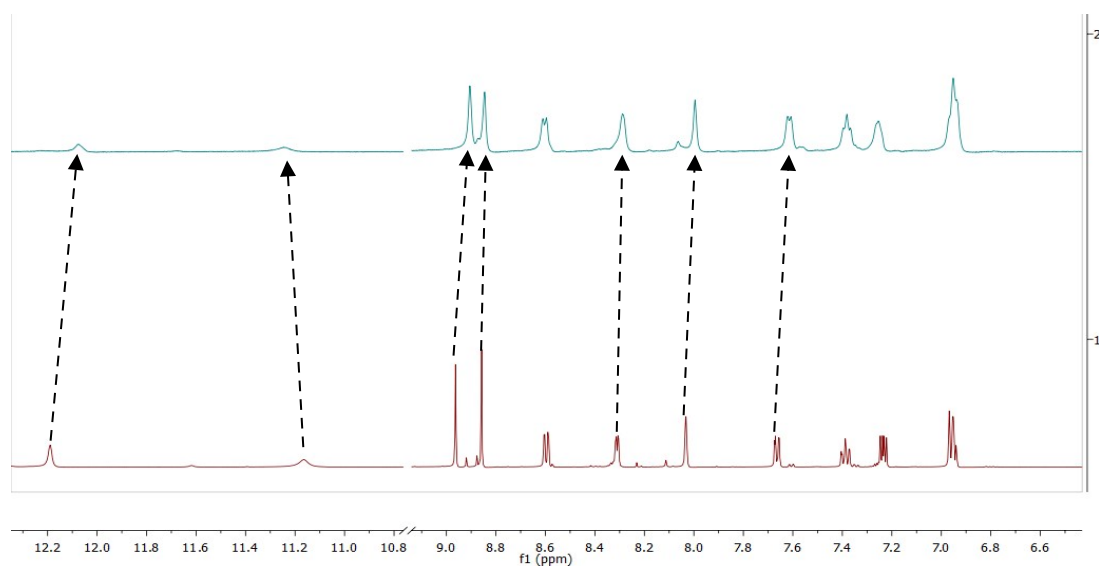
**Fig. S7** FT IR Spectrum of probe **1** in solid state showed intense peak at 1617 cm<sup>-1</sup> corresponding to carbonyl group.



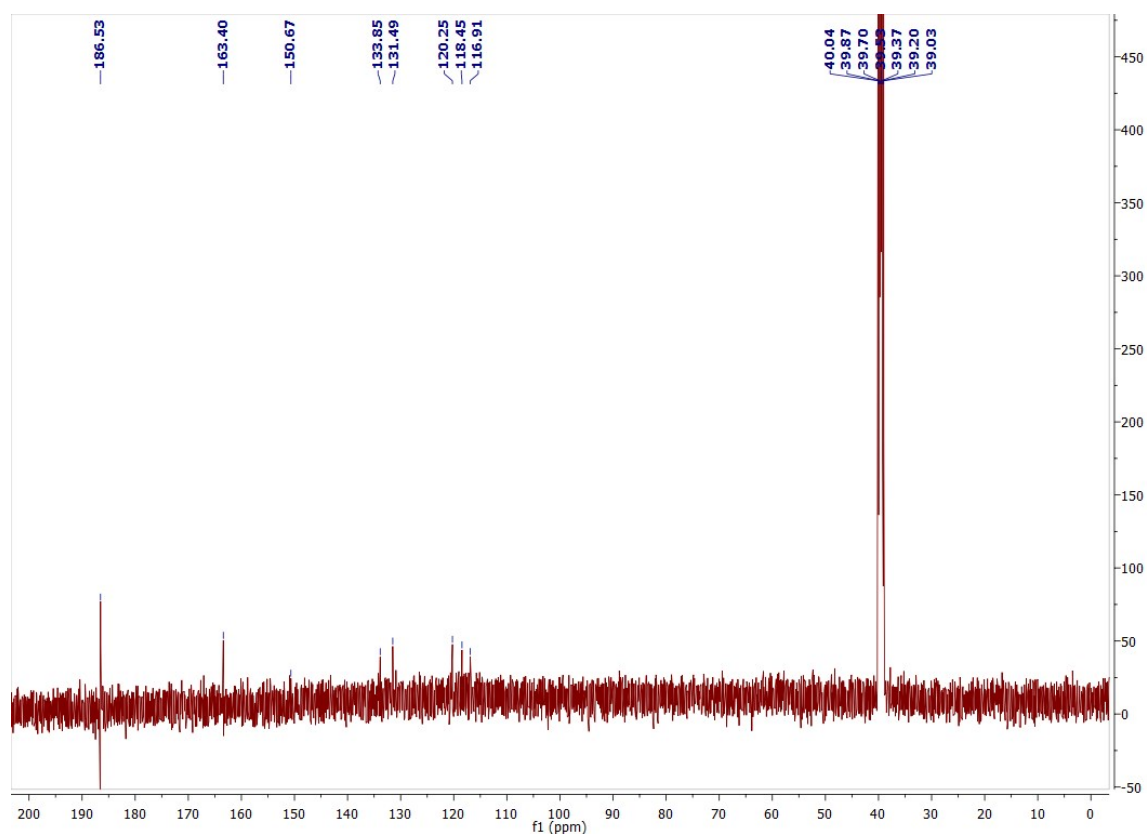
**Fig. S8** FT IR Spectrum of probe **1** in DMSO showed peak at 1625 cm<sup>-1</sup> and 3450 cm<sup>-1</sup> corresponding to carbonyl and hydroxyl group respectively.



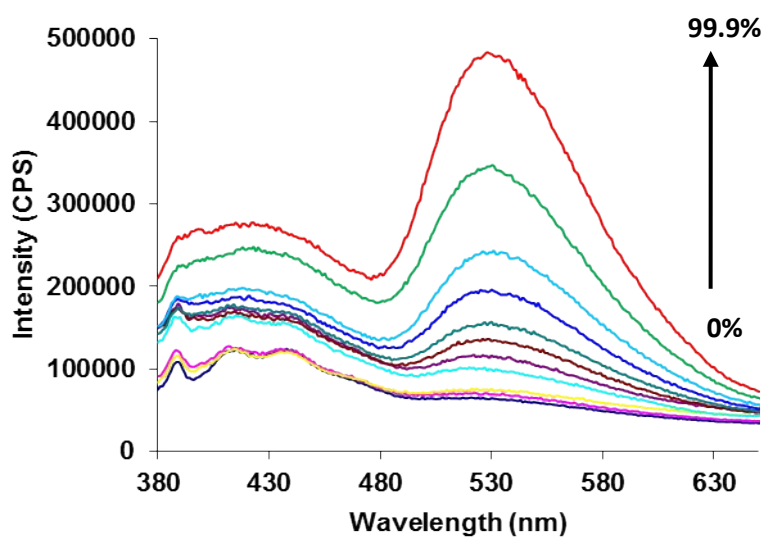
**Fig. S9** FTIR Spectrum of probe **1** in DMSO: H<sub>2</sub>O showed peak at 1632 cm<sup>-1</sup> and 3350 cm<sup>-1</sup> corresponding to carbonyl and hydroxyl group respectively.



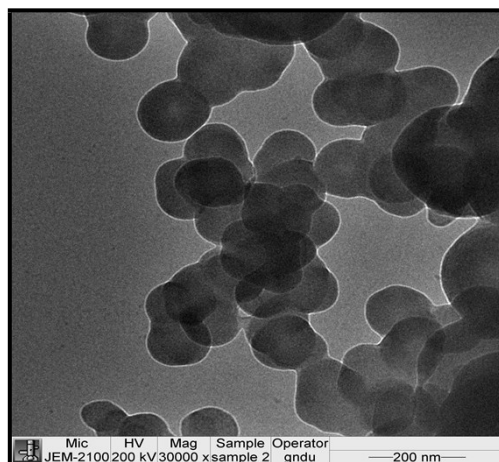
**Fig. S10** <sup>1</sup>H NMR of probe **1** in DMSO-*d*<sub>6</sub> and DMSO-*d*<sub>6</sub>: D<sub>2</sub>O (2:8) mixture showed upfield shifting of aromatic protons.



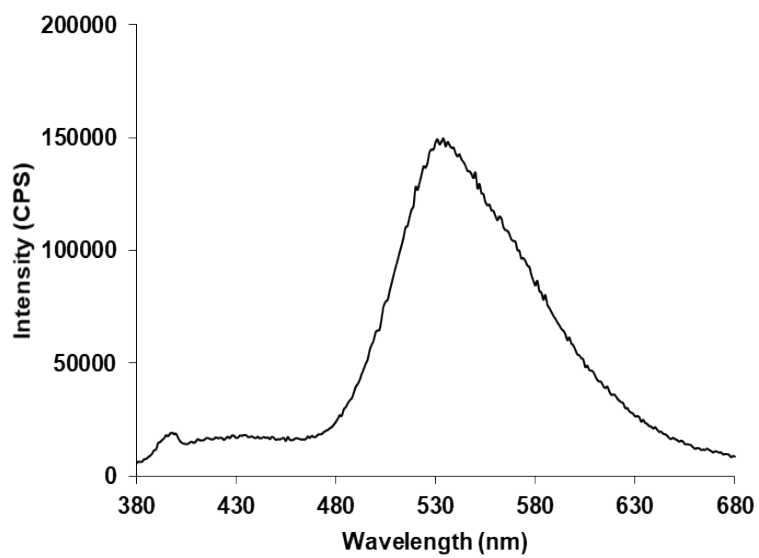
**Fig. S11**  $^{13}\text{C}$  NMR of probe **1** in DMSO- $d_6$ : D $_2$ O (2:8) mixture showed signal at 186.53 ppm corresponding to carbonyl group.



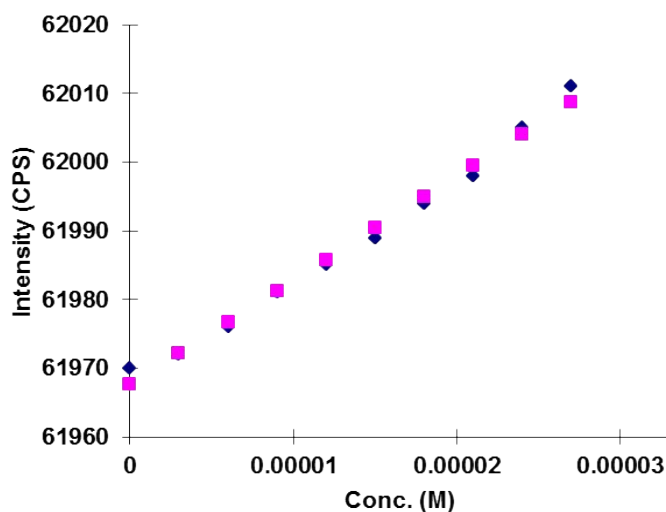
**Fig. S12** Fluorescence emission spectra of probe **1** (5.0  $\mu\text{M}$ ) in different fractions of glycerol: DMSO;  $\lambda_{\text{ex}} = 350\text{nm}$ .



**Fig. S13** TEM image of probe **1** in H<sub>2</sub>O/DMSO (8/2, v/v) mixture.



**Fig. S14** Fluorescence spectrum of probe **1** in PBS/DMSO (8/2, v/v) mixture;  $\lambda_{\text{ex}} = 350$  nm.



**Fig. S15** The fluorescence response of probe **1** (5.0  $\mu\text{M}$ ) to various concentrations of NaOCl in  $\text{H}_2\text{O}/\text{DMSO}$  (2/8, v/v) mixture;  $\lambda_{\text{ex}} = 350 \text{ nm}$ .

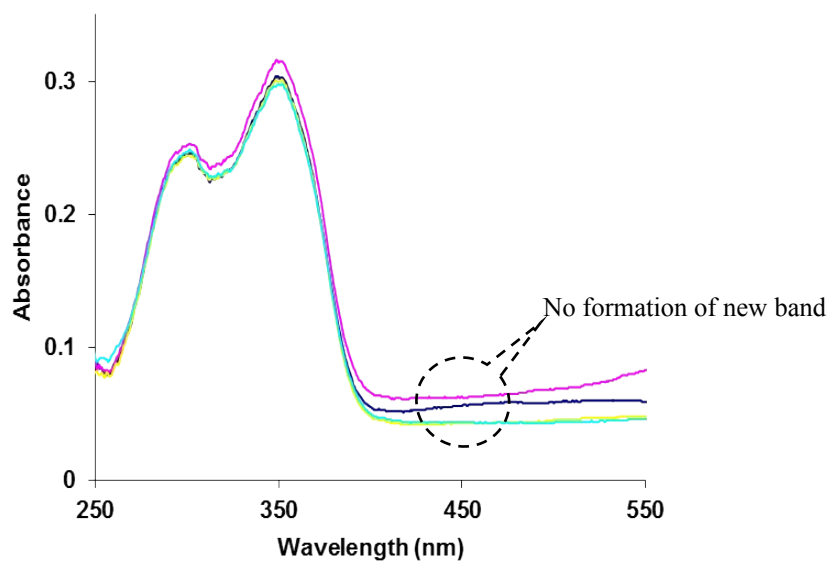
The detection limit was calculated based on the fluorescence titration. To determine the S/N ratio, the emission intensity of probe **1** without NaOCl was measured by 10 times and the standard deviation of blank measurements was determined. The detection limit is then calculated with the following equation:

$$\text{DL} = 3 \times \text{SD}/\text{S}$$

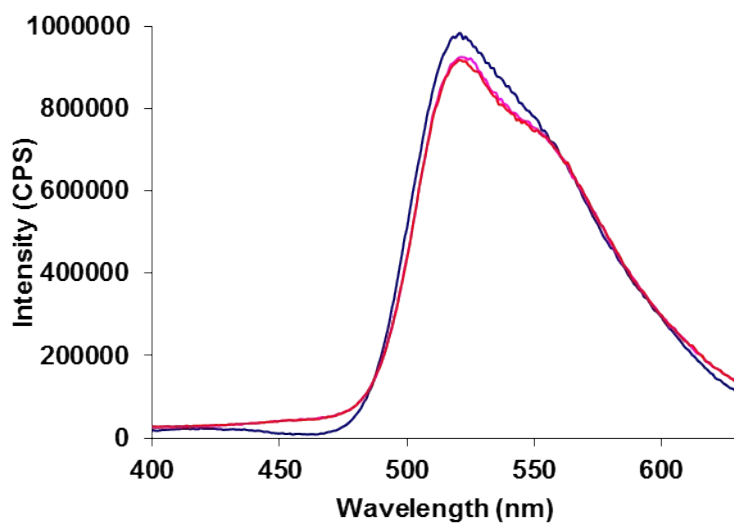
Where SD is the standard deviation of the blank solution measured by 10 times; S is the slope of the calibration curve.

From the graph, we get slope (S) = 1521212, and SD value is 0.0255

Thus using the formula we get the Detection Limit (DL) =  $50.2 \times 10^{-9} \text{ M}$ .

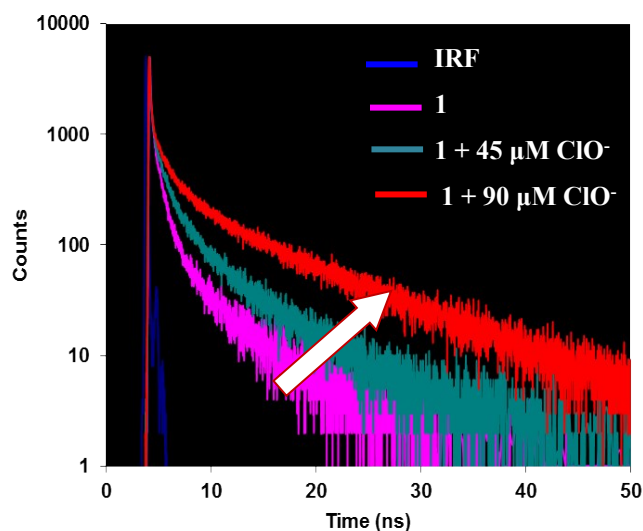


**Fig. S16** Absorption spectra of probe **1** upon increasing concentration of NaOCl (0-160  $\mu$ M) in water;  $\lambda_{\text{ex}} = 350$  nm.

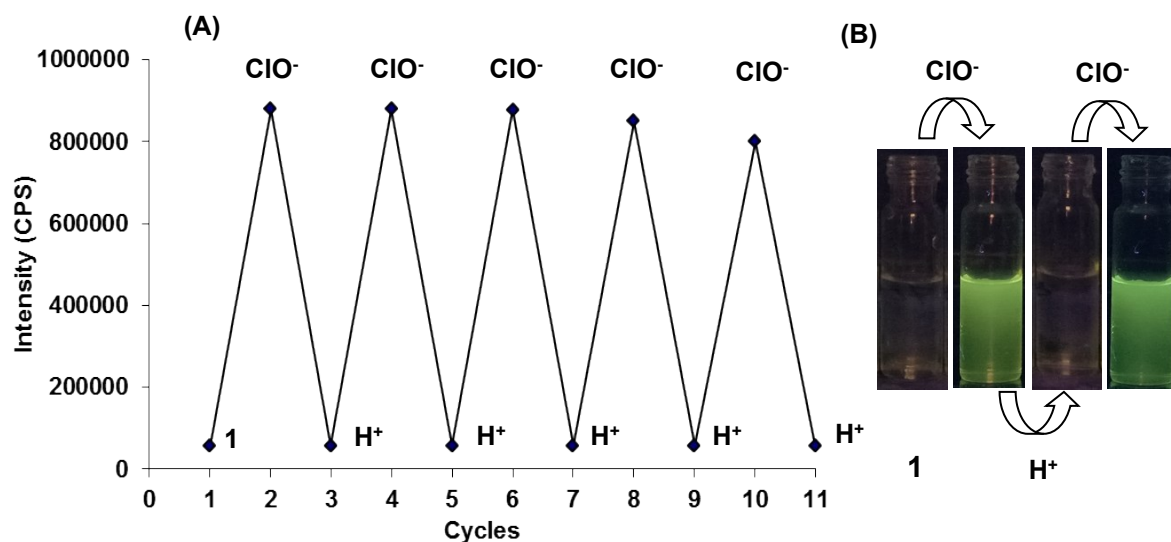


**Fig. S17** Fluorescence spectra of probe **1** upon increasing concentration of NaOCl (0-160  $\mu$ M) in water;  $\lambda_{\text{ex}} = 350$  nm.

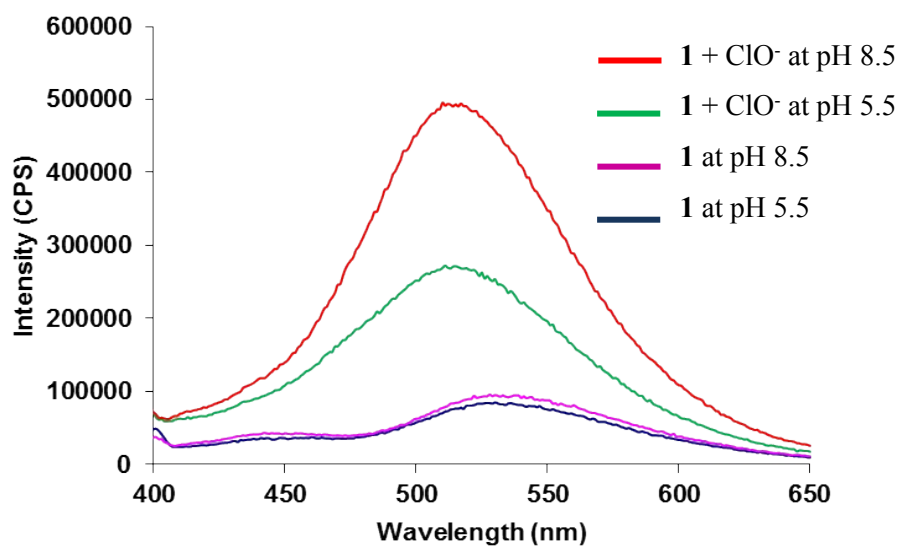




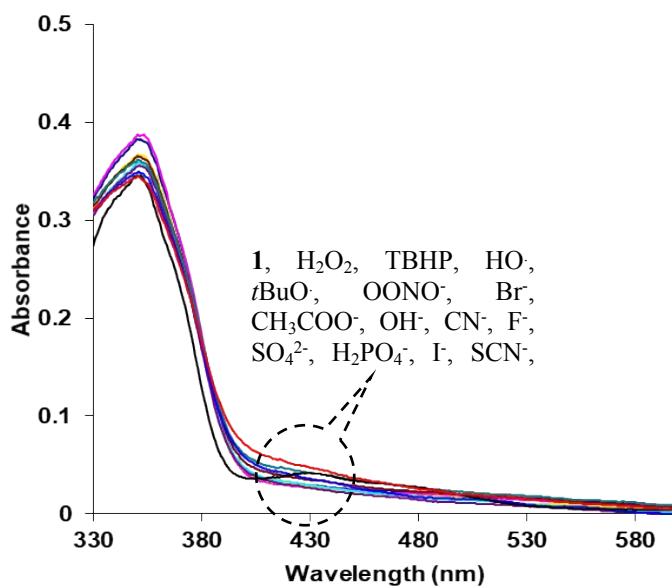
**Fig. S18** Fluorescence lifetime decay profiles of probe **1** in presence hypochlorite in PBS/DMSO (8/2, v/v). Arrow indicates the change in decay profile. IRF = instrument response function.  $\lambda_{\text{ex}} = 377$  nm and emission spectra are recorded at 510 nm with 32 slit width.



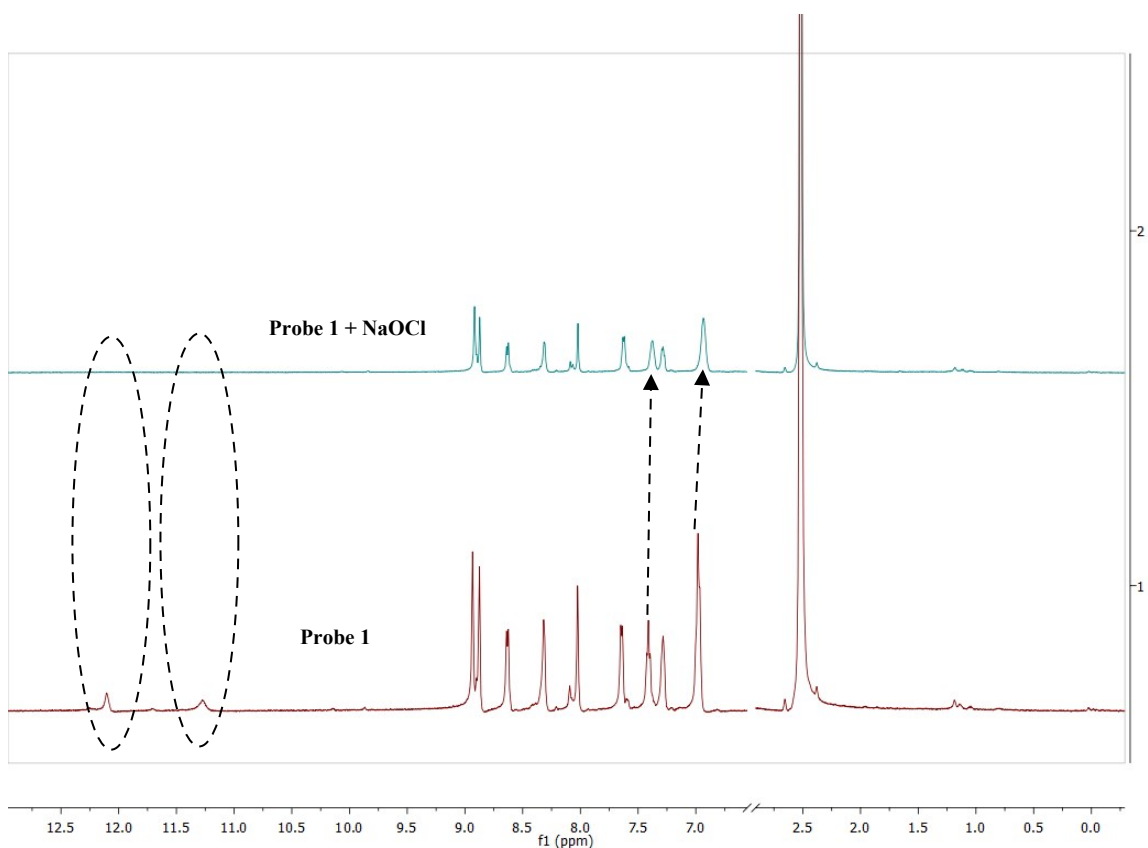
**Fig. S19** (A) Measuring the reversibility of fluorescence emission intensity at 510 nm by alternate addition of  $\text{ClO}^-$  and  $\text{H}^+$  to solution of **1** in PBS/DMSO (8/2, v/v), pH = 7.4;  $\lambda_{\text{ex}} = 350$  nm. (B) Photographs showed the emission change of free probe **1** under 365 nm UV lamp illumination and after alternate addition of  $\text{ClO}^-$  ions and  $\text{H}^+$ .



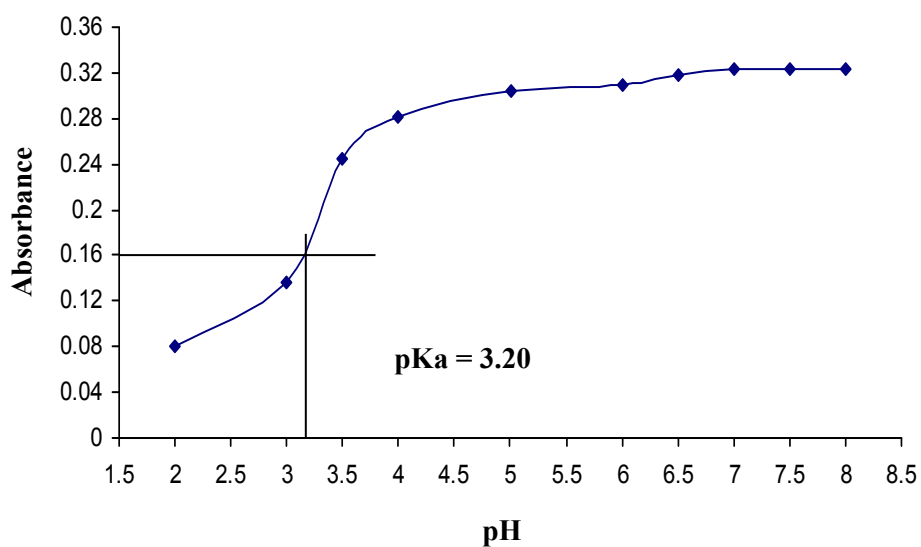
**Fig. S20** Fluorescence spectra of probe **1** (5.0  $\mu\text{M}$ ) in PBS/DMSO (8/2, v/v) mixture at pH 5.5 and 8.5 upon addition of NaOCl (100  $\mu\text{M}$ );  $\lambda_{\text{ex}} = 350 \text{ nm}$ .



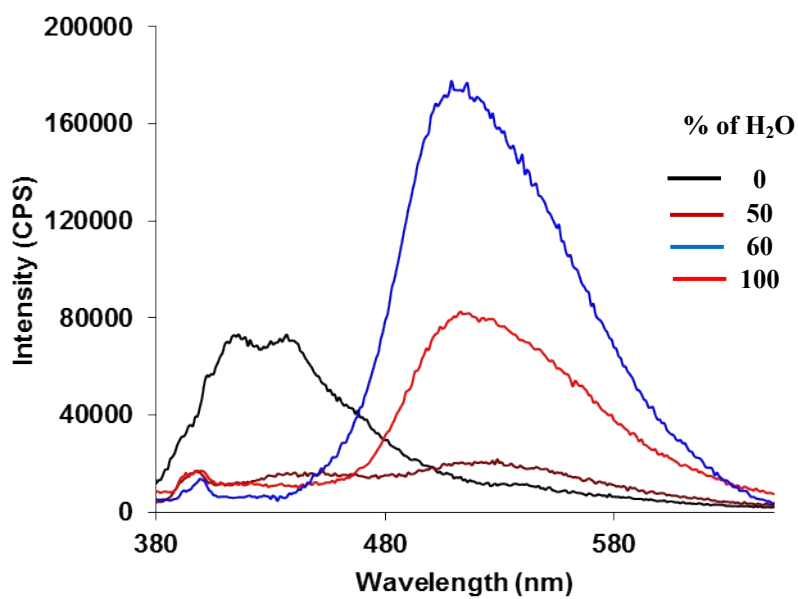
**Fig. S21** UV-vis spectra of probe **1** upon addition of different ROS and anions (0–100  $\mu\text{M}$ ) in PBS/DMSO (8/2, v/v) mixture, pH = 7.4.



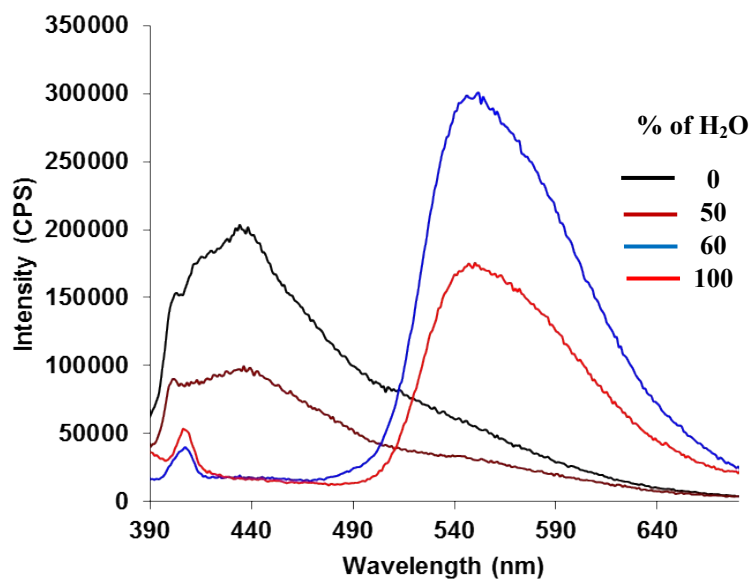
**Fig. S22**  $^1\text{H-NMR}$  titration of probe **1** in presence of sodium hypochlorite ( $15.0\ \mu\text{M}$ ) in  $\text{D}_2\text{O}:\text{DMSO-}d_6$  (8:2) in 500 MHz



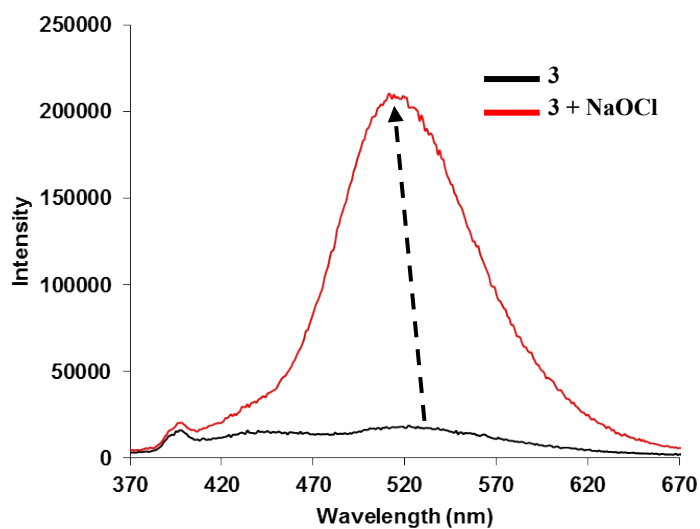
**Fig. S23** Absorbance intensity at 350 nm of probe **1** ( $5.0\ \mu\text{M}$ ) in  $\text{DMSO}:\text{PBS}$  (2:8) solutions with different pH values.



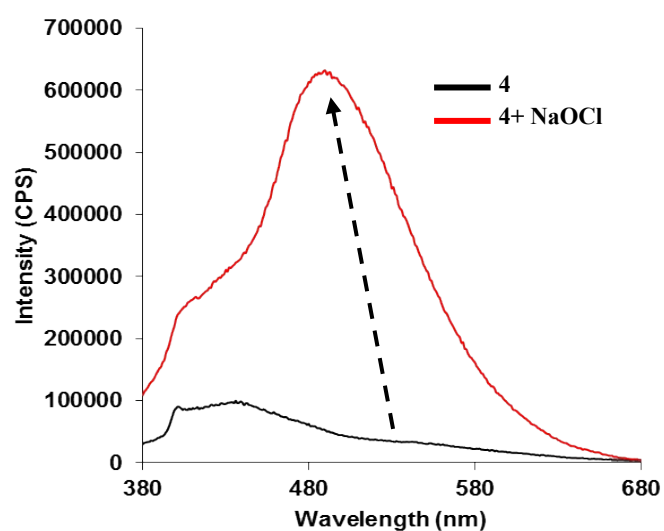
**Fig. S24** Fluorescence emission spectra of derivative **3** in different fractions of H<sub>2</sub>O/DMSO mixture;  $\lambda_{\text{ex}} = 350$  nm.



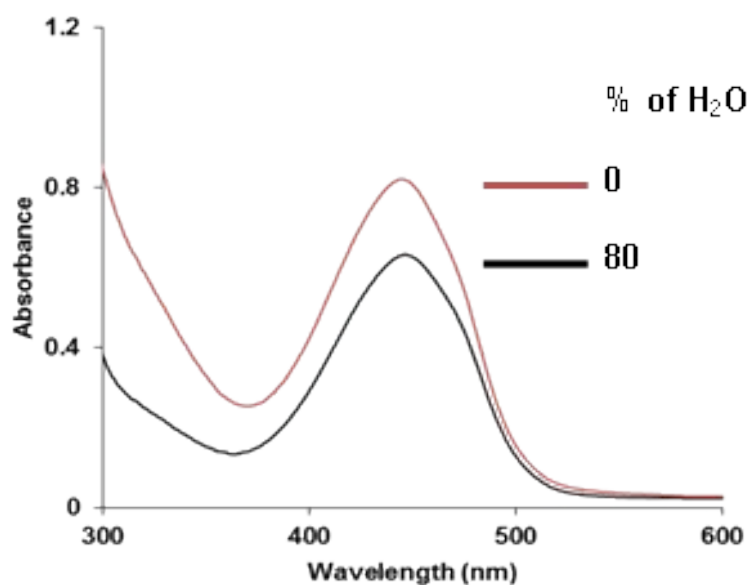
**Fig. S25** Fluorescence spectra of derivative **4** (5.0 μM) in different fraction of H<sub>2</sub>O/DMSO mixture;  $\lambda_{\text{ex}} = 350$  nm.



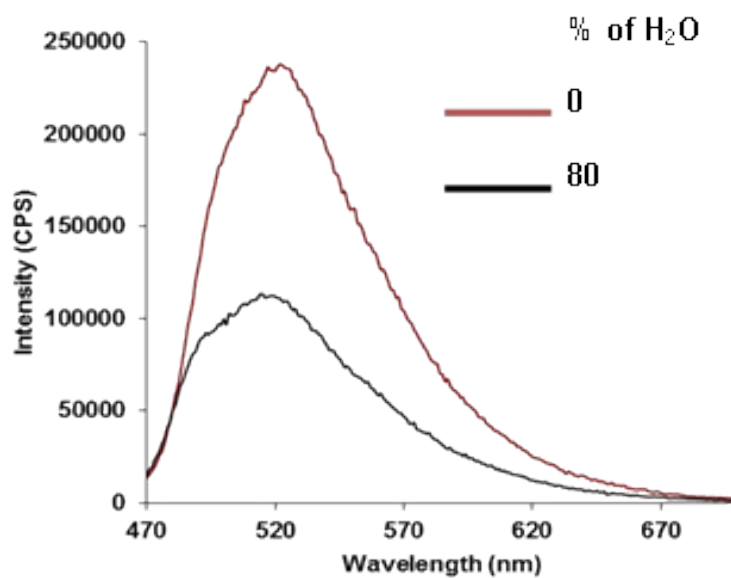
**Fig. S26** Fluorescence emission spectra of derivative **3** upon increasing concentration of hypochlorite (0-175  $\mu$ M) in PBS/DMSO (8/2, v/v) (50 mM), pH = 7.4;  $\lambda_{\text{ex}}$  = 350 nm.



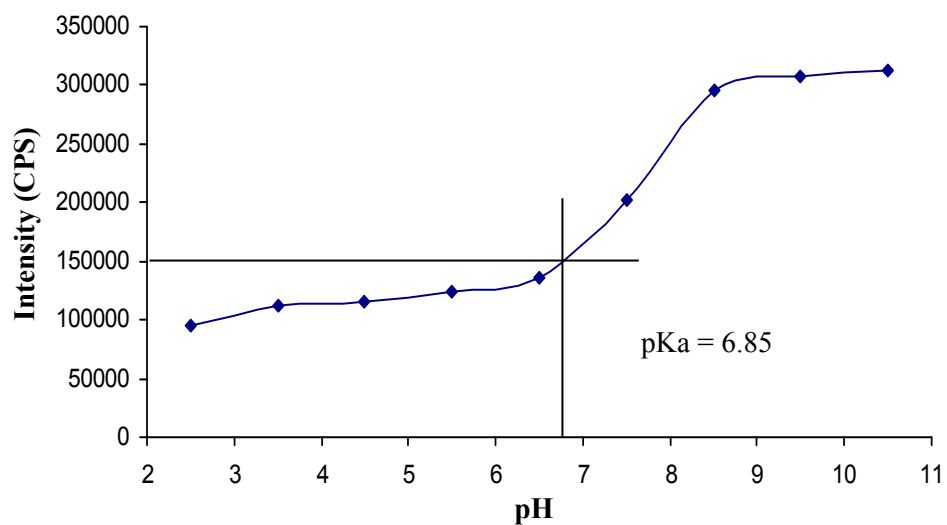
**Fig. S27** Fluorescence emission spectra of derivative **4** upon increasing concentration of hypochlorite (0-144  $\mu$ M) in PBS/DMSO (1/1, v/v) (50 mM), pH = 7.4;  $\lambda_{\text{ex}}$  = 350 nm.



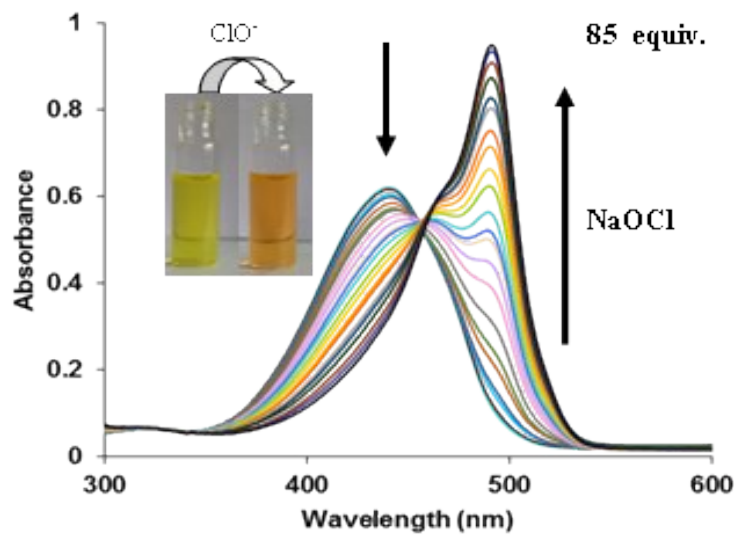
**Fig. S28** UV-vis spectra of probe **5** (5.0  $\mu\text{M}$ ) in DMSO and DMSO: PBS (2/8, v/v) (50 mM) solvent mixture, pH = 7.4.



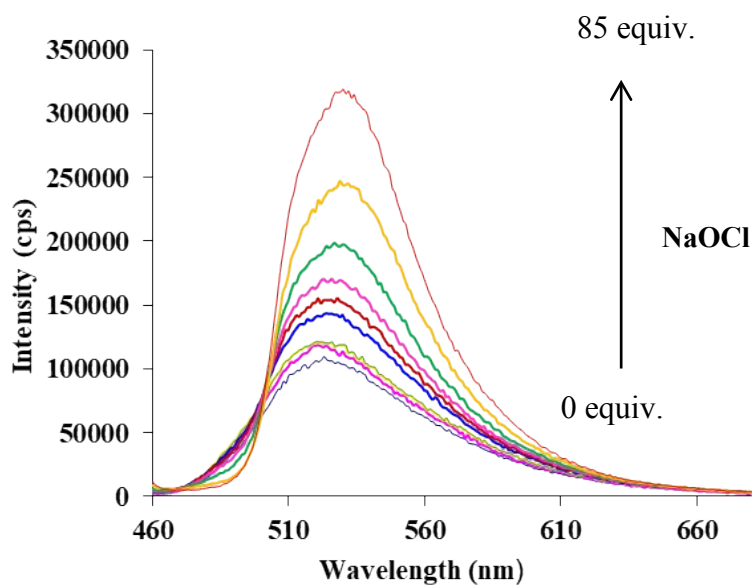
**Fig. S29** Fluorescence spectra of probe **5** (5.0  $\mu\text{M}$ ) in DMSO and DMSO: PBS (2/8, v/v) (50 mM) solvent mixture, pH = 7.4;  $\lambda_{\text{ex}}$  = 450 nm.



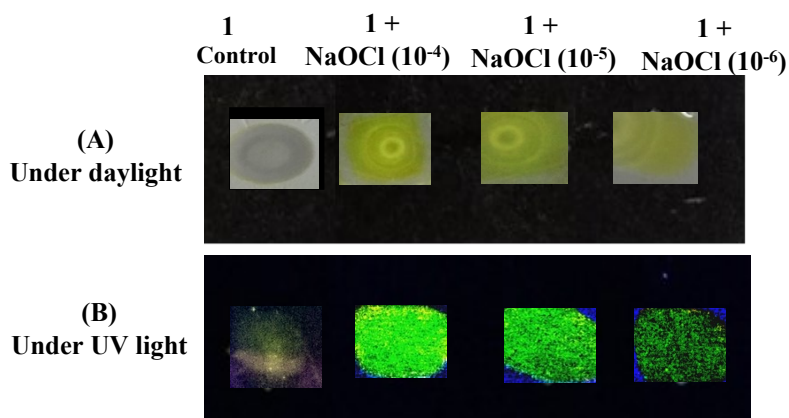
**Fig. S30** Fluorescence intensity at 520 nm of probe **5** (5.0  $\mu\text{M}$ ) under excitation at 450 nm in DMSO:PBS (2:8) solutions with different pH values.



**Fig. S31** UV-vis spectra of probe **5** upon increasing concentration of hypochlorite (0-85 equiv.) in PBS (50mM)/DMSO (8/2, v/v).

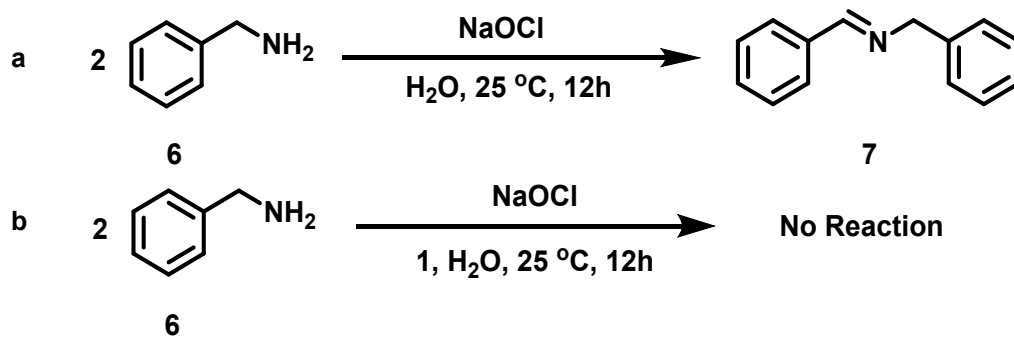


**Fig. S32** Fluorescence spectra of probe **5** upon increasing concentration of hypochlorite (0-85 equiv.) in PBS (50mM)/DMSO (8/2, v/v);  $\lambda_{\text{ex}} = 450$  nm.

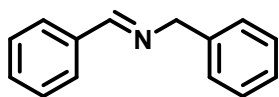


**Fig. S33** Portable kit for detecting hypochlorite, probe **1** coated holes treated with different concentration of NaOCl ranging from  $10^{-4}$  to  $10^{-6}$  M solutions. (A) under day light, (B) under UV lamp excitation wavelength 365 nm.

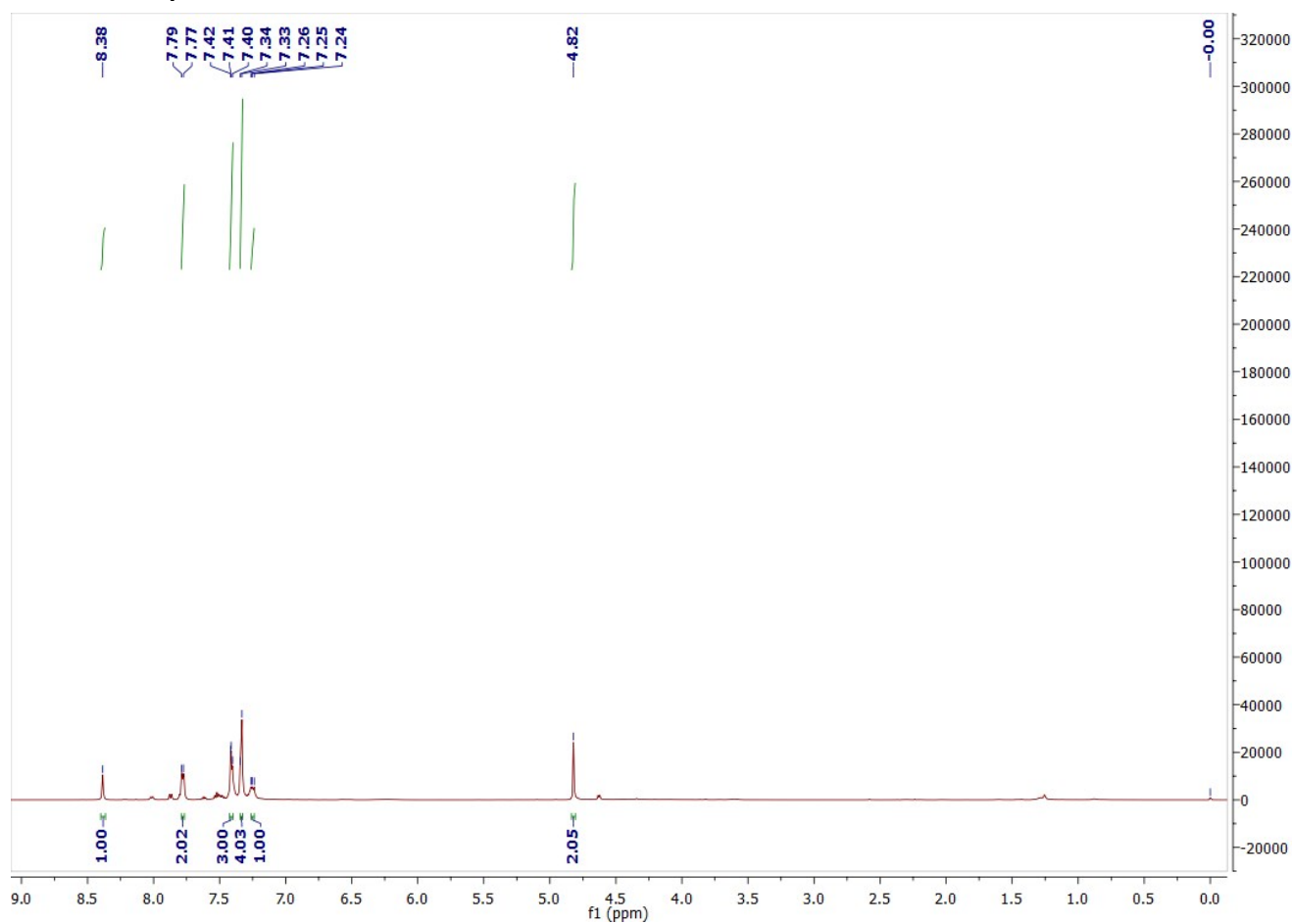




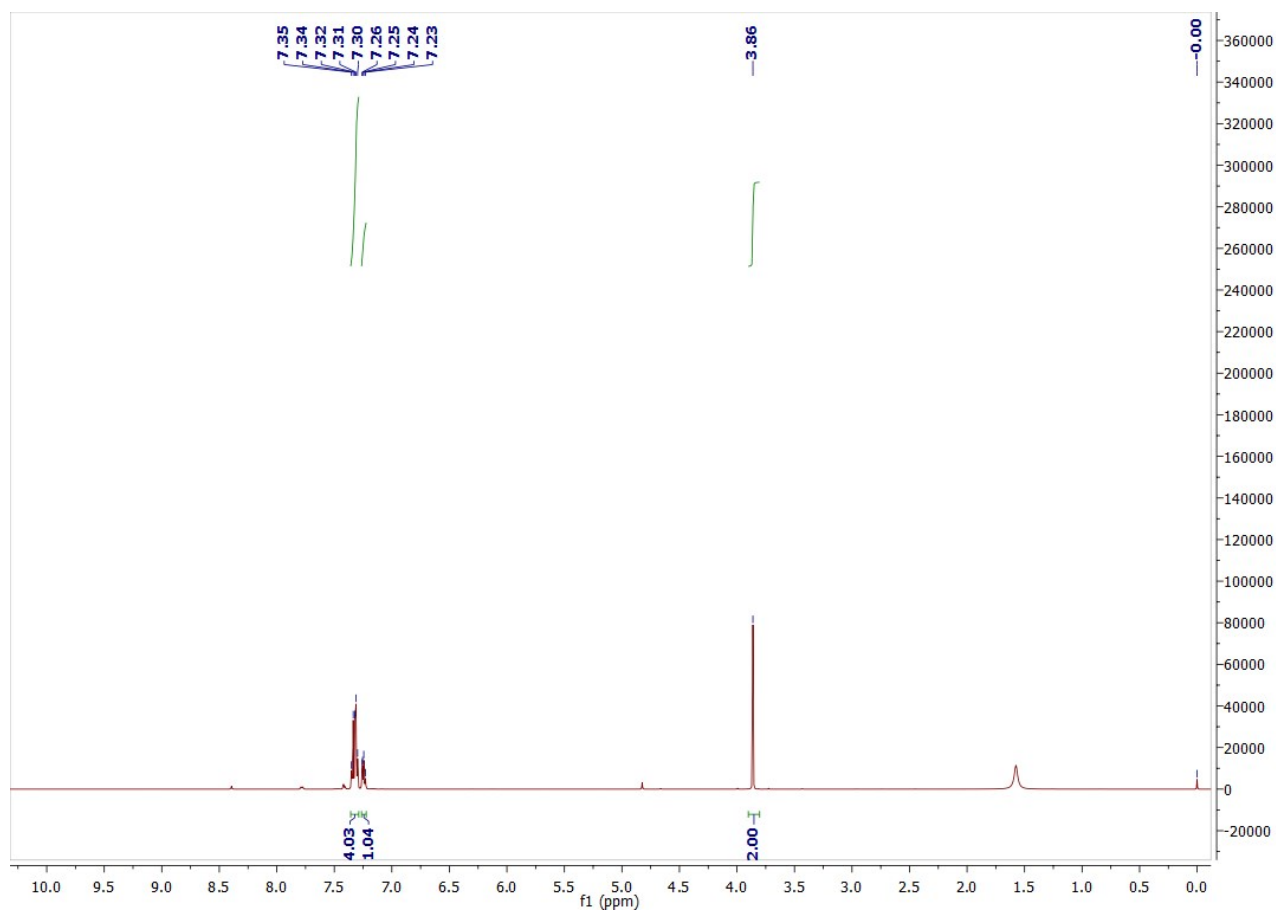
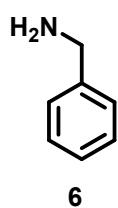
**Scheme S2** Inhibition of homocoupling reaction of benzylamine.



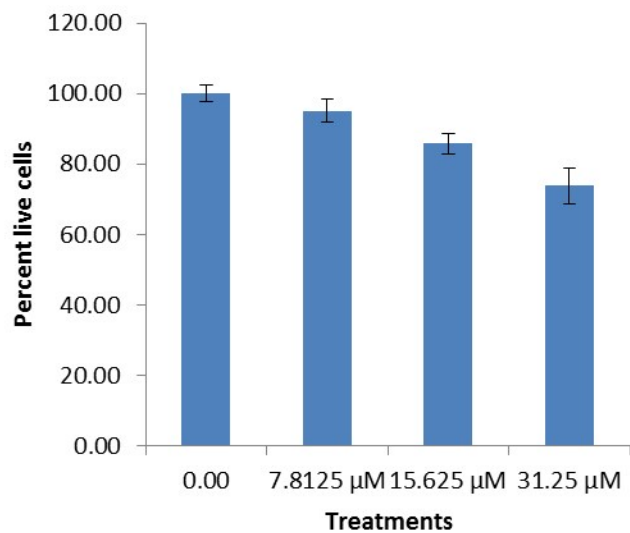
**7**



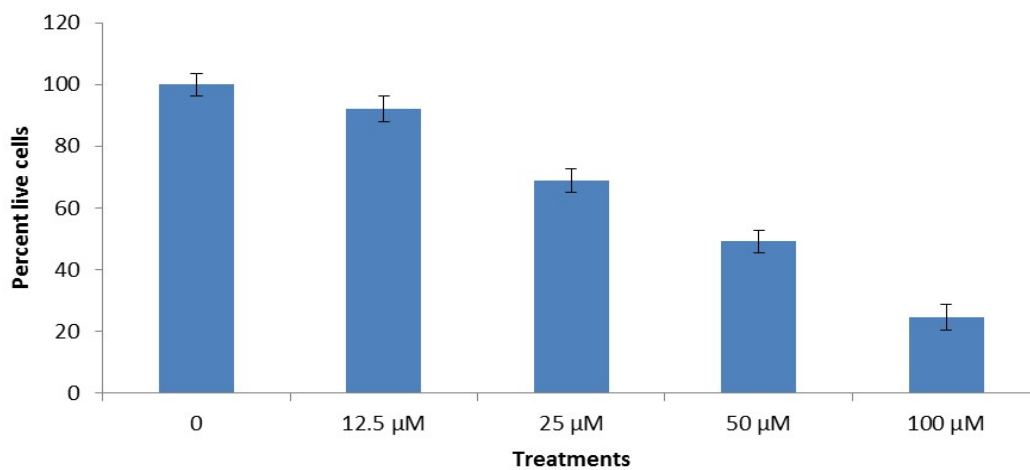
**Fig. S34**  $^1\text{H}$  NMR spectrum of **7** in  $\text{CDCl}_3$ .



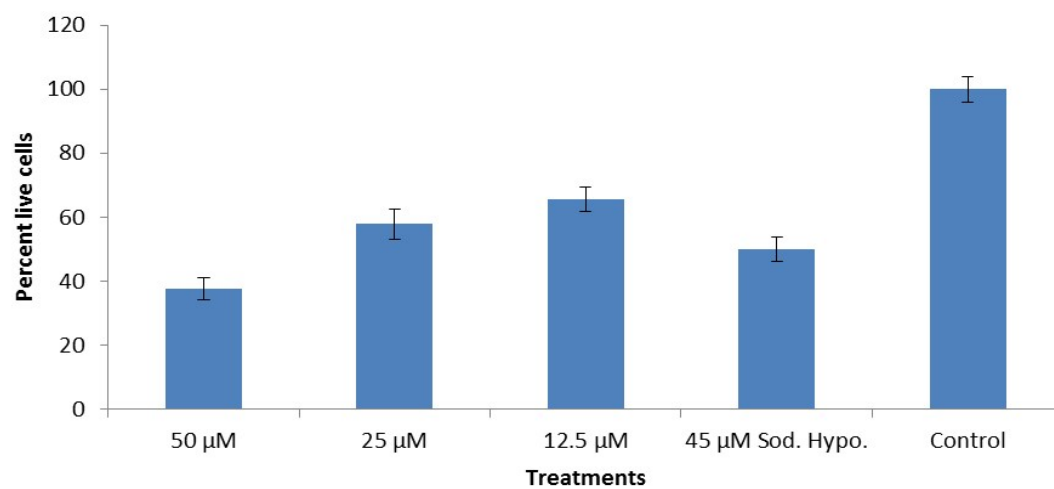
**Fig. S35**  $^1\text{H}$  NMR spectrum of **6** in  $\text{CDCl}_3$ .



**Fig. S36** Cell cytotoxicity of probe **1** in L929 cell line at different concentrations.

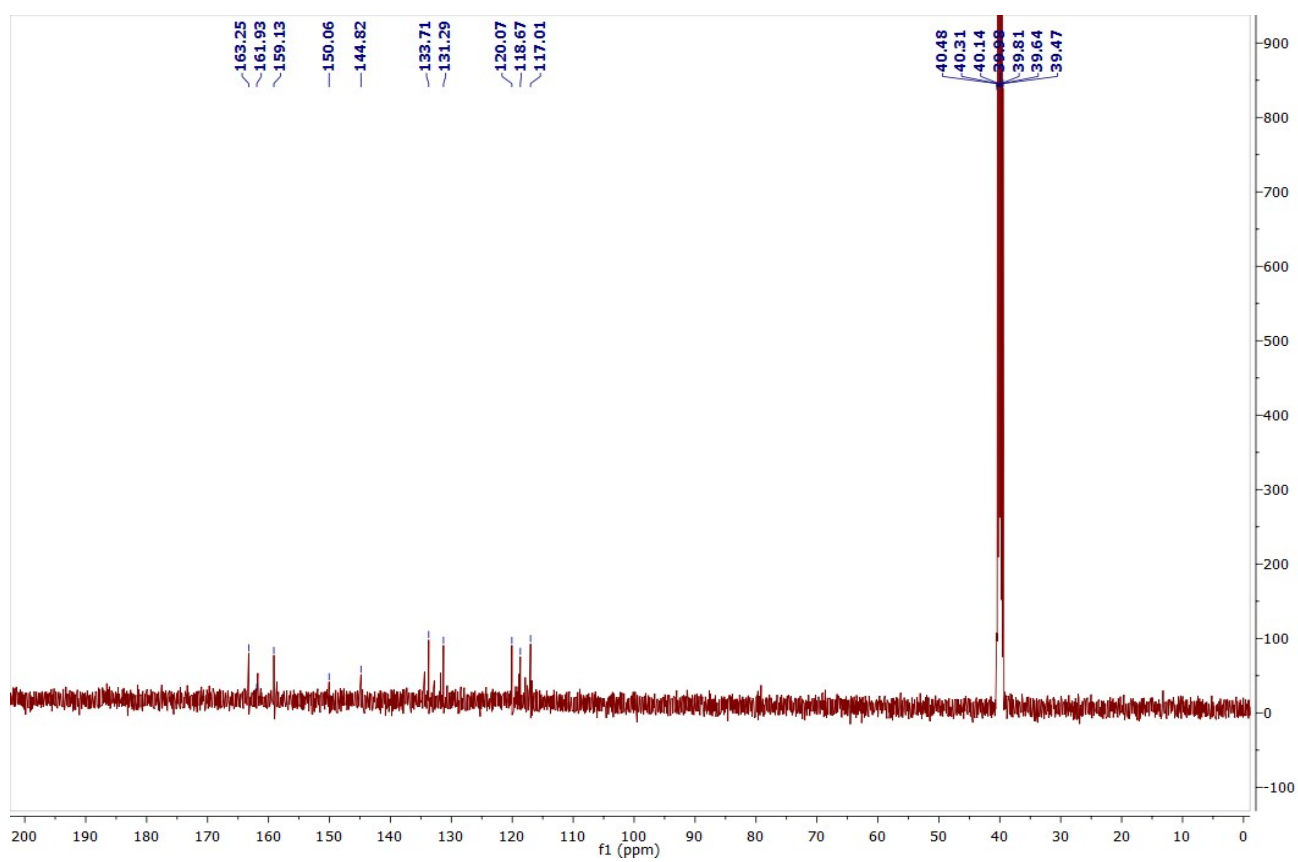


**Fig. S37** Cell cytotoxicity of sodium hypochlorite in L929 cell line at different concentrations. The calculated  $IC_{30}$ ,  $IC_{50}$  and  $IC_{70}$  are 24.81 μM, 45.57 μM and 83.71 μM respectively.



**Fig. S38** Percentage of live cells in presence of sodium hypochlorite in L929 cell lines at different concentrations of probe **1**.





**Fig. S40**  $^{13}\text{C}$  NMR spectrum of **1** in  $\text{DMSO-}d_6$ .

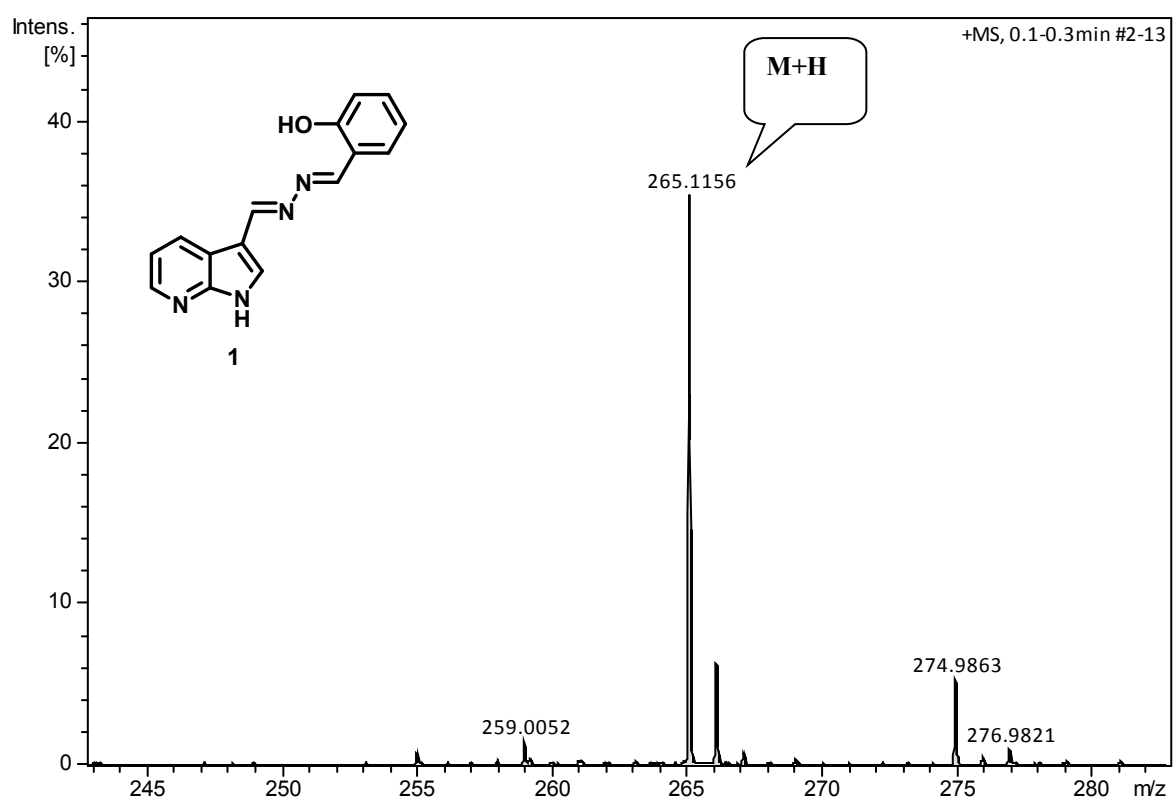
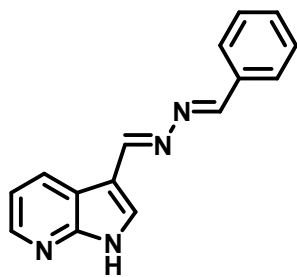


Fig. S41 Mass spectrum of compound 1.



2

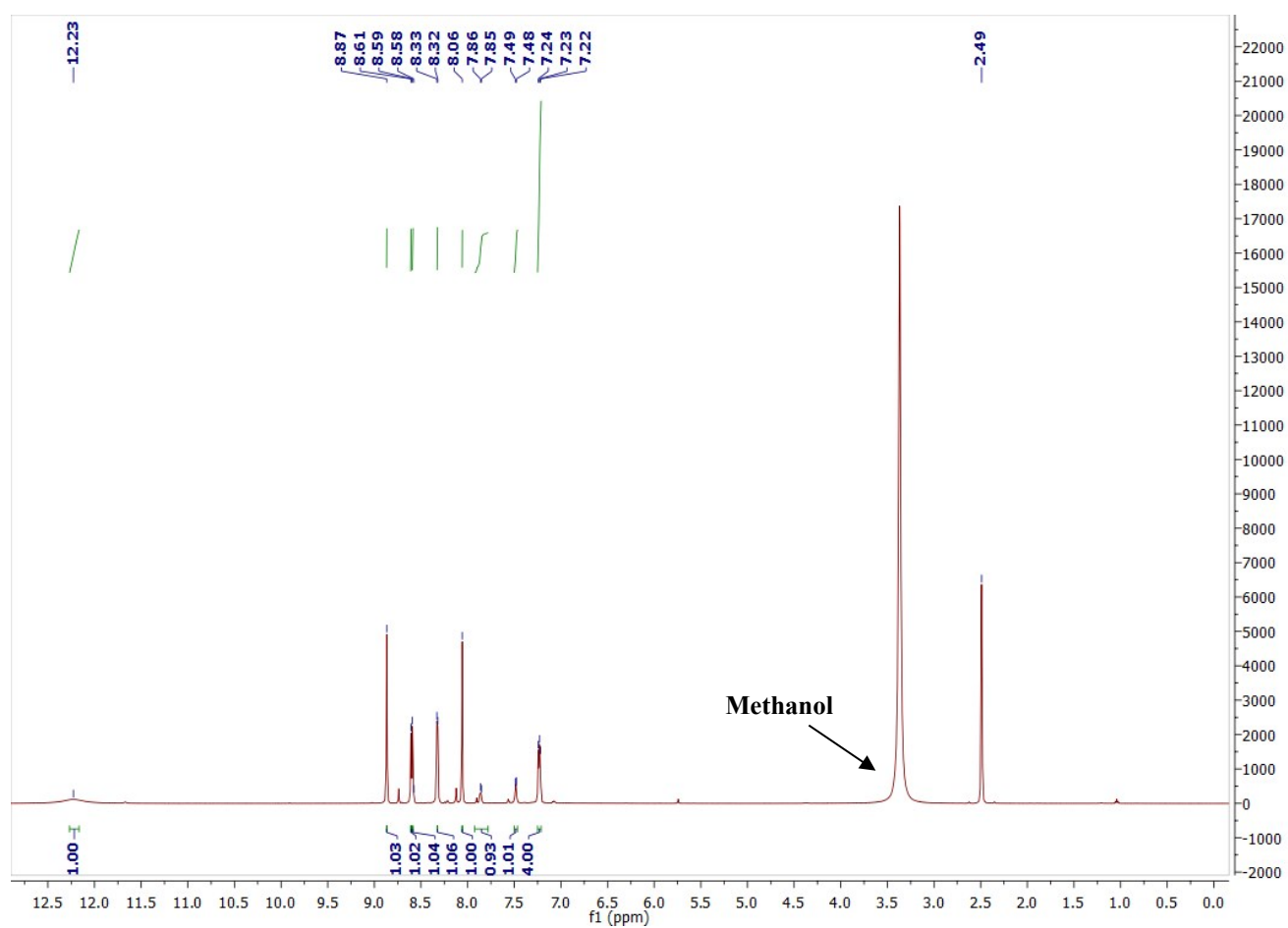
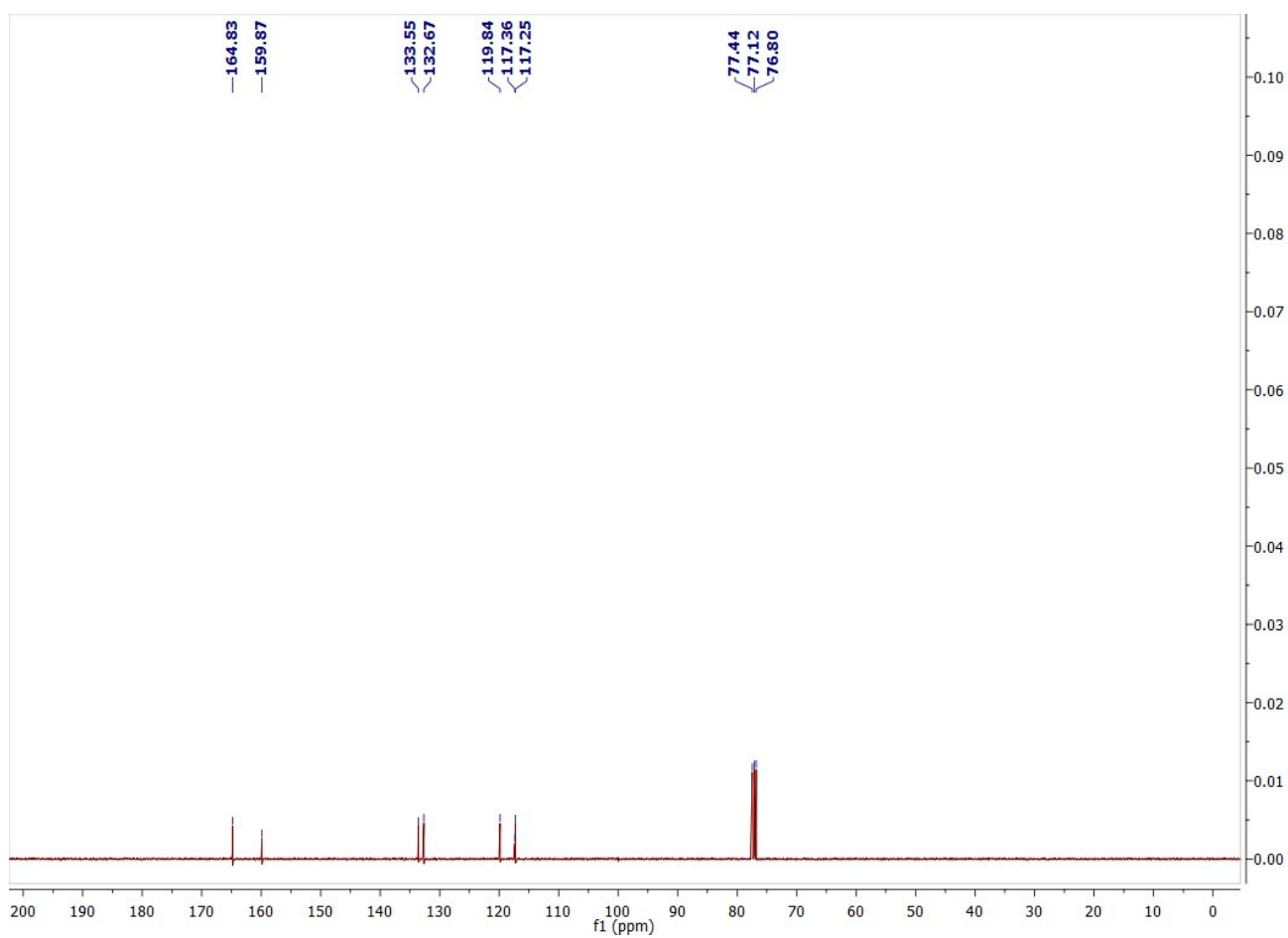
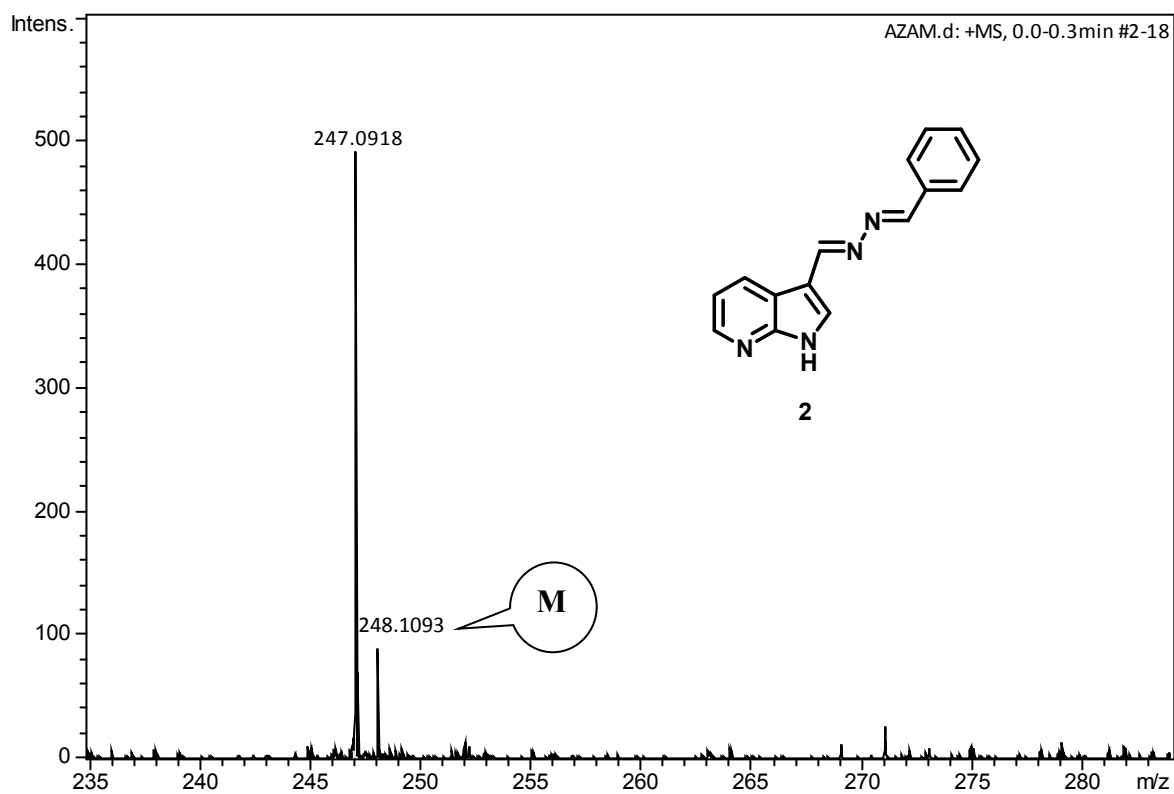


Fig. S42  $^1\text{H}$  NMR spectrum of **2** in  $\text{DMSO-}d_6$ .





**Fig. S43**  $^{13}\text{C}$  NMR spectrum of **2** in  $\text{CDCl}_3$ .



**Fig. S44** Mass spectrum of compound **2**.

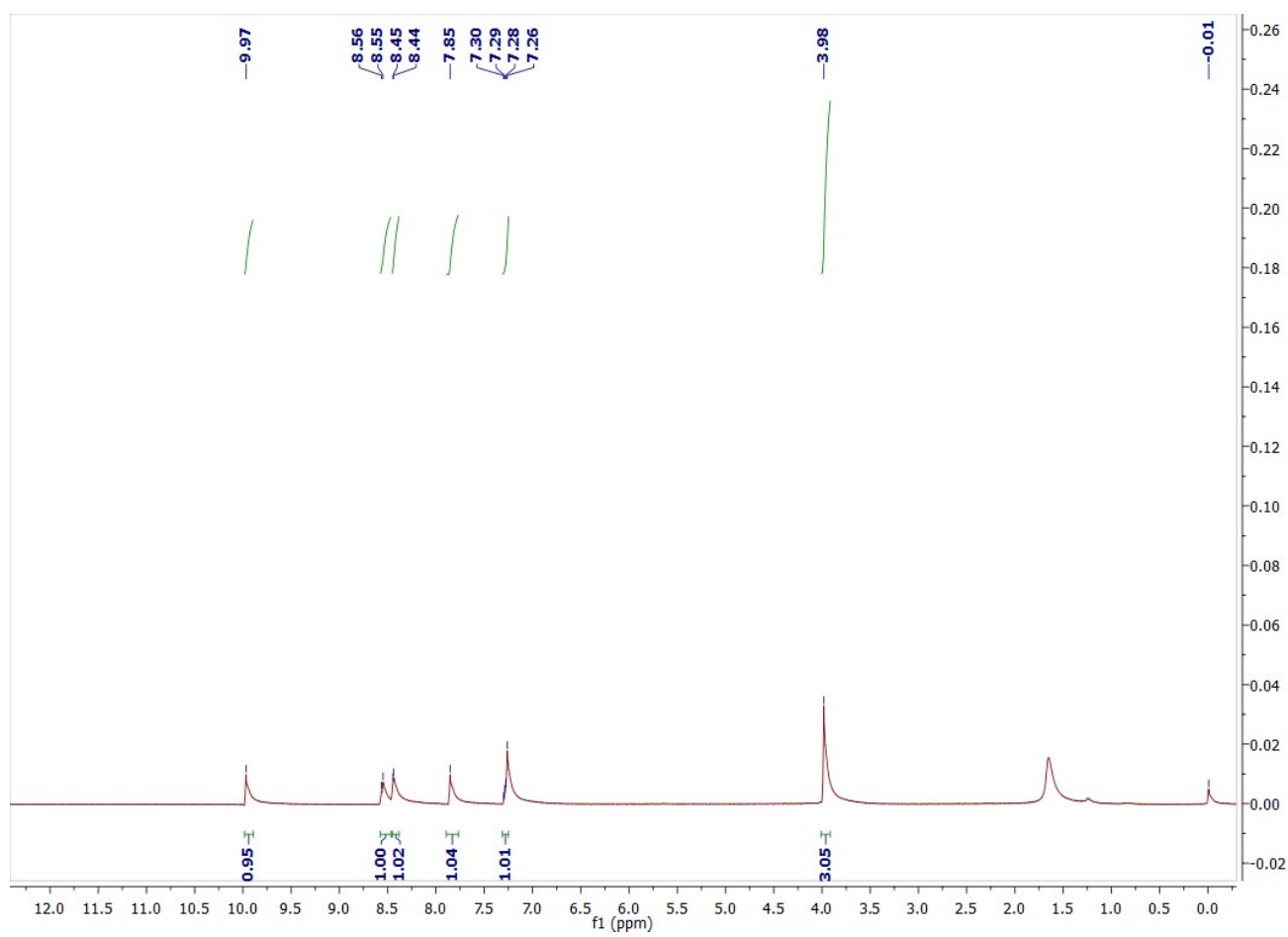
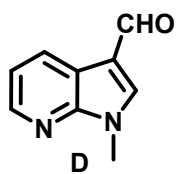


Fig. S45 <sup>1</sup>H NMR spectrum of **D** in CDCl<sub>3</sub>.

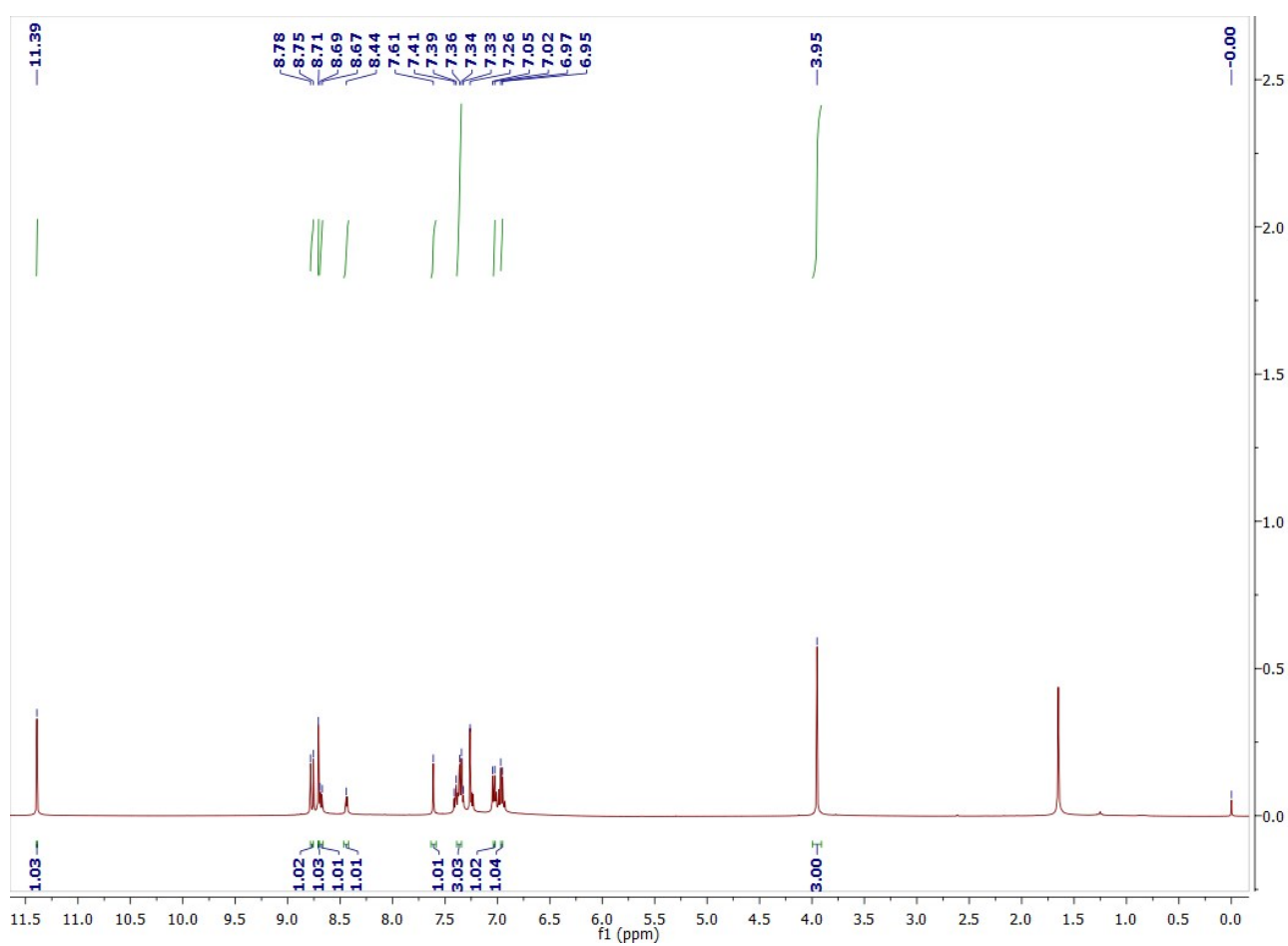
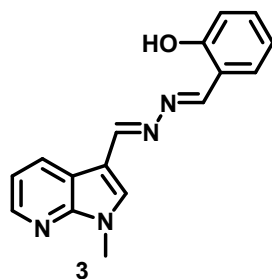
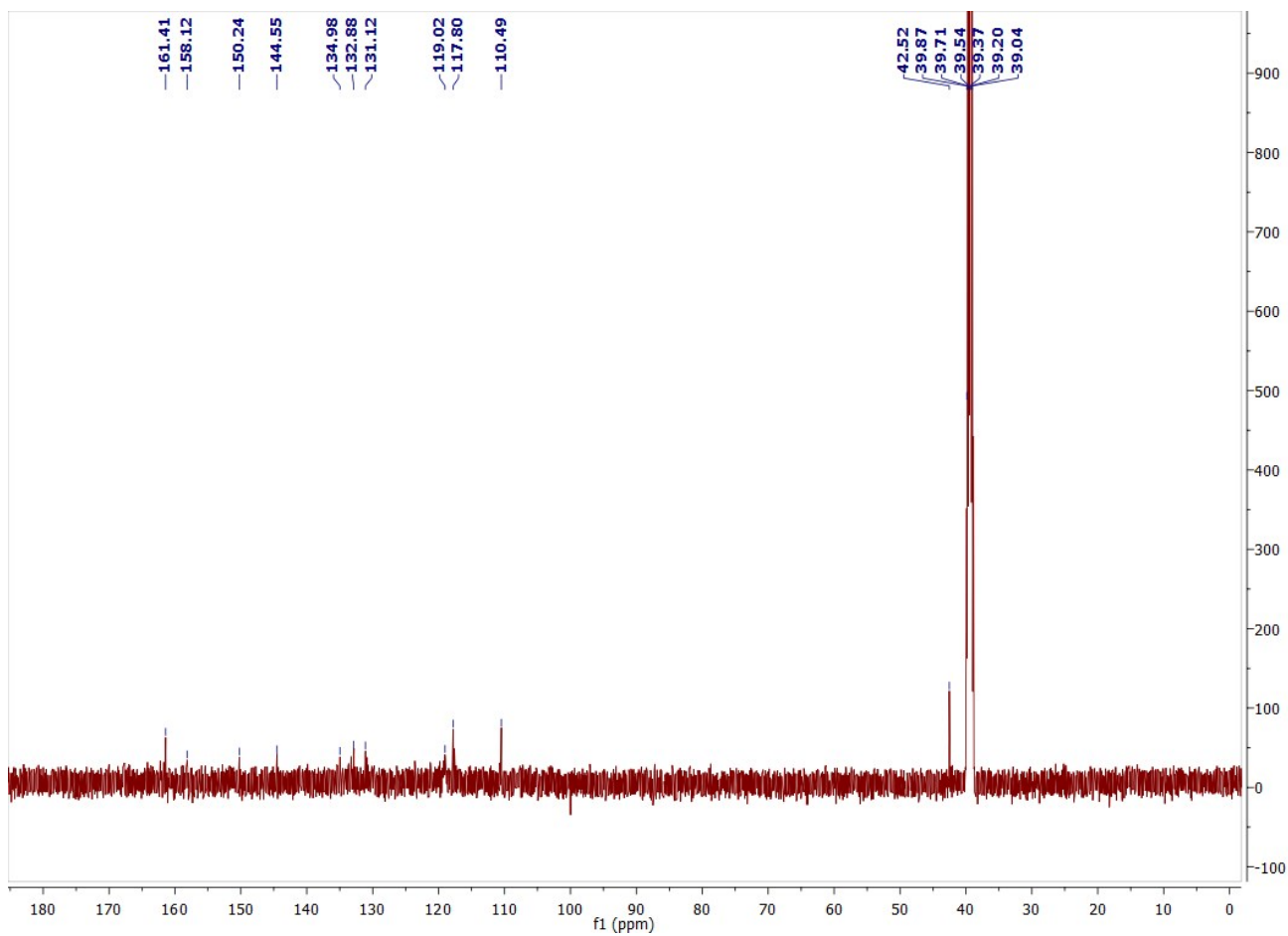
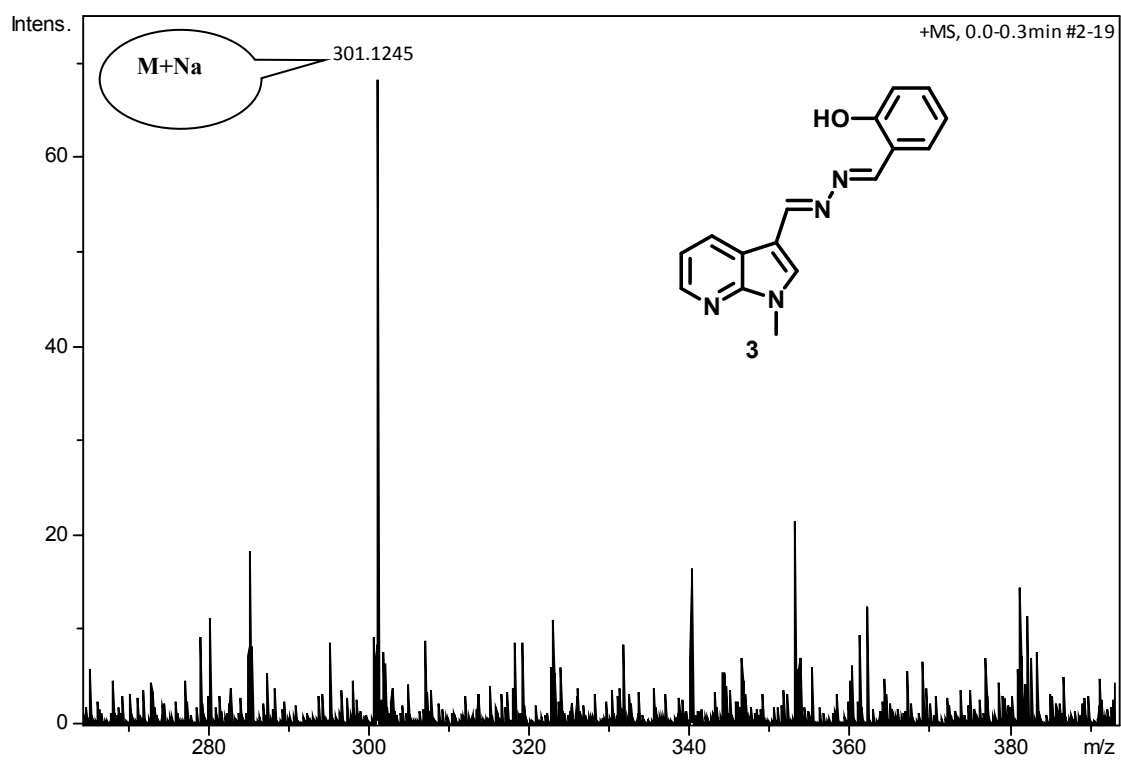


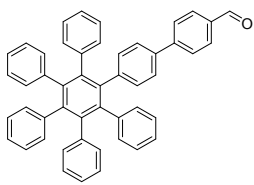
Fig. S46  $^1\text{H}$  NMR spectrum of **3** in  $\text{CDCl}_3$ .



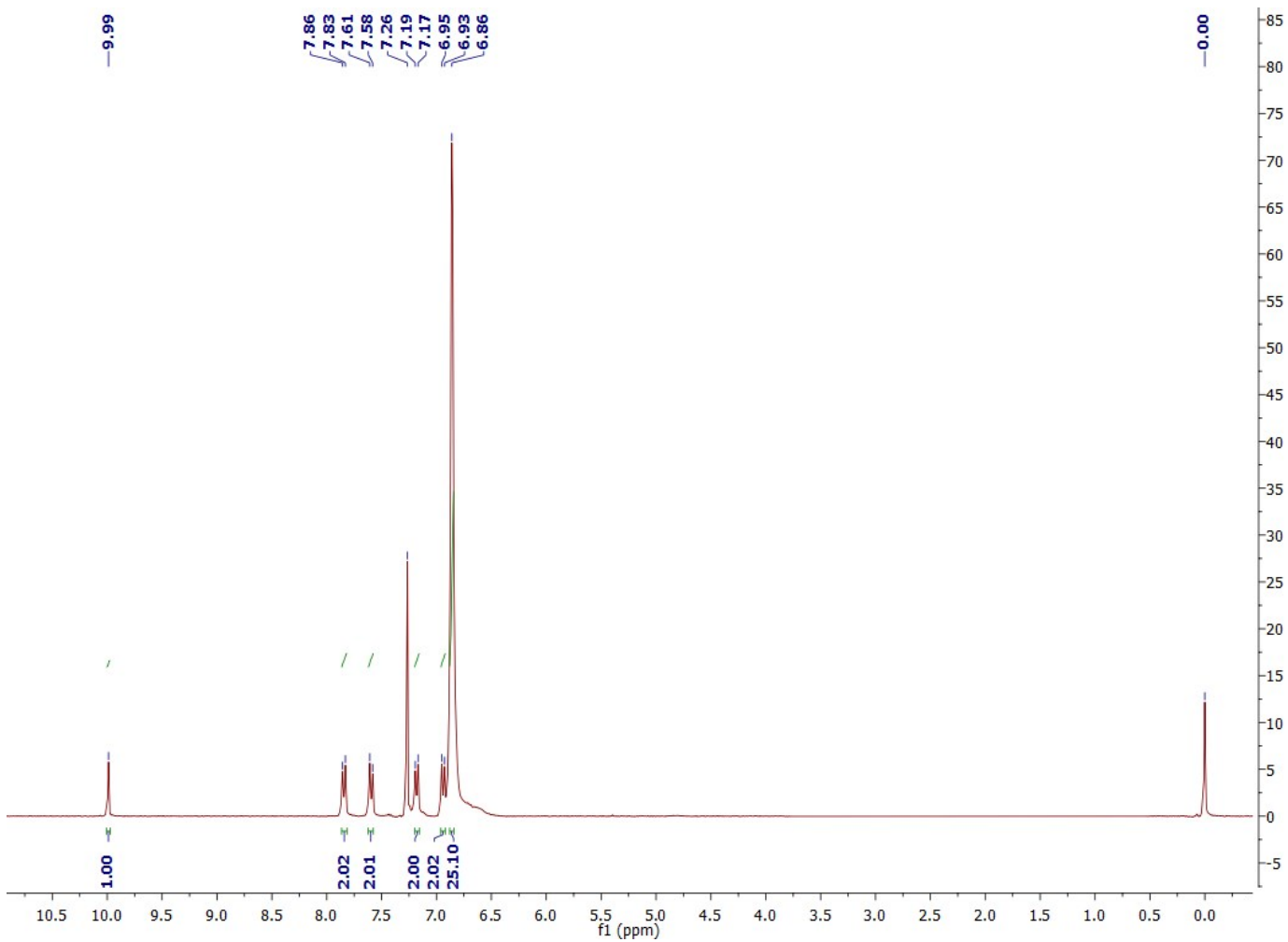
**Fig. S47**  $^{13}\text{C}$  NMR spectrum of **3** in  $\text{DMSO-}d_6$ .



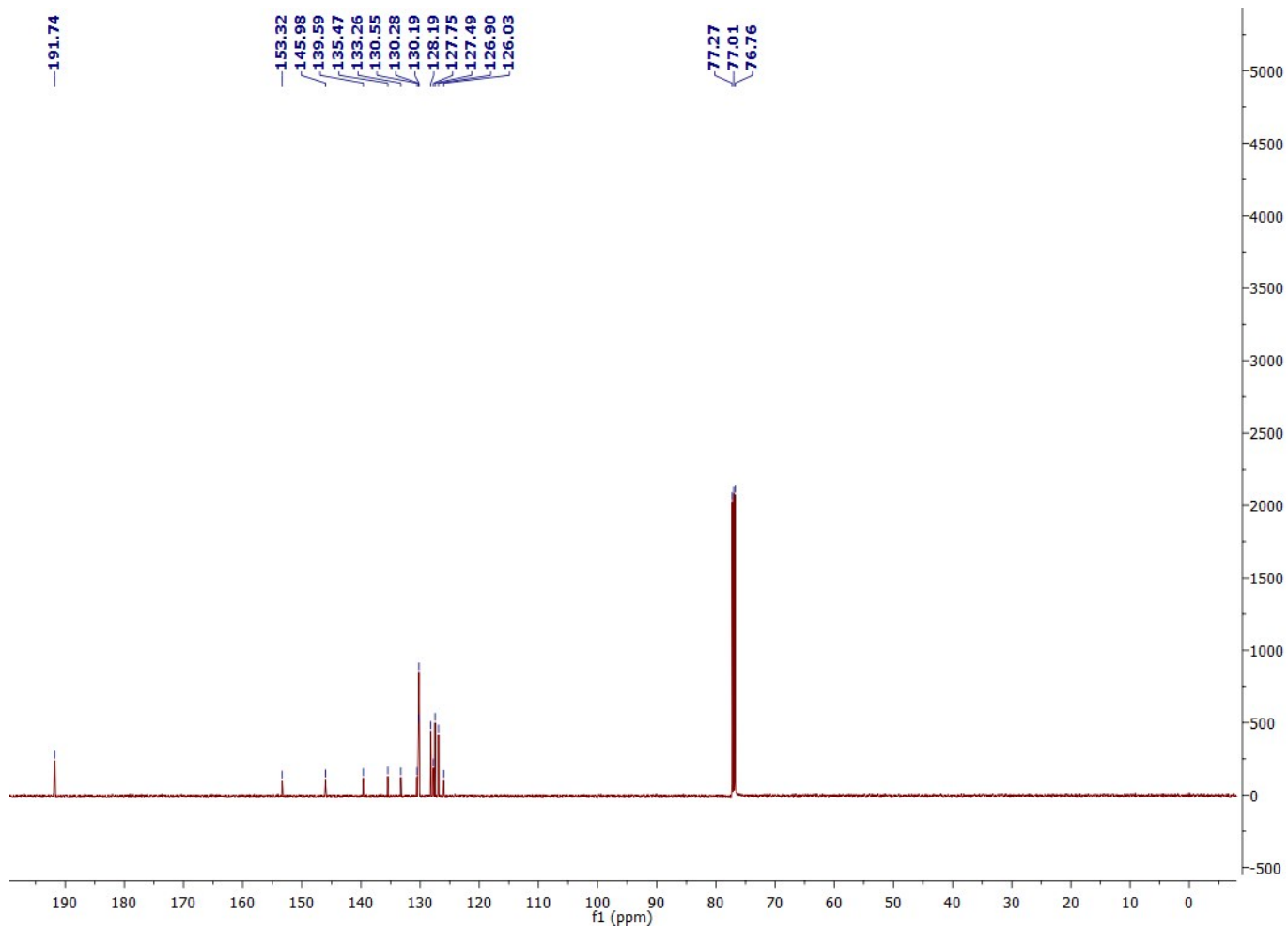
**Fig. S48** Mass spectrum of compound **3**.



**G**

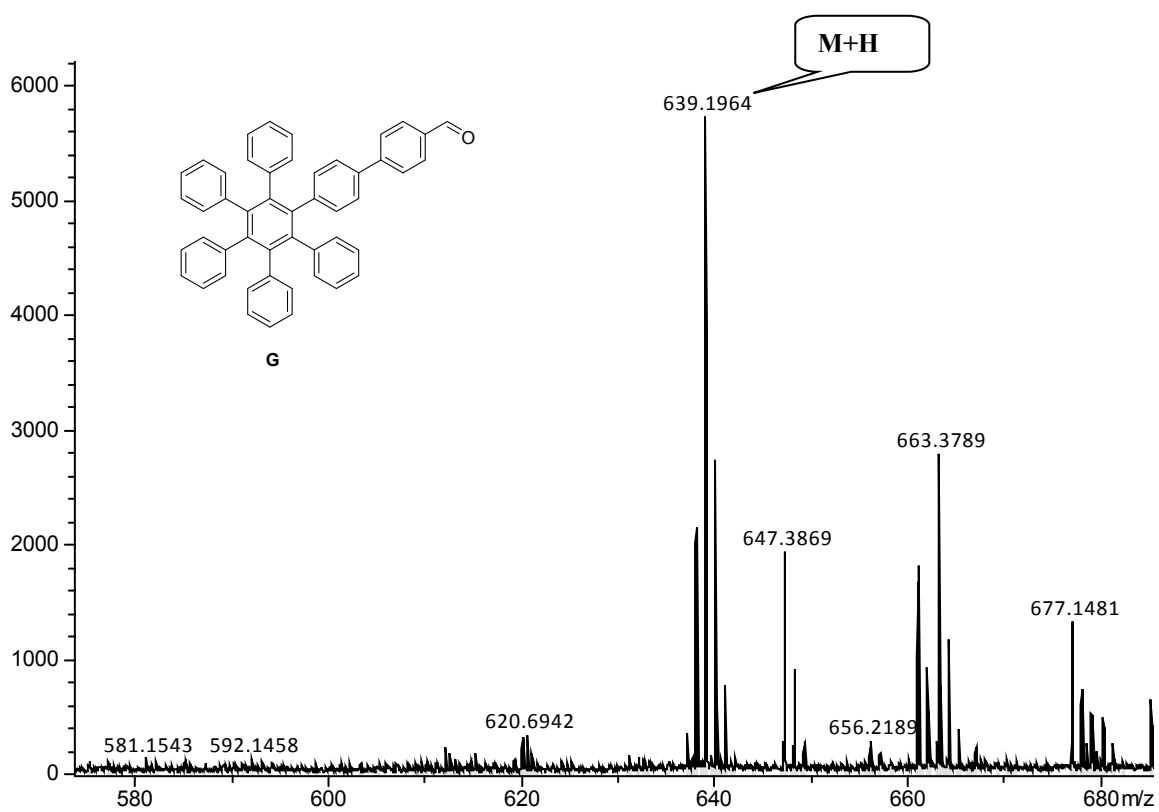


**Fig. S49**  $^1\text{H}$  NMR spectrum of compound **G** in  $\text{CDCl}_3$ .



**Fig. S50**  $^{13}\text{C}$  NMR spectrum of compound G in  $\text{CDCl}_3$ .





**Fig. S51** Mass spectrum of compound **G**.

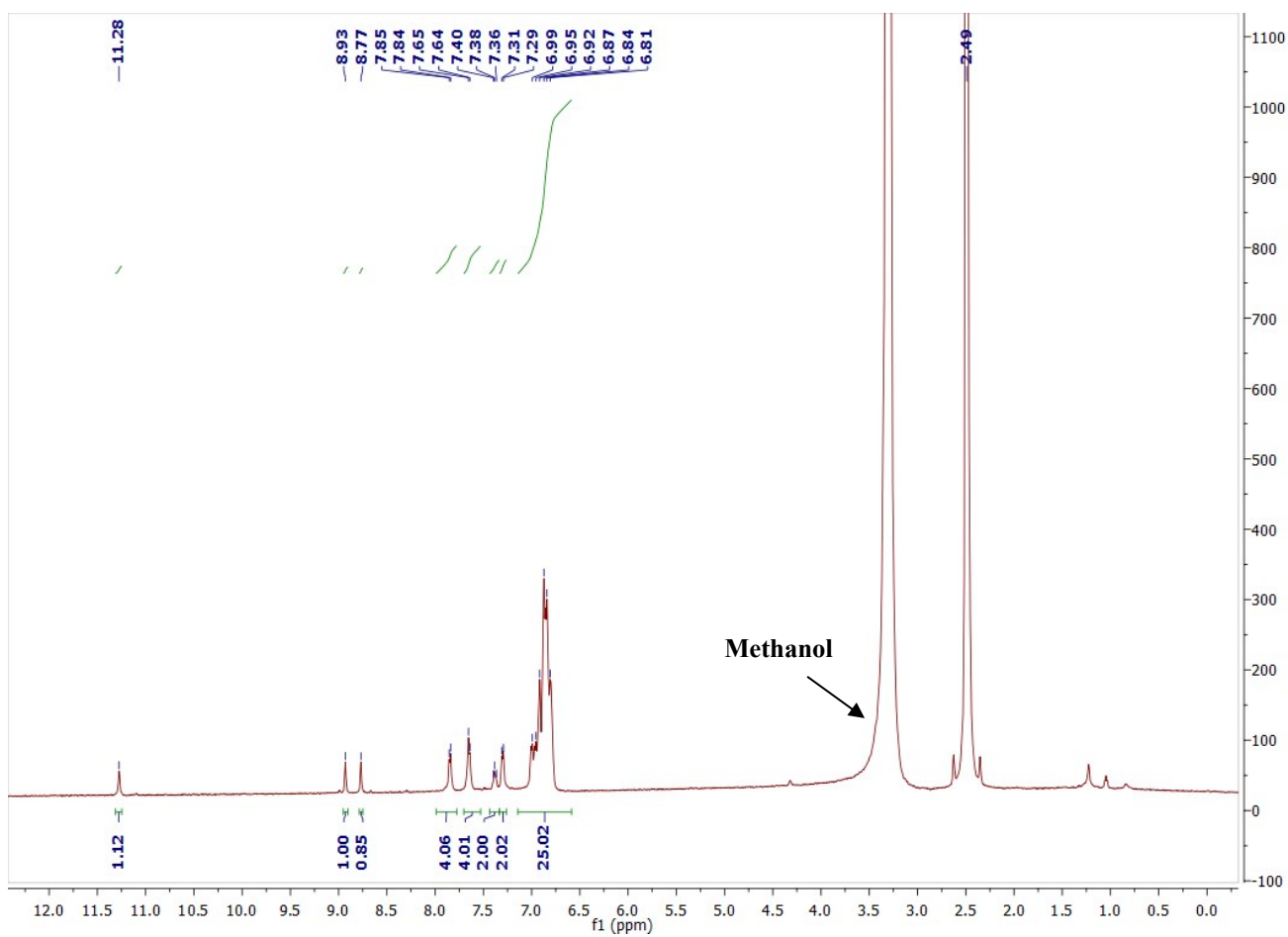
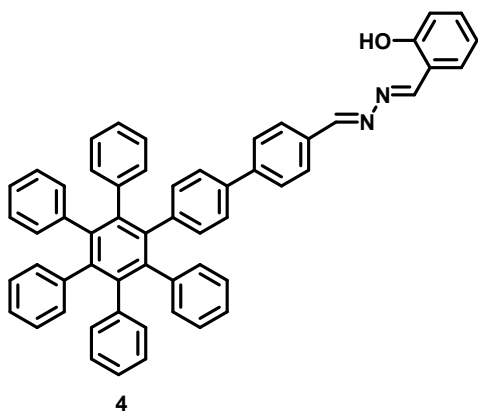


Fig. S52  $^1\text{H}$  NMR spectrum of compound 4 in  $\text{DMSO-}d_6$ .

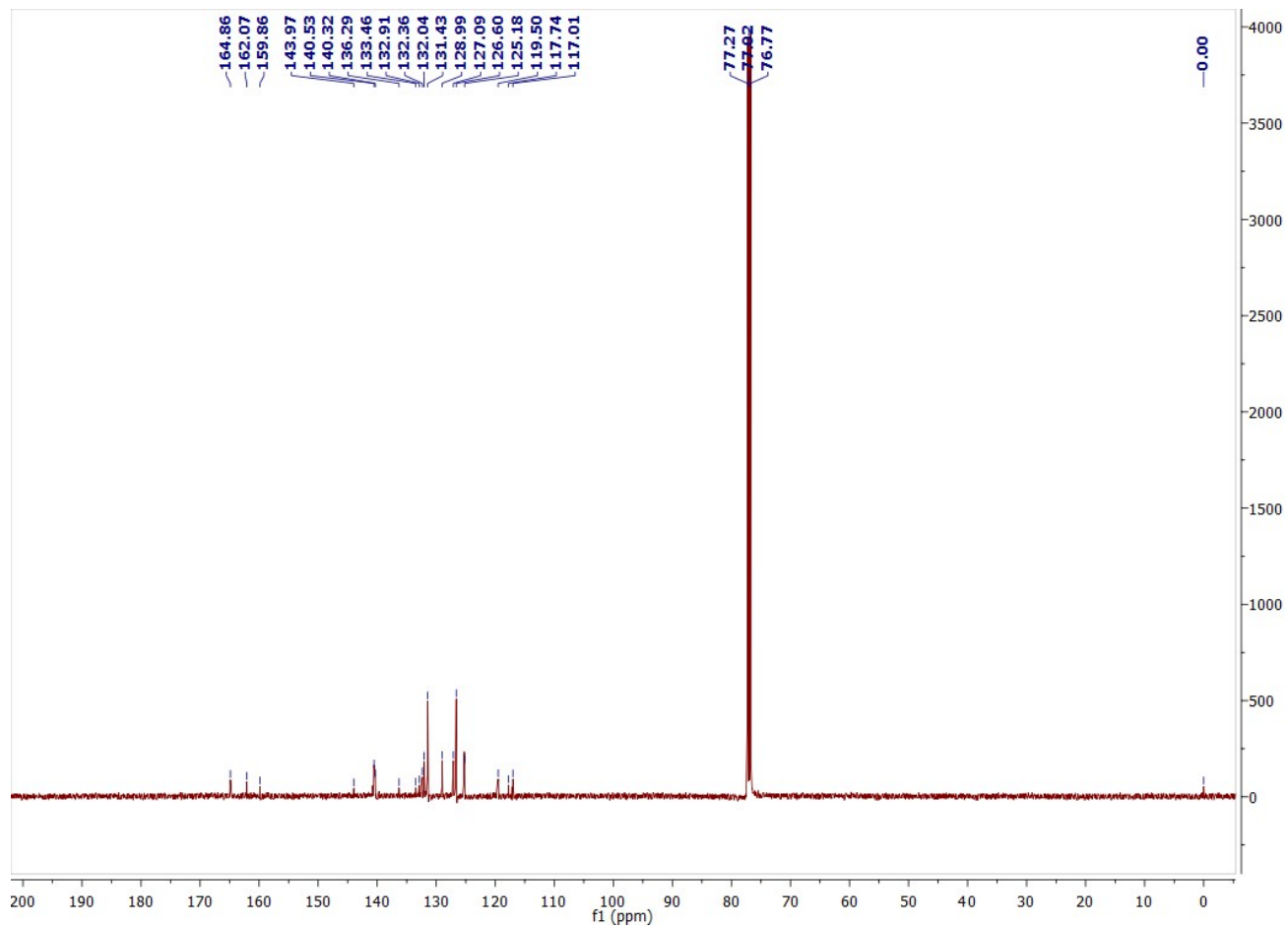
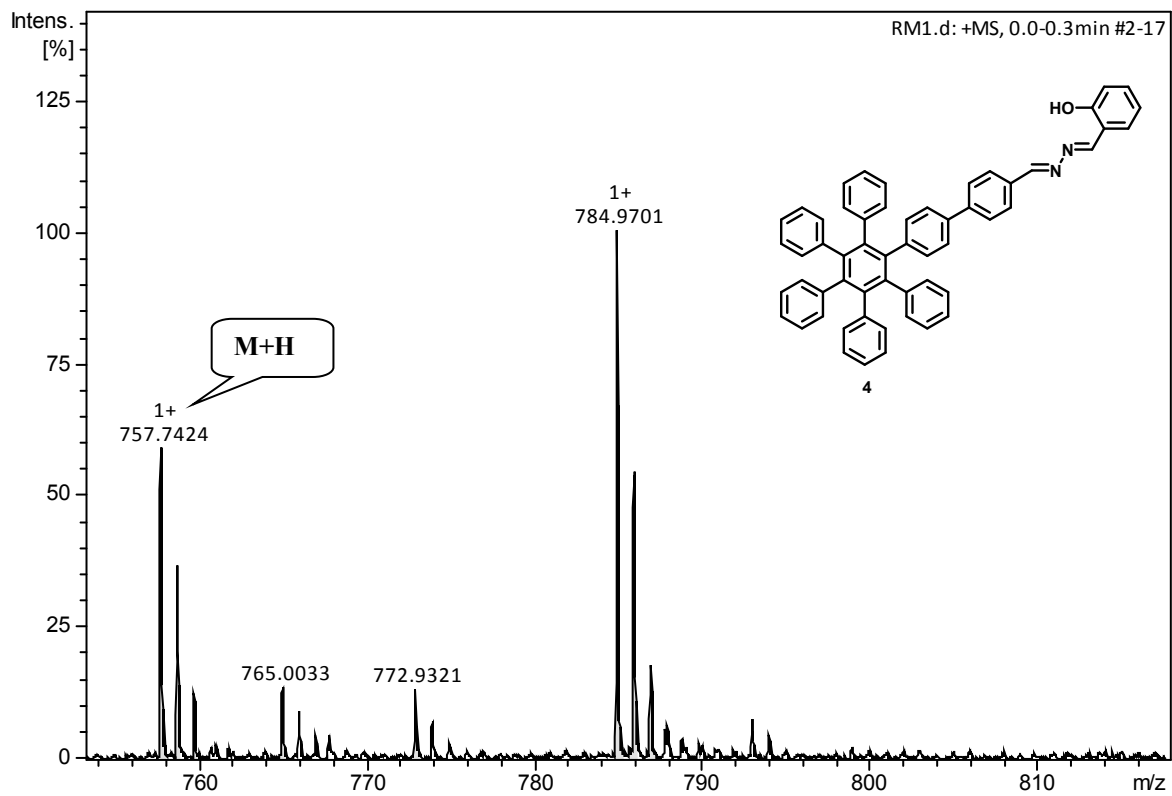


Fig. S53  $^{13}\text{C}$  NMR spectrum of compound 4.



**Fig. S54** Mass spectrum of compound 4.

## References

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2. H. Fei, J. Yu, Y. Jiang, H. Guo, J. Cheng, *Org. Biomol. Chem.*, 2013, **11**, 7092.
3. R. Chopra, V. Bhalla, M. Kumar, S. Kaur, *RSC Adv.*, 2015, **5**, 24336.