Electronic Supplementary Information (ESI)

Aggregation-induced emission logic gates based on metal ion sensing of phenanthroline-tetraphenylethene conjugates

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1. Experimental

All commercially available starting materials, reagents and solvents were used as supplied, unless otherwise stated, and were purchased from Aladdin, Acros Organics and Puyang Huicheng Chemical Co. Ltd. All reactions were carried out under a dry nitrogen atmosphere unless water was used as a solvent or reagent and the temperatures were measured externally. THF was dried using sodium wire and benzophenone indicator. Reported yields are isolated yields. Purification of most intermediates and all final products was accomplished in most cases by gravity column chromatography, using silica gel. For qualitative purity tests of all intermediates and final products, a single spot (visualised using UV-light at 254 nm and 365 nm) was obtained. Elemental analysis was used for quantitative purity checks of all final products. ^{1}H NMR spectra are reported in parts per million (PPM) relative to tetramethylsilane as an internal standard.
KCl, NaCl, $AlCl_3$, $MgCl_2$, $CdCl_2$, $AgNO_3$, $InCl_3$, $PbCl_2$, $CaCl_2$, $Zn(CH_3COO)_2$, $FeCl_2$, $FeCl_3$, CuCN, $CuCl_2$, $SnCl_2$ and LiCl $NaNO_3$, $NaNO_2$, CH_3COONa, Na_2SO_4 were of analytical grade.

(a) INSTRUMENTATION

UV-VIS: Shimadzu UV-VIS-NIR Spectrophotometer (UV-3600)
PL: Edinburgh instruments (FLSP920 spectrometers)
^{1}HNMR: (Bruker AV400).
Mass Spectrometry: Agilent (1100 LC/MSD Trap), MALDI-TOF
Elemental Analysis: Elementar (Vario Micro-cube).

(b) SYNTHESIS

Synthesis of 3-bromo-1,10-phenanthroline（1）and 3,8-dibromo-1,10-phenanthroline（2）
1 and 2 were synthesis by one reaction in the following procedure:
A solution of 1, 10-phenanthroline (5.00g, 21.3mmol) in nitrobenzene (10mL) was heated to 130-140℃ in a 100mL 3-neck flask. Bromine (6.82g, 42.6mmol in 5mL nitrobenzene) was added drop-wise over a period of 1h. Upon the addition of bromine, 1, 10-phenanthroline was added into the reacting solution. After stirring for 3h at the same temperature, the reaction mixture was cooled to room temperature, treated with concentrated ammonium hydroxide (50mL) and extracted with DCM (3×25mL). The combined organic layers were washed with water (3×25mL), dried ($MgSO_4$) and filtered. Concentration in vacuum afforded a suspension of the products in nitrobenzene. The nitrobenzene was removed by dissolving the suspension in DCM (5mL). The mixture was purified by column chromatography using DCM: EA=1:1 as eluent. **1** and **2** were obtained successfully with yield 42% and 35%.
^{1}H-NMR (400MHz, $CDCl_3$, ppm) for **1**: δ9.19 (d, J=2.4Hz, 2H), δ8.42 (d, J=2Hz, 1H), δ8.28(t, J=2Hz, 1H), δ7.86(d, J=9.2Hz, 1H), δ7.74(d, J=8.8Hz, 1H), δ7.69(m, 1H)
ESI (+)-MS: Calcd for $C_{12}H_7BrN_2$:259.10 [M], found 258.8 and 260.7 $[M+H]^+$
^{1}H-NMR (400MHz, $CDCl_3$, ppm) for **2**: δ9.19 (d, J=2.4Hz, 2H), δ 8.42(d, J=2.4Hz, 2H), δ7.77(s, 2H)
ESI (+)-MS: Calcd for $C_{12}H_6Br_2N_2$:338 [M], found 338.9 $[M+H]^+$

1-(4-Bromophenyl)-1, 2, 2-triphenylethylene and TPE boronic acid

The synthesis procedure could be found in our former research work.[14, 15]

Phen-1TPE (3-tetraphenylethene-1, 10-phenanthroline) (4)

In a 50mL 2-neck flask, 1-(4-Bromophenyl)-1, 2, 2-triphenylethylene (0.45g 1.2mmol), 3-bromo-1, 10-phenanthroline (0.26g, 1.0mmol), potassium carbonate (0.83g, 6.0mmol) and $Pd(Pph_3)_4$ (0.10g, 0.09mmol) as the catalyst were dissolve in 10mL toluene and 5mL water. The mixture was heated to 90℃, protected under atmosphere of N_2 and stirred for 2 days. When the reaction was finished, the mixture was cooled down to room temperature, washed with water (3×25mL), dried ($MgSO_4$) and filtered. After removing the solvent, the residue was purified by column chromatography using Methanol: DCM=5:95 as eluent, and 0.36g yellow solid was obtained, yield 70.6%.

^{1}H-NMR (400MHz, $CDCl_3$, ppm) for **4**: δ9.41(d, J=2Hz, 1H), δ9.22(d, J=3.2Hz, 1H), δ8.37(d, J=30.8Hz, 1H), δ 8.29(d, J=8Hz, 1H), δ7.83(d, J=1.6Hz, 2H), δ7.67(m, 1H), δ7.56(d, J=8.4Hz, 2H), δ7.0-7.22(m, 17H)

^{13}C-NMR (400MHz, $CDCl_3$, ppm) for **4**: δ123.01, δ126.58, δ126.63, δ126.69, δ126.72, δ126.92, δ127.69, δ127.81, δ127.90, δ128.62, δ131.33, δ131.36, δ131.40, δ132.28, δ133.17, δ136.18, δ140.15, δ141.69, δ143.54, δ143.58, δ144.17, δ149.21, δ150.35

ESI (+)-MS: Calcd for $C_{38}H_{26}N_2$:510.63 [M], found 511.3 $[M+H]^+$

Elemental Analysis: Anal calcd for $C_{38}H_{26}N_2$ C, 89.38; H, 5.13; N, 5.49 Found: C, 89.26; H, 5.16; N, 5.54

Phen-2TPE (3, 8-ditetraphenylethene-1, 10-phenanthroline) (5)

In a 25mL 2-neck flask, 1-(4-Bromophenyl)-1, 2, 2-triphenylethylene (0.24g 0.65mmol), 3, 8 dibromo-1, 10-phenanthroline (0.11g, 0.32mmol), potassium carbonate (0.27g, 2.0mmol) and $Pd(Pph_3)_4$ (0.1g, 0.09mmol) as the catalyst were dissolved in 5mL toluene and 2.5mL water. The mixture was heated to 90℃, protected under atmosphere of N_2 and stirred for 2 days. When the reaction was finished, the mixture was cooled down to room temperature, washed with water (3×25mL), dried ($MgSO_4$) and filtered. After removing the solvent, the residue was purified by column chromatography using Methanol: DCM=10:90 as eluent. 0.20g light yellow solid was obtained, yield 74.1%.

^{1}H-NMR (400MHz, $CDCl_3$, ppm) for **5**: δ9.46 (s, 2H), δ8.42(s, 4H), δ7.88(s, 2H), δ7.56 (d, J=8Hz, 4H), δ7.00-7.23(m, 34H)

^{13}C-NMR (400MHz, $CDCl_3$, ppm) for **5**: δ126.59, δ126.65, δ126.68, δ126.74, δ127.12, δ127.69, δ127.82, δ127.91, δ128.53, δ131.33, δ131.37, δ131.41, δ132.31, δ133.36, δ135.03, δ135.45, δ140.13, δ141.73, δ143.54, δ143.58, δ144.24, δ149.20

ESI (+)-MS: Calcd for $C_{64}H_{44}N_2$:841.05, found 841.6

Elemental Analysis: Anal calcd for C, 91.40; H, 5.27; N, 3.33 Found: C, 91.35; H, 5.32; N, 3.36

(c)Preparation of the solutions

Both Phen-1TPE and Phen-2TPE are dissolved in distilled THF with concentration equals to 10^{-3}mol/L. To a 2mL solution with different f_w, 20μL of the above solutions are added by a microsyringe (V_{max} = 25μL). In this way, Phen-1TPE and Phen-2TPE solutions with f_w (0%- 95 %) and concentration equal to 10^{-5}mol/L are made.

Metal ion solutions are prepared by dissolving 0.1mmol the metal salts in 10ml distilled water to obtain solution with concentration of 10^{-2}mol/L, and then diluted to 2×10^{-3}mol/L. The amount of metal salts is used as below:

1.$CuCl_2$ 17mg, 2.$AgNO_3$ 17mg, 3.$InCl_3$ 29mg, 4.$CaCl_2$ 11mg, 5.$SnCl_2$ 23mg, 6.LiBr 9mg, 7.$FeCl_3$ 16mg, 8.$AlCl_3$ 13mg, 9.KCl 7mg, 10.$CdCl_2$ 23mg, 11.$PbCl_2$ 28mg, 12.$Zn(OAc)_2$ 22mg, 13.CuCN 9mg, 14.NaCl 6mg, 15.$FeCl_2$ 13mg, 16.$MgSO_4$ 12mg,

17.$NaNO_3$ 8mg, 18.$NaNO_2$ 7mg, 19.NaCl 6mg, 20.Na_2SO_4 14mg, 21.NaOAc 8mg

During the titration experiment, metal ions are added into gradually with a fixed interval of 2min.

For titration experiments in THF, Phen-1TPE is prepared with concentration equals to 10^{-4}mol/L and Phen-2TPE 5×10^{-5}mol/L. Metal salts including $Zn(CH_3COO)_2$, $CdCl_2$, $InCl_3$, and $SnCl_2$ are dissolved in THF: Methanol = 25:1(v:v) with concentration equals to 2×10^{-3}mol/L. The titration experiment is similar to the former experiment.

2. Fluorescent and UV-Vis spectra of Phen-1TPE and Phen-2TPE titration with metal ions

2.1(a) Fluorescent and UV-vis spectra of Phen-1TPE in f_w = 90 % (2 mL, 10^{-5} mol/L) titrating with different amount of metal aqueous solution ($[M^{n+}] = 2\times10^{-3}$ mol/L)

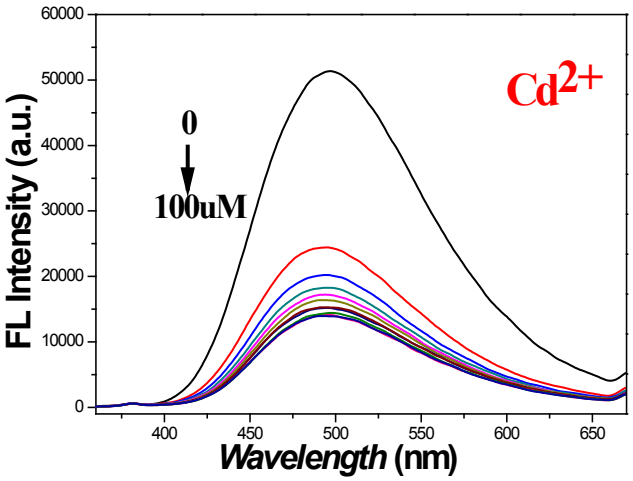


Fig.S1. Fluorescent spectra titrating with [Cd^{2+}]

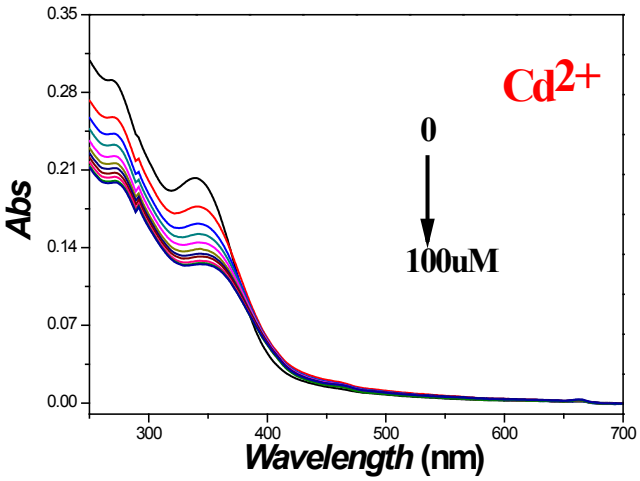


Fig.S2. UV-vis spectra titrating with [Cd^{2+}]

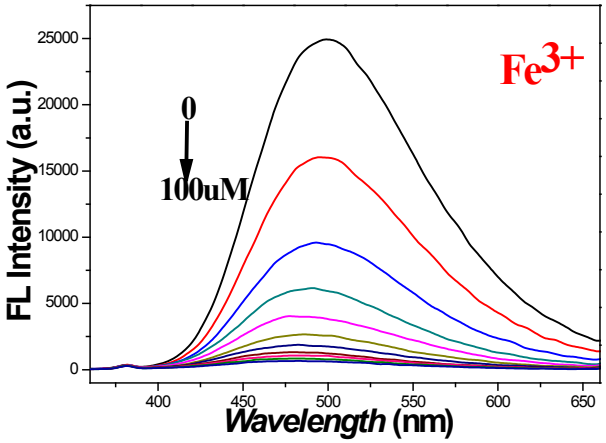


Fig.S3. Fluorescent spectra titrating with [Fe^{3+}]

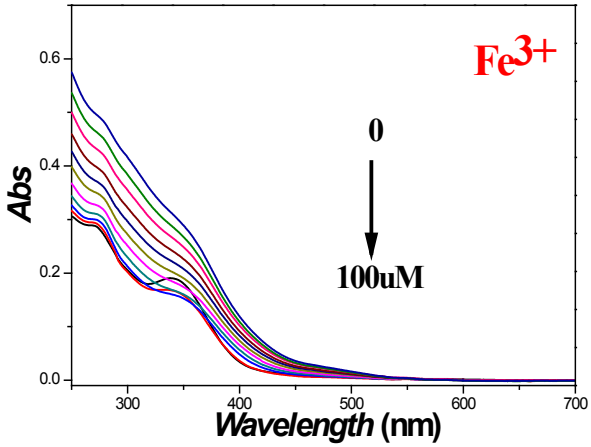


Fig.S4. UV-vis spectra titrating with [Fe^{3+}]

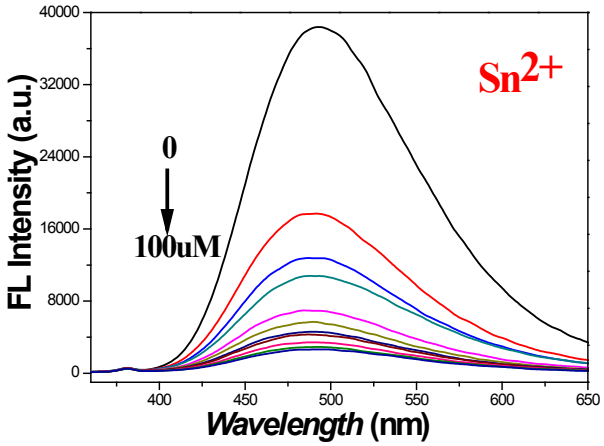


Fig.S5. Fluorescent spectra titrating with [Sn^{2+}]

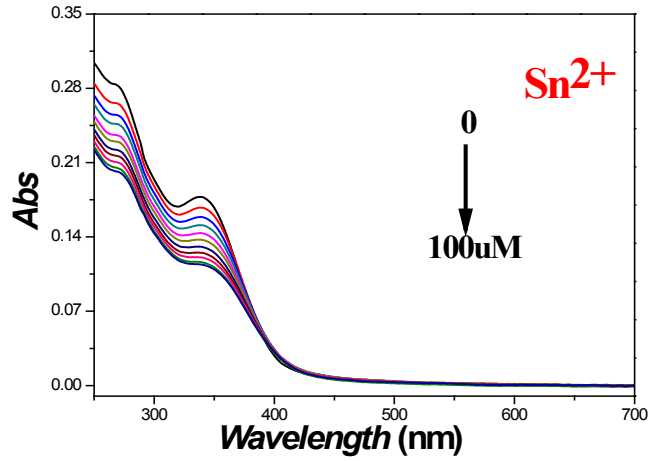


Fig.S6. UV-vis spectra titrating with [Sn^{2+}]

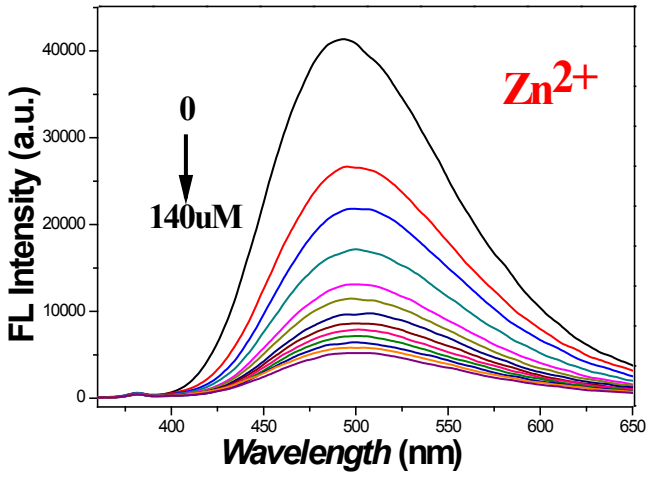


Fig.S7. Fluorescent spectra titrating with [Zn^{2+}]

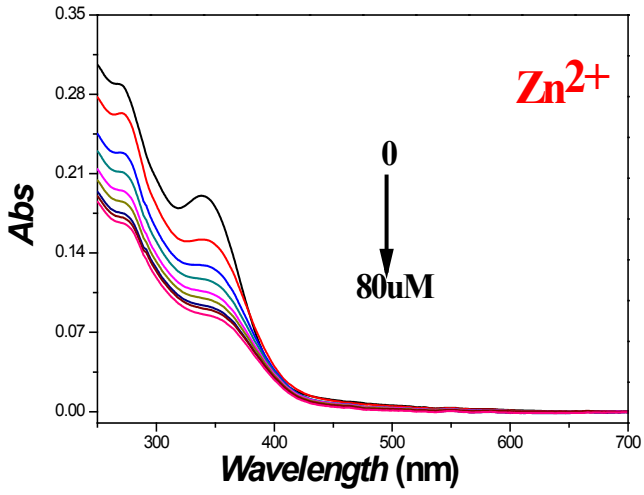


Fig.S8. UV-vis spectra titrating with [Zn^{2+}]

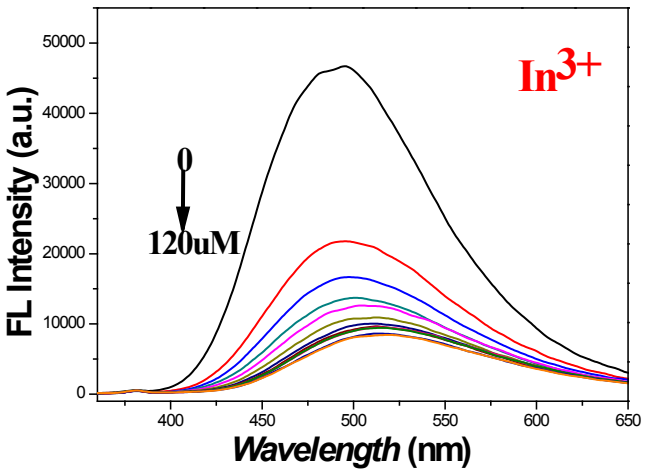


Fig.S9. Fluorescent spectra titrating with [In^{3+}]

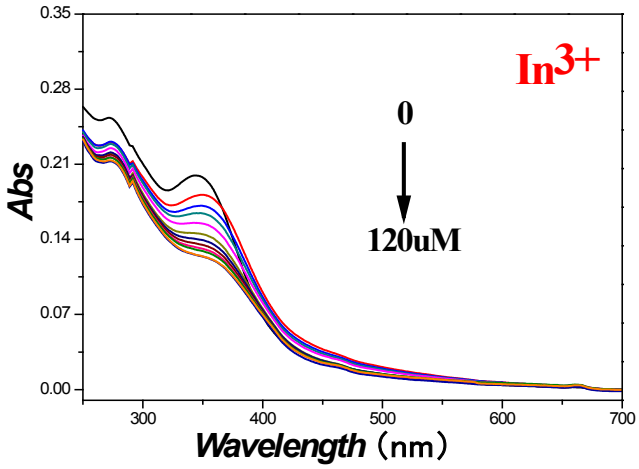


Fig.S10. UV-vis spectra titrating with [In^{3+}]

2.1(b) Fluorescent and UV-vis spectra of Phen-1TPE (2mL, 10^{-4}mol/L) titrating with different metal ions in THF

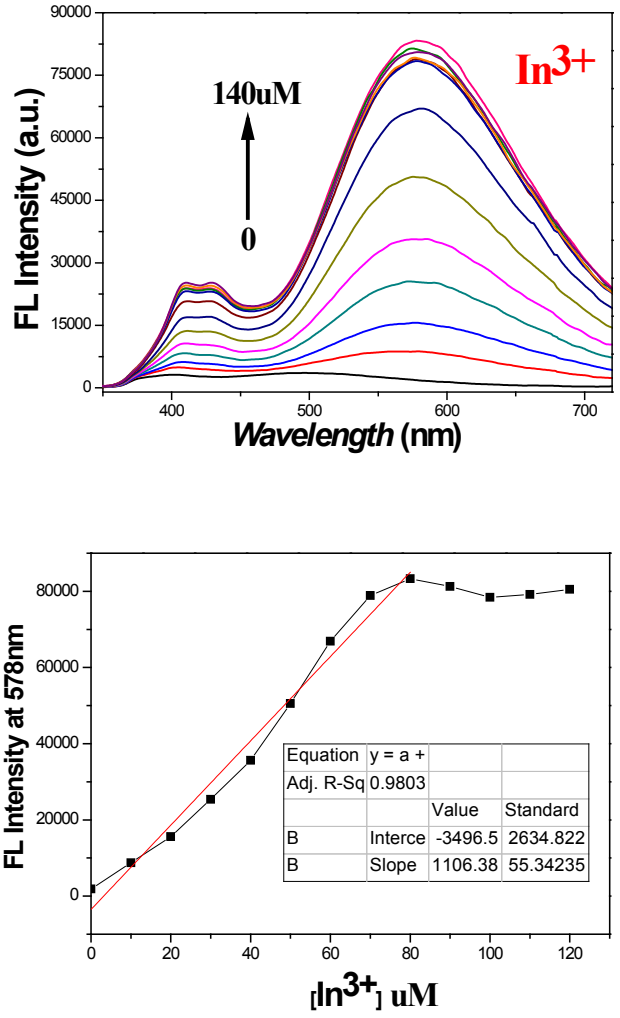


Fig.S11. Fluorescent spectra titrating with [In^{3+}]

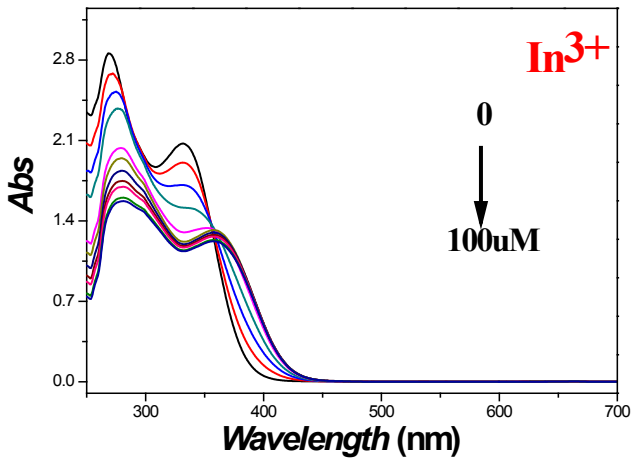


Fig.S12. UV-vis spectra titrating with [In^{3+}]

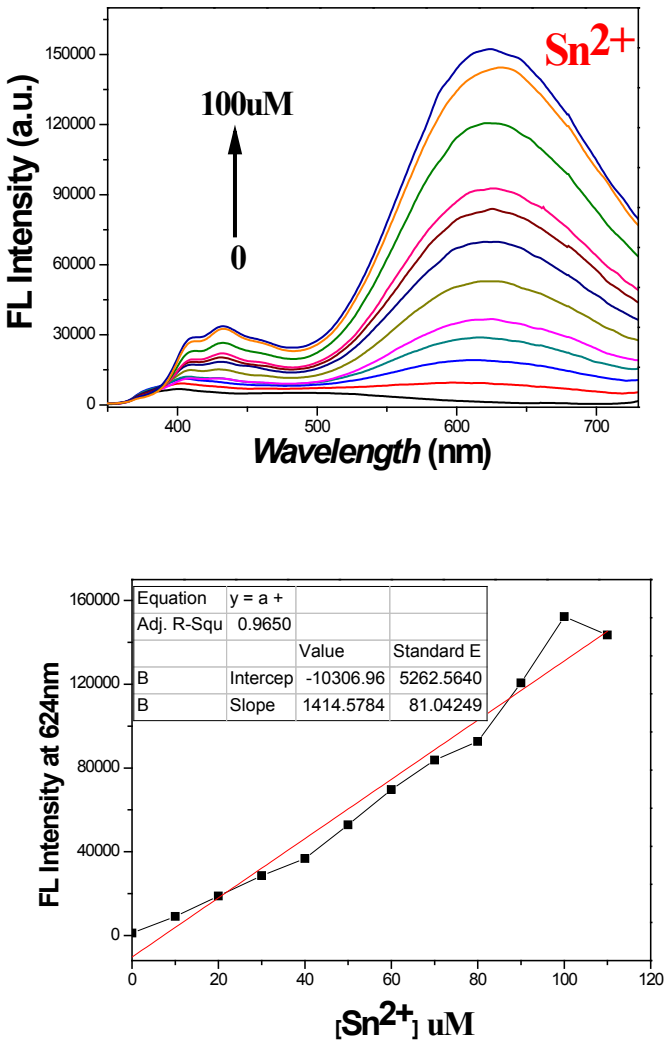


Fig.S13. Fluorescent spectra titrating with [Sn^{2+}]

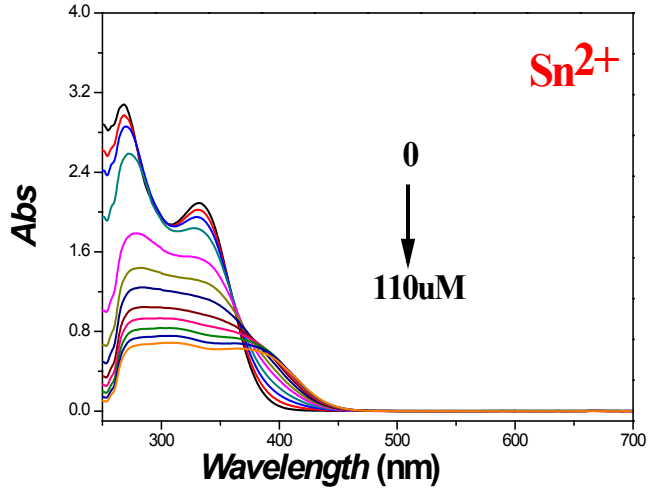
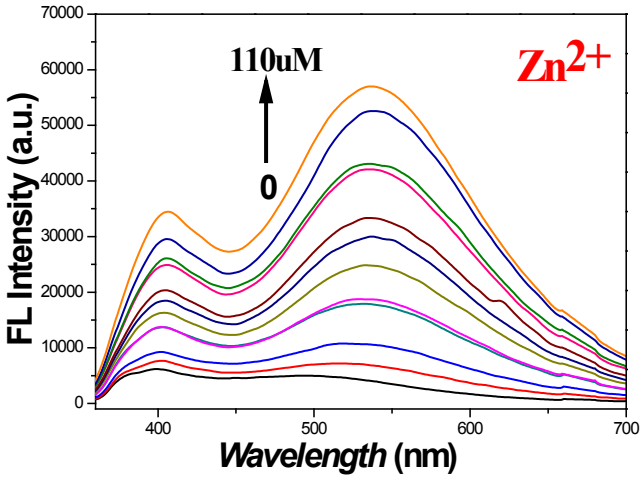


Fig.S14. UV-vis spectra titrating with [Sn^{2+}]



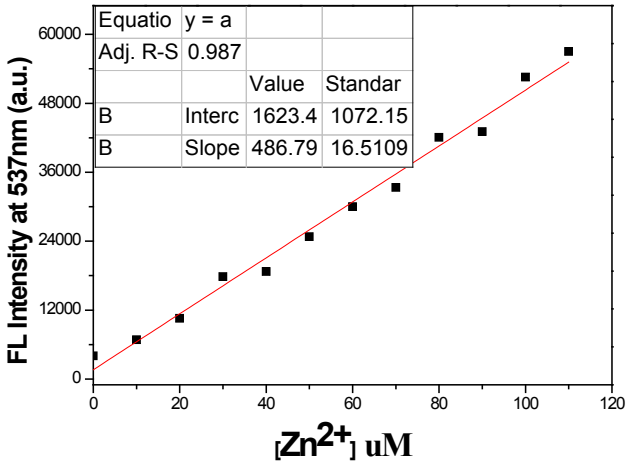


Fig.S15. Fluorescent spectra titrating with [Zn^{2+}]

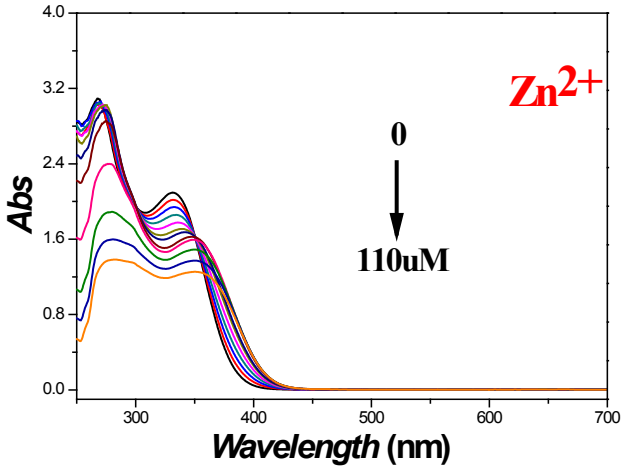


Fig.S16. UV-vis spectra titrating with [Zn^{2+}]

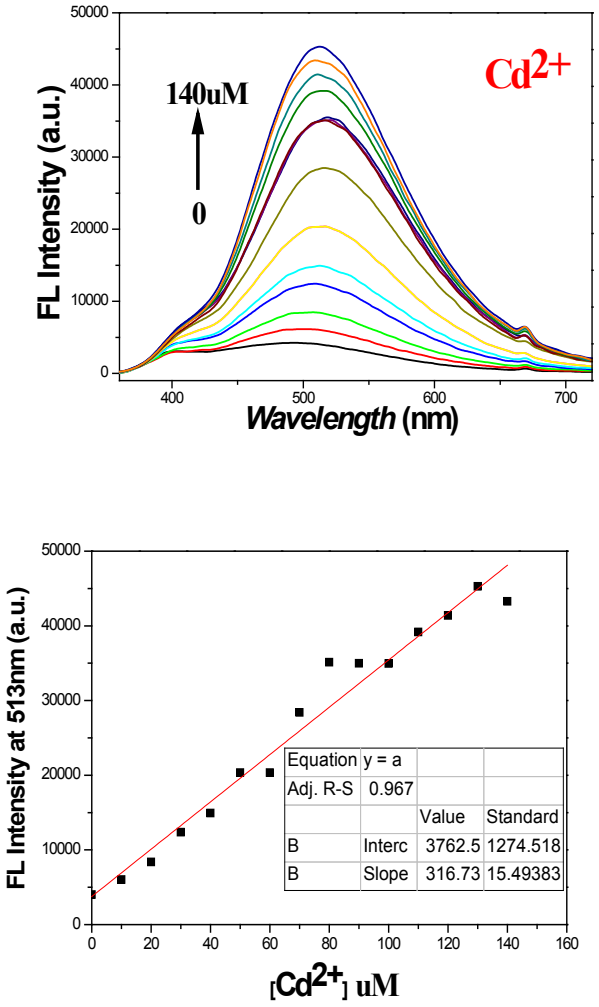


Fig.S17. Fluorescent spectra titrating with [Cd^{2+}]

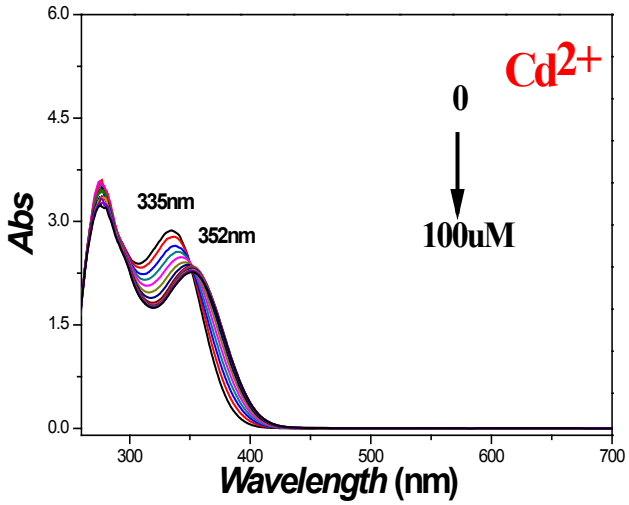


Fig.S18. UV-vis spectra titrating with [Cd^{2+}]

2.2(a) Fluorescent and UV-vis spectra of Phen-2TPE in f_w = 90 % (2mL, 10^{-5}mol/L) titrating with different amount of metal aqueous solution ([M^{n+}] = 2×10^{-3}mol/L)

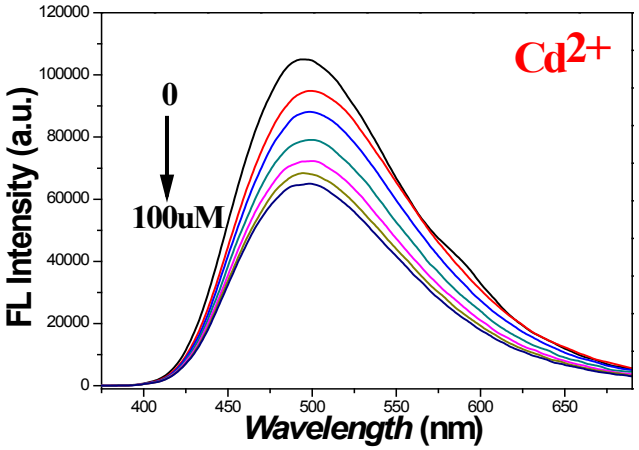


Fig.S19. Fluorescent spectra titrating with [Cd^{2+}]

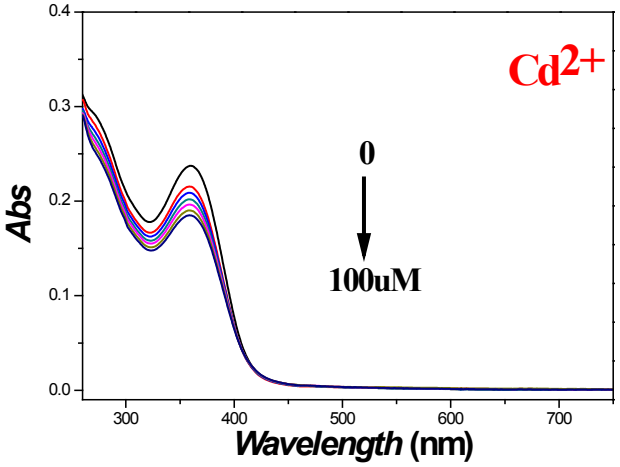


Fig.S20. UV-vis spectra titrating with [Cd^{2+}]

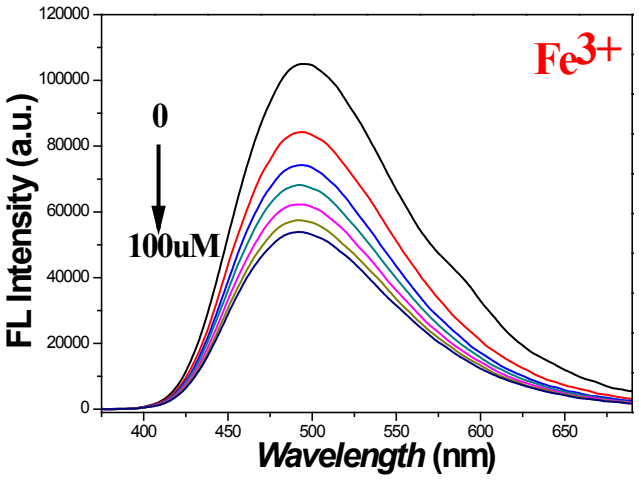


Fig.S21. Fluorescent spectra titrating with [Fe^{3+}]

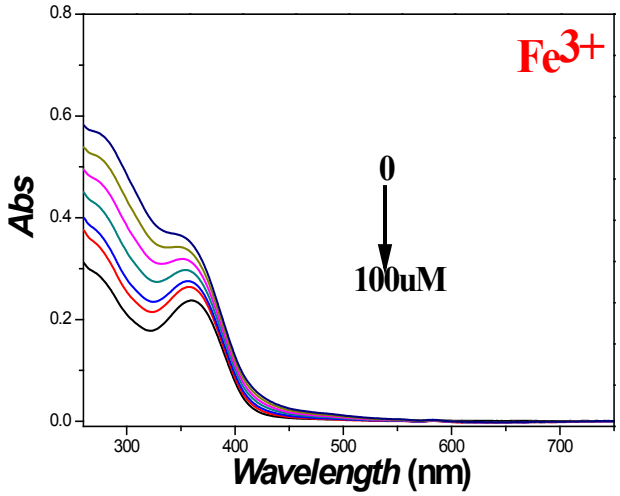


Fig.S22. UV-vis spectra titrating with [Fe^{3+}]

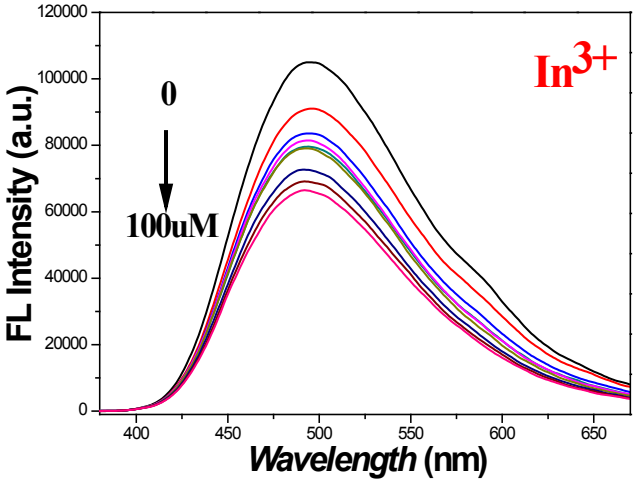


Fig.S23. Fluorescent spectra titrating with [In^{3+}]

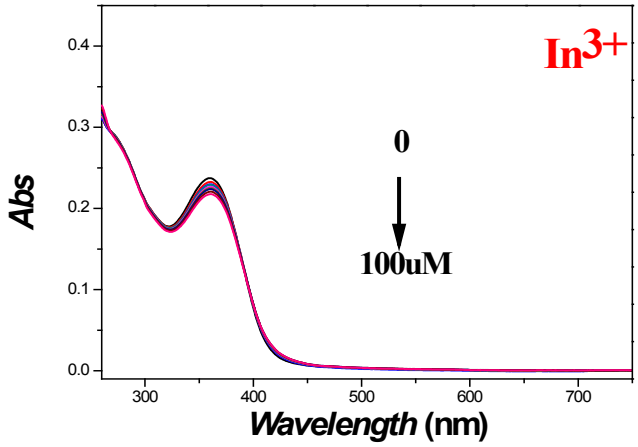


Fig.S24. UV-vis spectra titrating with [In^{3+}]

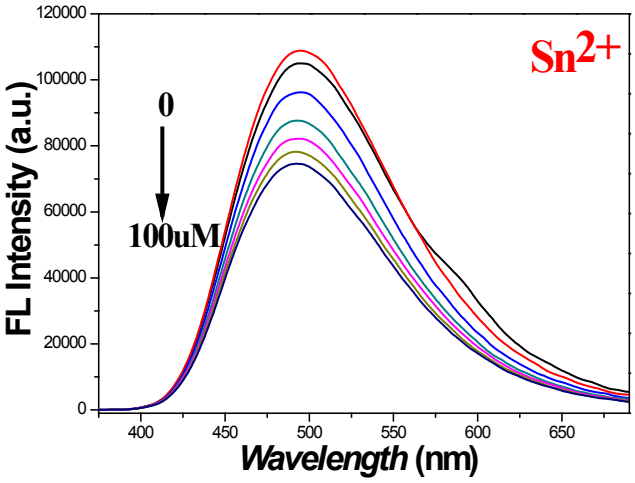


Fig.S25. Fluorescent spectra titrating with [Sn^{2+}]

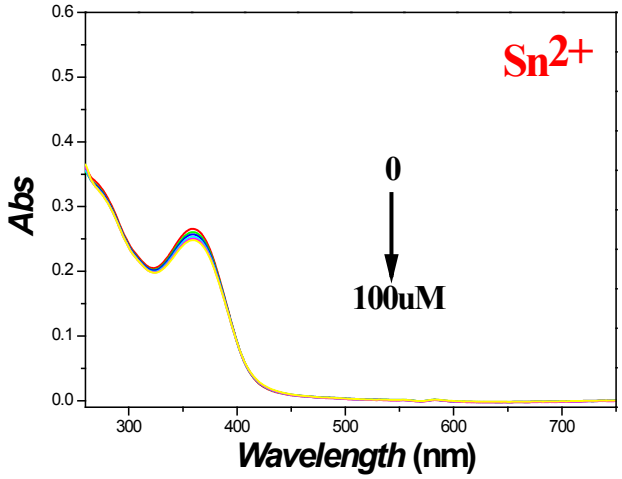


Fig.S26. UV-vis spectra titrating with [Sn^{2+}]

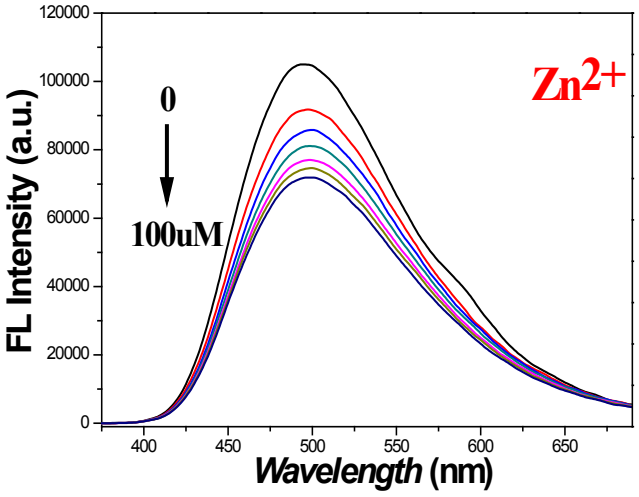


Fig.S27. Fluorescent spectra titrating with [Zn^{2+}]

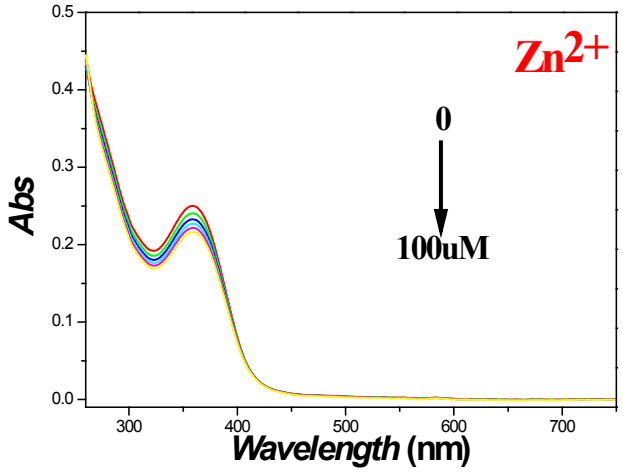


Fig.S28. UV-vis spectra titrating with [Zn^{2+}]

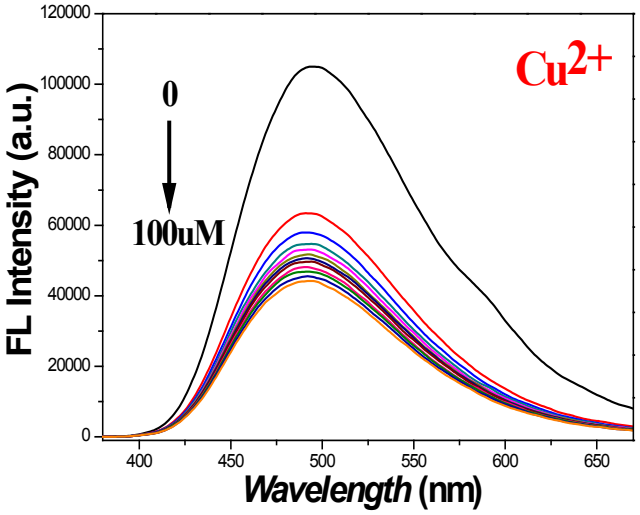


Fig.S29. Fluorescent spectra titrating with [Cu^{2+}]

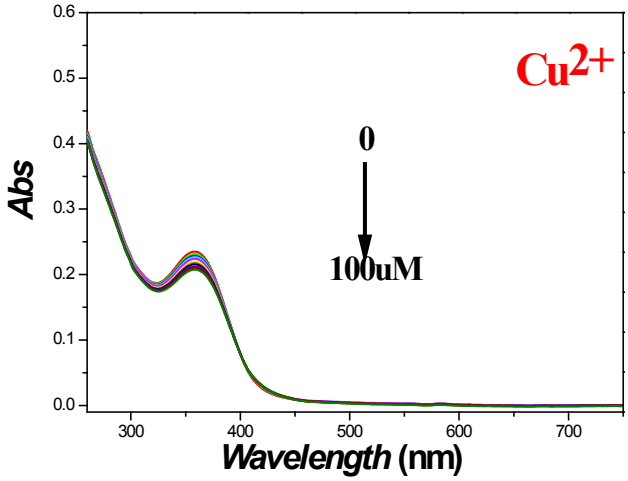


Fig.S30. UV-vis spectra titrating with [Cu^{2+}]

2.2(b) Fluorescent and UV-vis spectra of Phen-2TPE (2mL, 5×10^{-5} mol/L) titrating with different metal ions in THF

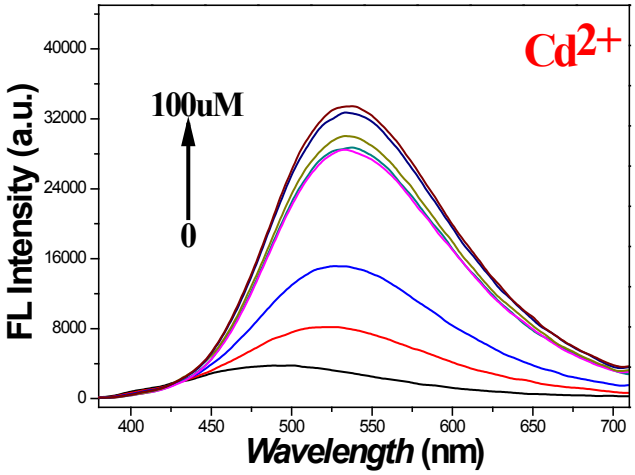


Fig.S31. Fluorescent spectra titrating with [Cd^{2+}]

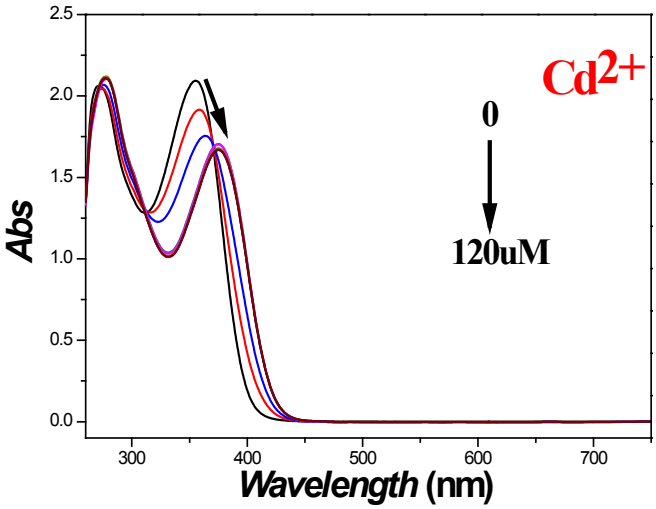


Fig.S32. UV-vis spectra titrating with [Cd^{2+}]

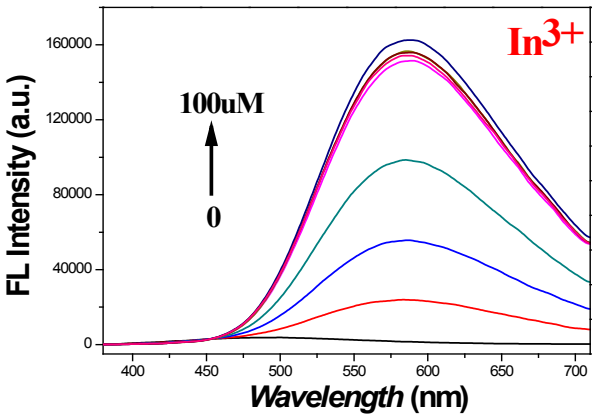


Fig.S33. Fluorescent spectra titrating with [In^{3+}]

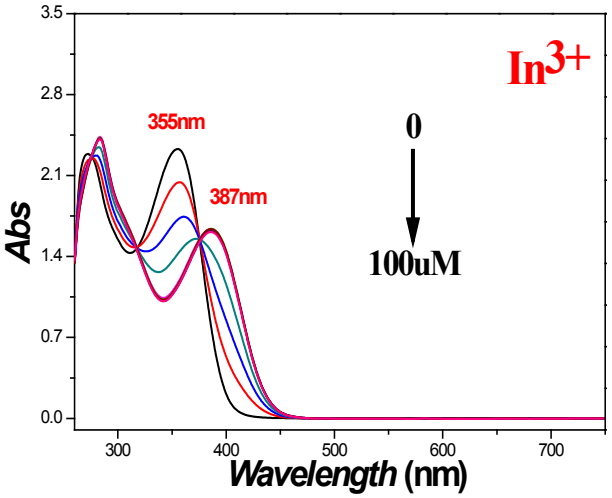


Fig.S34. UV-vis spectra titrating with [In^{3+}]

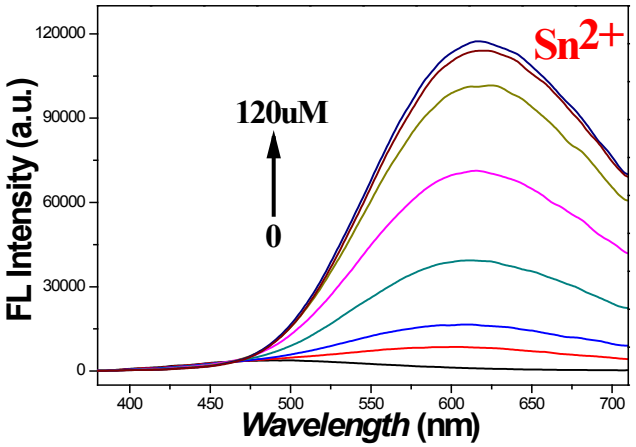


Fig.S35. Fluorescent spectra titrating with [Sn^{2+}]

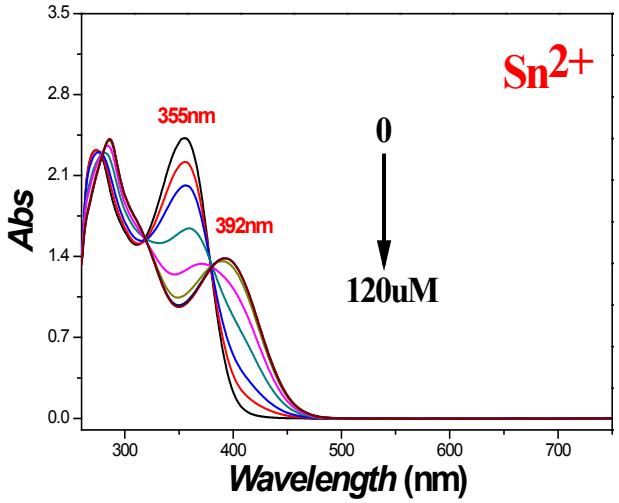


Fig.S36. UV-vis spectra titrating with [Sn^{2+}]

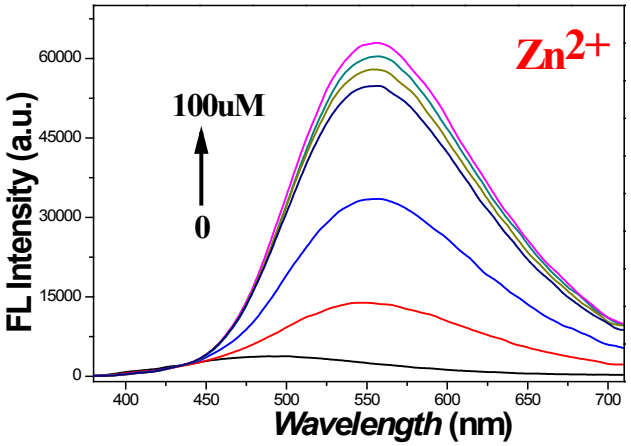


Fig.S37. Fluorescent spectra titrating with [Zn^{2+}]

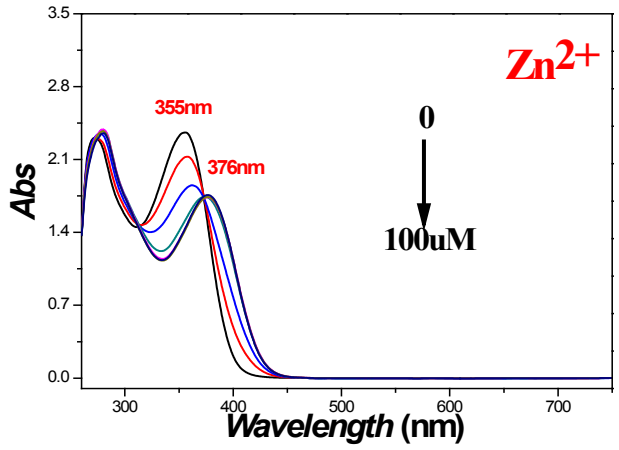


Fig.S38. UV-vis spectra titrating with [Zn^{2+}]

3. Fluorescence spectra of Phen-1TPE in f_w =90 %(10^{-5}mol/L, 2mL) titrating with different anions

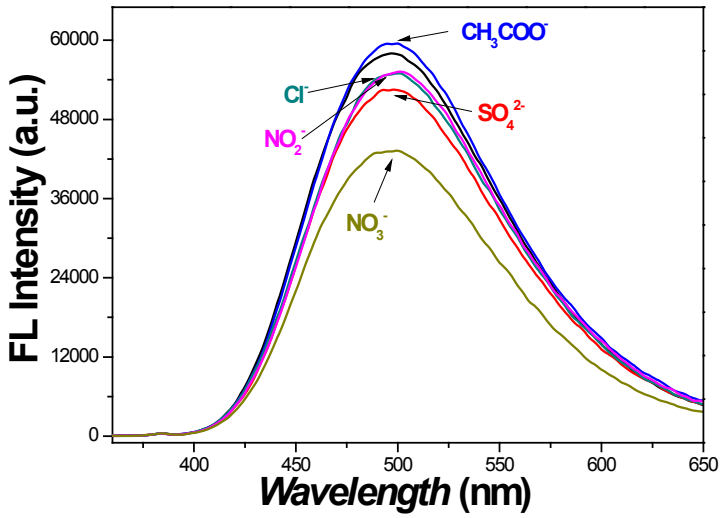


Fig.S39. FL spectra of Phen-1TPE in f_w = 90% (2mL, 10^{-5} mol/L) titration with 10 equivalent Na_2SO_4, NaCl, $NaNO_3$ and $NaNO_2$ and CH_3COONa

4. UV-vis Absorption and FL spectra of Phen-1TPE in THF (10^{-5}mol/L), f_w= 90 % (10^{-5}mol/L), f_w = 90 % (10^{-5}mol/L) + Cu^{2+}

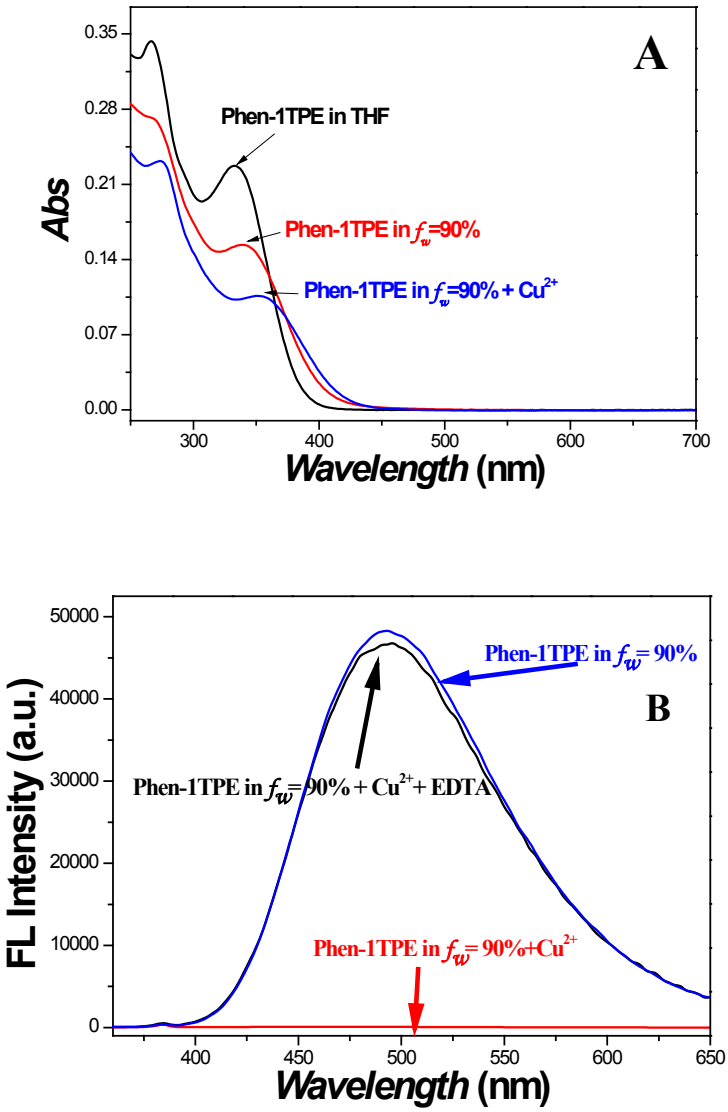


Fig.S40. (A) UV-vis absorption of Phen-1TPE in pure THF (10^{-5}mol/L), f_w = 90 % (10^{-5}mol/L), and in f_w = 90 % (10^{-5}mol/L) after adding 1 equivalent Cu^{2+}, **(B)** FL spectra of Phen-1TPE in f_w = 90 %, adding 1equiv Cu^{2+}, and 1euqiv Cu^{2+}+ 3equiv EDTA

5. DLS measurement

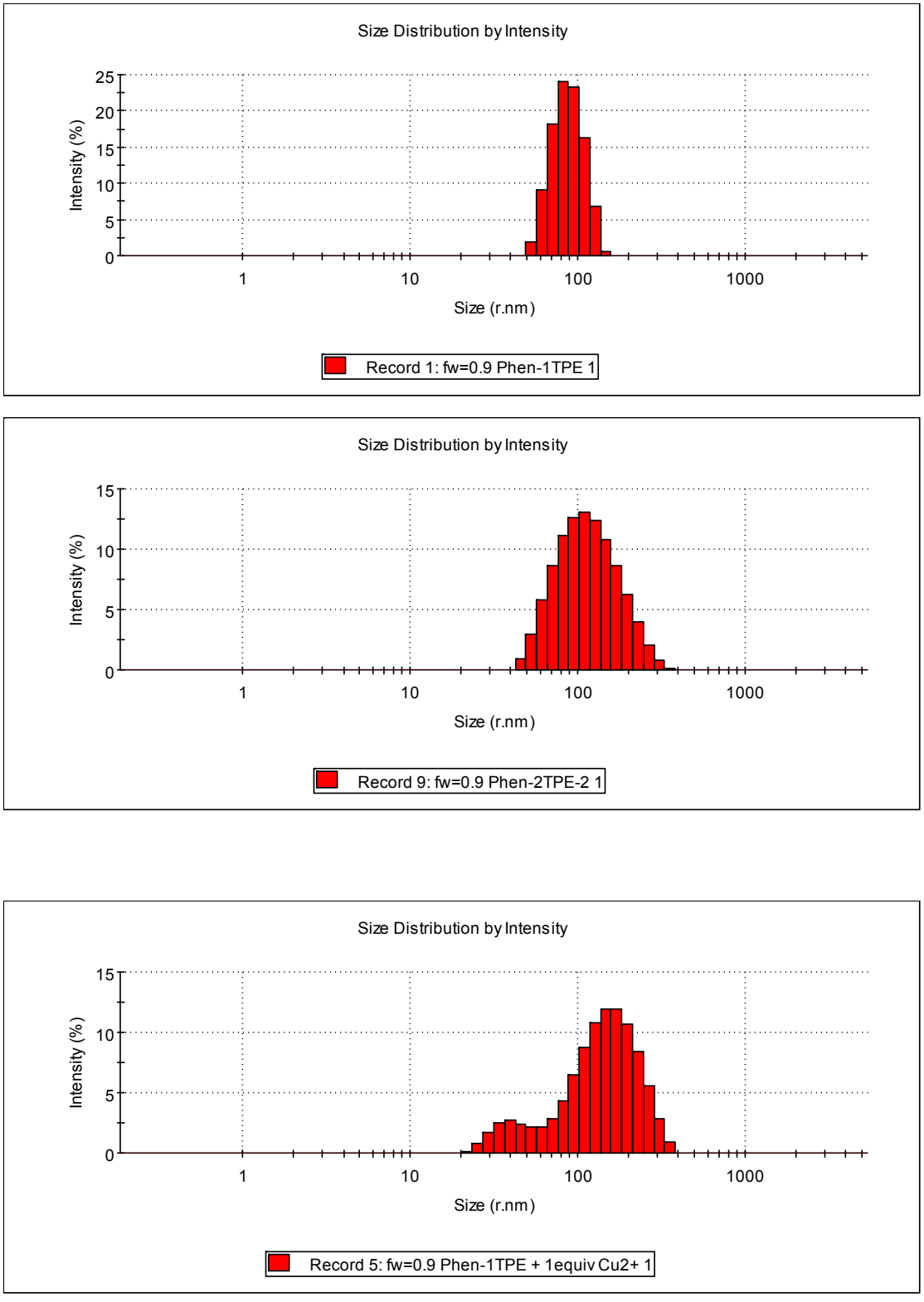


Fig.S41 DLS column diagrams of Phen-1TPE, Phen-2TPE (10^{-5}mol/L) in f_w = 90% and Phen-1TPE (10^{-5}mol/L) in f_w = 90% + 1 equiev Cu^{2+}

6. Table of metal ion test in THF for Phen-1TPE and Phen-2TPE

Metal ions	Phen-1TPE	Phen-2TPE
Li^+	×	×
K^+	×	×
Na^+	×	×
Mg^{2+}	×	×
Ca^{2+}	×	×
Al^{3+}	×	×
Ag^+	×	×
Pb^{2+}	×	×
In^{3+}	√	√
Cd^{2+}	√	√
Zn^{2+}	√	√
Sn^{2+}	√	√
Fe^{2+}	×	×
Fe^{3+}	×	×
Cu^+	×	×
Cu^{2+}	×	×

Fig.S42 Table of Phen-1TPE and Phen-2TPE in THF adding different metal ions. "×" means after adding the corresponding metal ions into it, no fluorescent enhancement is observed or detected; "√"means after adding the corresponding metal ions into it, fluorescence enhancement is observed or detected.

7. Phen-2TPE in f_w = 90% metal ion titration

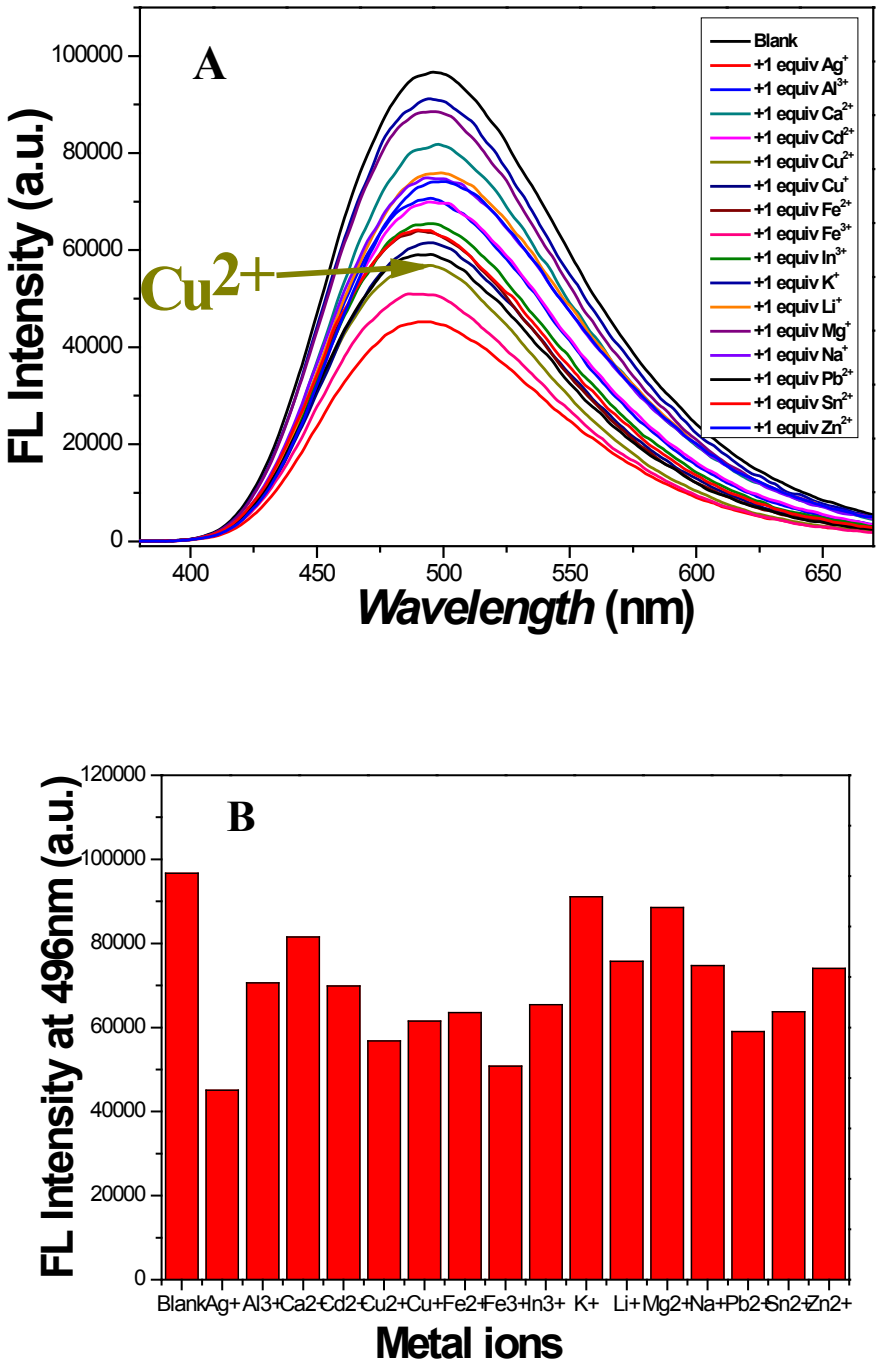


Fig.S43. FL spectra **(A)** and column diagram **(B)** of Phen-2TPE in f_w = 90 % by adding 1 equivalent amount of metal ions (1) Blank, (2) K^+, (3)Na^+, (4) Mg^{2+}, (5) Al^{3+}, (6) Cd^{2+}, (7) Ag^+, (8)In^{3+}, (9)Pb^{2+}, (10) Ca^{2+}, (11) Zn^{2+}, (12) Fe^{2+}, (13) Fe^{3+}, (14) Cu^+, (15) Cu^{2+}, (16) Sn^{2+}, (17) Li^+

8. The effect of pH to Phen-1TPE in f_w = 90%

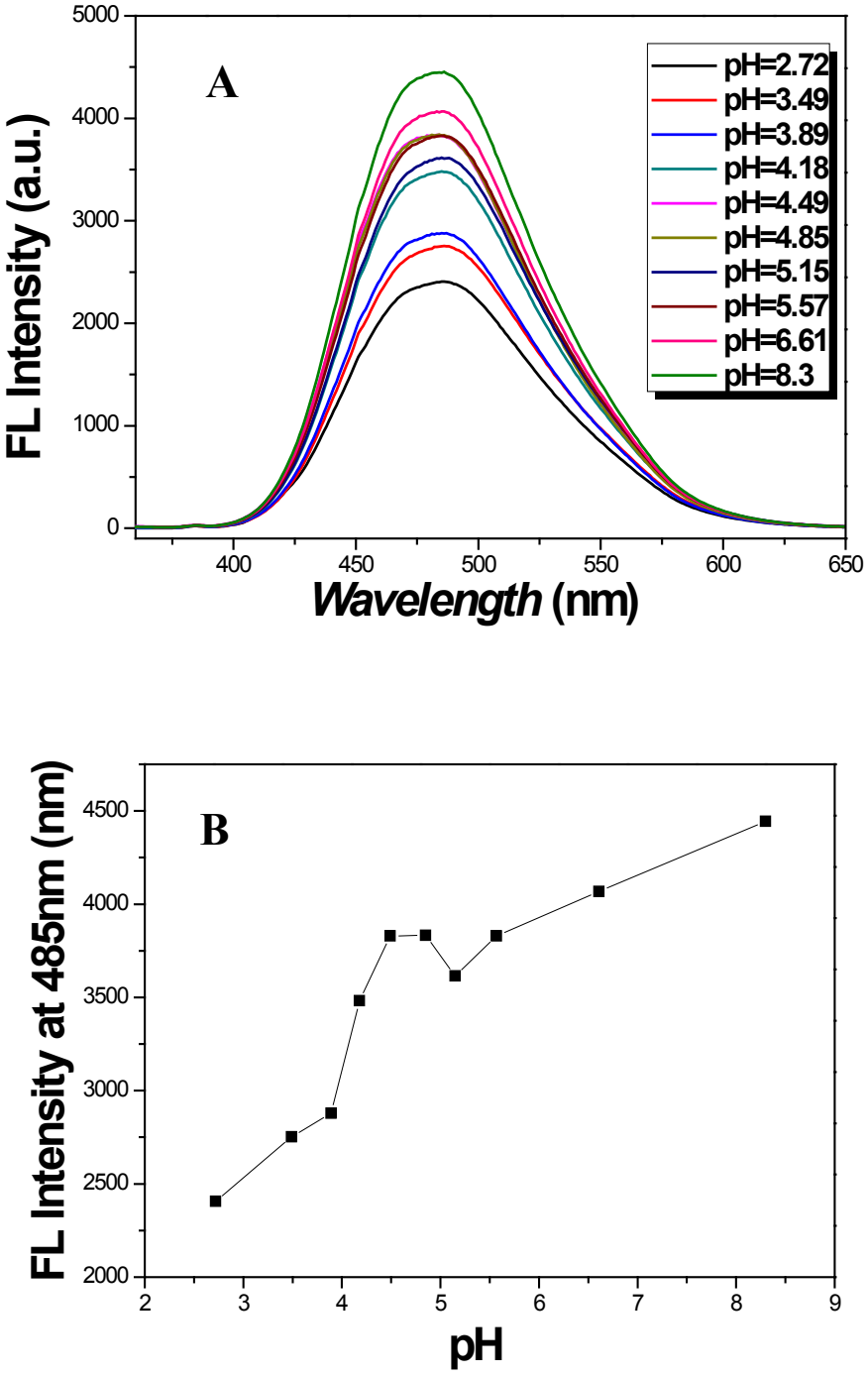
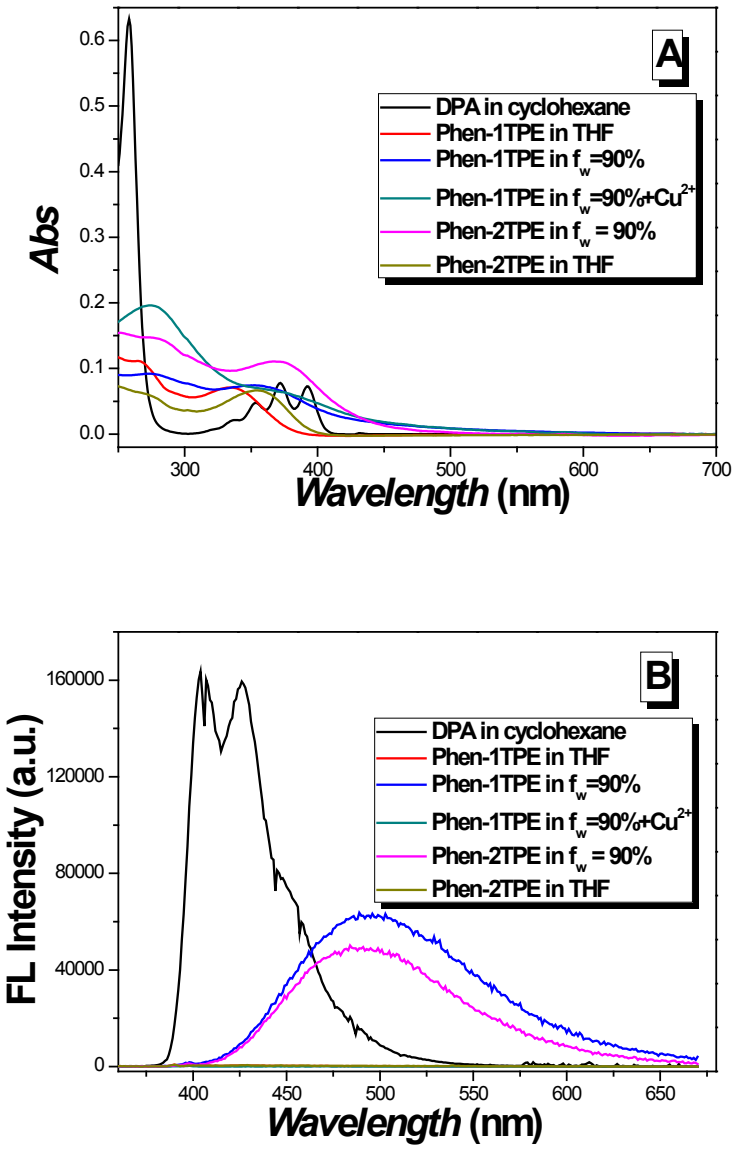


Fig.S44 (A) Fluorescent spectra of Phen-1TPE in f_w = 99 % at pH values from 2.72 to 8.3, (**B**) fluorescent intensity at 494nm at different pH from 2.72 to 8.3.

9. Fluorescence quantum yield and fluorescence lifetime

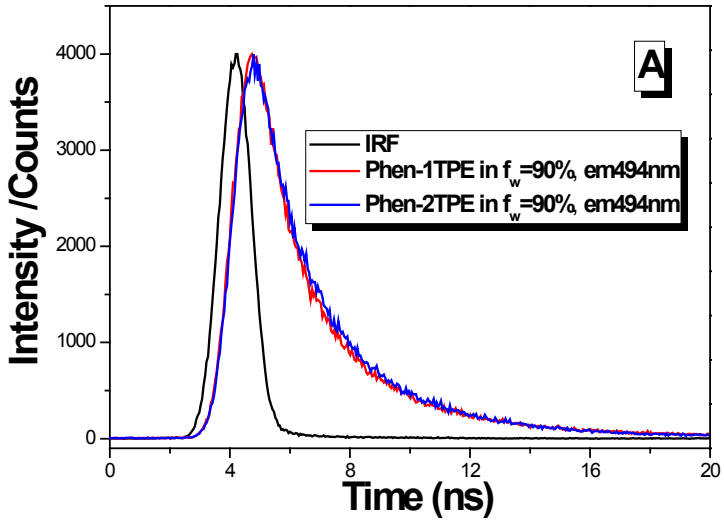
9.1 fluorescence quantum yield of Phen-1TPE in THF, f_w = 90%, f_w = 90% + Cu^{2+}, Phen-2TPE in THF and Phen-2TPE in f_w = 90%



C	Phen-1TPE			Phen-2TPE	
	In THF	In f_w = 90%	In f_w = 90%+Cu^{2+}	In THF	In f_w = 90%
Φ_F (%)	0.39	36.8	0.06	0.43	30.0

Fig.S45 (A) UV-vis, (**B**) FL spectra and (**C**) fluorescence quantum yield (Φ_F) of Phen-1TPE in THF, in f_w = 90 %, in f_w = 90 %+ Cu^{2+}, Phen-2TPE in THF and Phen-2TPE in f_w = 90% using DPA as standard (in cyclohexane, Φ_F = 0.9),

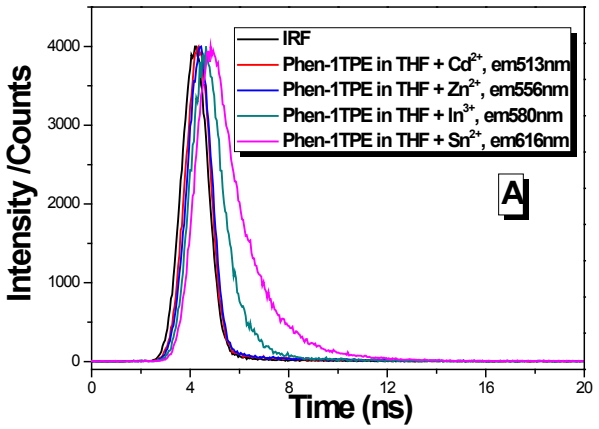
9.2 Fluorescence lifetime (τ) of Phen-1TPE and Phen-2TPE in f_w = 90%

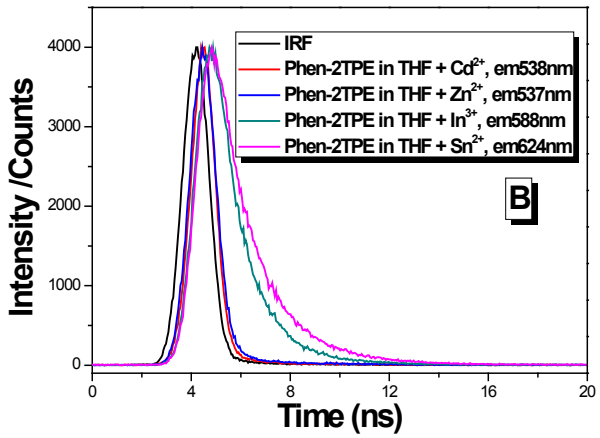


B	Phen-1TPE in f_w = 90%	Phen-2TPE in f_w = 90%
Fluorescence lifetime τ (ns)	τ_1 = 1.508, 66.46%, τ_2 = 4.607, 33.53% χ^2 = 1.422	τ_1 = 1.578, 59.76%, τ_2 = 3.983, 40.24% χ^2 = 1.218

Fig.S46 (A) Fluorescence decay curves of Phen-1TPE (red) and Phen-2TPE (blue) in f_w = 90% (10^{-5}M, k_{ex} = 340nm and k_{em} = 494nm) (**B**) table list for the corresponding spectra

9.3 Fluorescence lifetime (τ) of Phen-1TPE and Phen-2TPE in THF by adding metal ions (Cd^{2+}, Zn^{2+}, In^{3+} and Sn^{2+})





C	Fluorescence lifetime τ (ns)
Phen-1TPE in THF + Cd^{2+}	τ_1 = 0.0312, 97.33%, τ_2 = 5.1098, 2.67%, χ^2 = 1.392
Phen-1TPE in THF + Zn^{2+}	τ_1 = 0.0681, 97.08%, τ_2 = 5.1929, 2.92%, χ^2 = 1.465
Phen-1TPE in THF + In^{3+}	τ_1 = 0.052, 96.86%, τ_2 = 5.08, 3.14%, χ^2 = 1.41
Phen-1TPE in THF + Sn^{2+}	τ_1 = 1.2, 96.67%, τ_2 = 2.94, 3.33%, χ^2 = 1.281
Phen-2TPE in THF + Cd^{2+}	τ_1 = 0.1126, 99.24%, τ_2 = 5.7369, 0.76%, χ^2 = 1.195
Phen-2TPE in THF + Zn^{2+}	τ_1 = 0.0648, 97.20%, τ_2 = 5.2495, 2.8%, χ^2 = 1.424
Phen-2TPE in THF + In^{3+}	τ_1 = 1.0007, 85.15%, τ_2 = 2.067, 14.85%, χ^2 = 1.236
Phen-2TPE in THF + Sn^{2+}	τ_1 = 0.991, 38.05%, τ_2 = 1.8478, 61.95%, χ^2 = 1.14

Fig.S47 (A) Fluorescence decay curves of Phen-1TPE in THF by adding metal ions (10^{-5}M, k_{ex} = 340nm and k_{em} Cd^{2+} (513 nm), Zn^{2+} (556 nm), In^{3+} (580 nm)) and Sn^{2+} (616 nm), (**B**) Fluorescence decay curves of Phen-2TPE in THF by adding metal ions (10^{-5}M, k_{ex} = 340 nm and k_{em} Cd^{2+} (538 nm), Zn^{2+} (537 nm), In^{3+} (588 nm) and Sn^{2+} (624 nm), (**C**) table list for the corresponding spectra.

10. SEM image of Phen-1TPE and Phen-2TPE

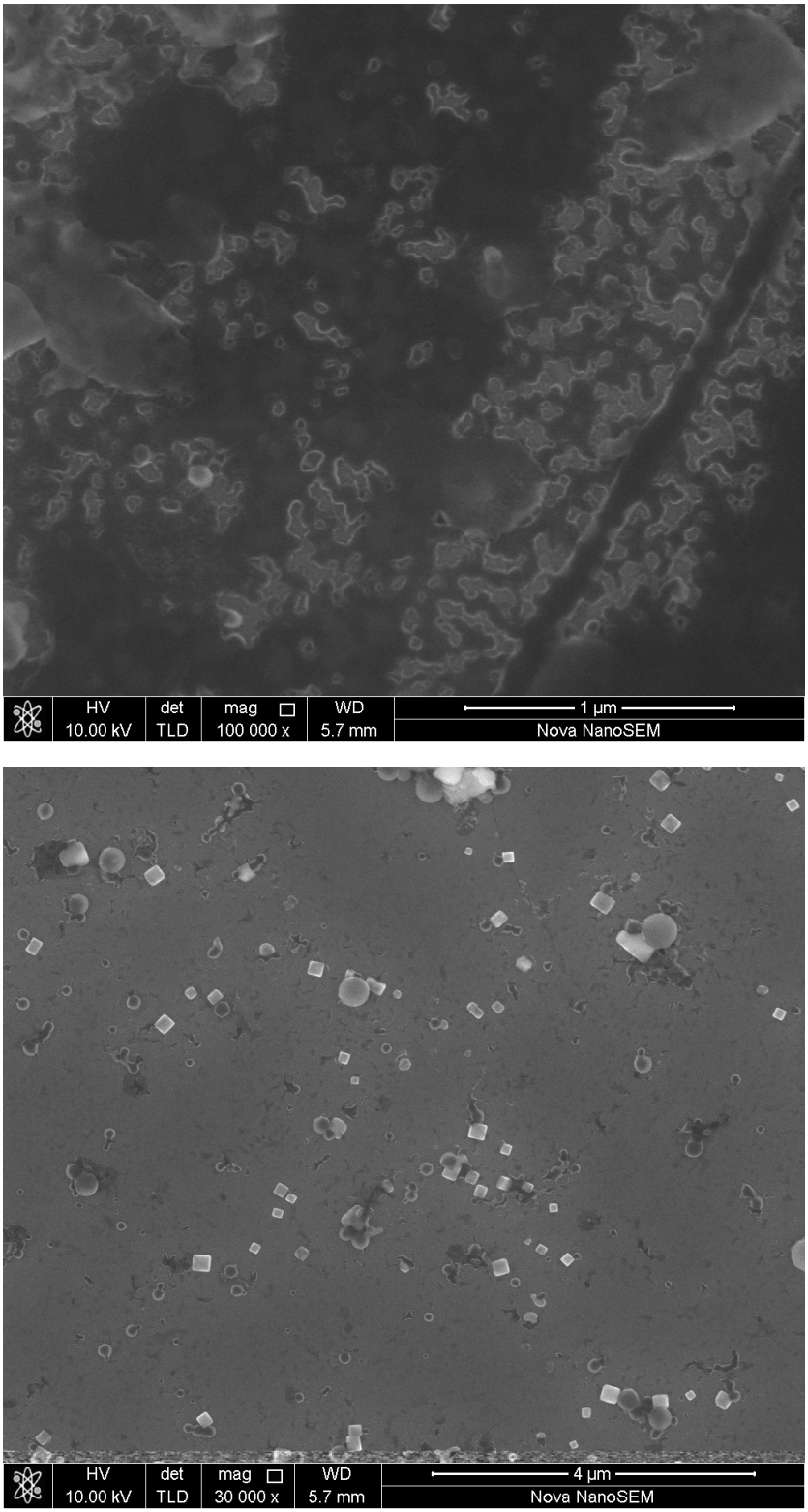


Fig.S48 SEM image of Phen-1TPE (upside) and Phen-2TPE (downside) prepared from f_w = 90%, concentration equals 10-5mol/L

11.^{1}H-NMR and ^{13}C-NMR Spectra

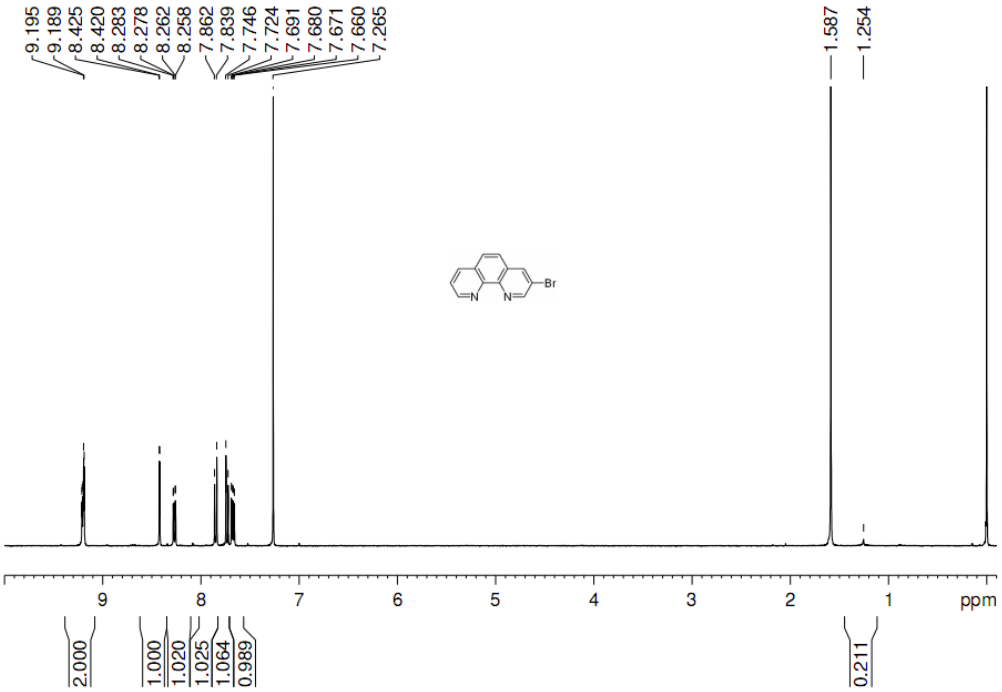


Fig S49.^{1}H-NMR spectrum of 3-bromo-1, 10-phenanthroline in $CDCl_3$

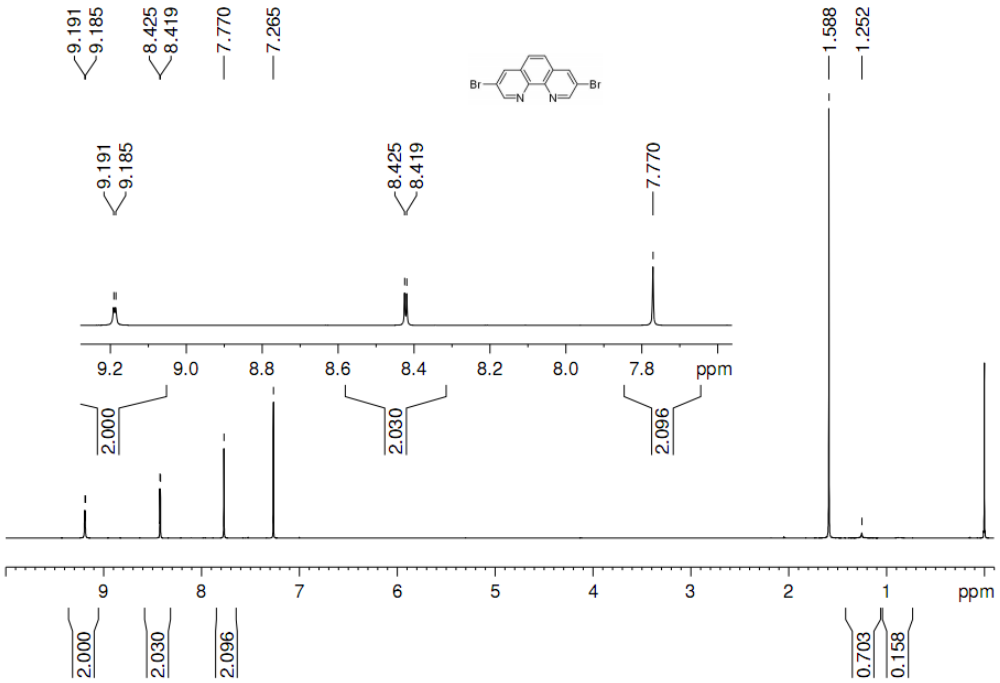


Fig S50.^{1}H-NMR spectrum of 3, 8-dibromo-1, 10-phenanthroline in $CDCl_3$

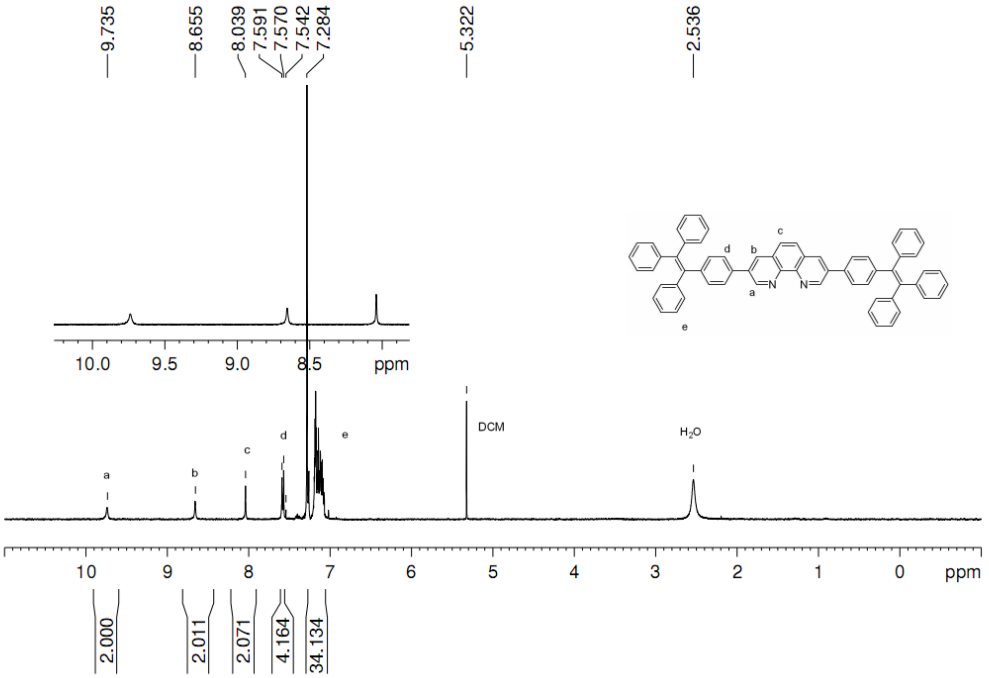


Fig S51. ^{1}H-NMR spectrum of 3, 8-ditetraphenylethene-1, 10-phenanthroline in $CDCl_3$

Supplementary Information

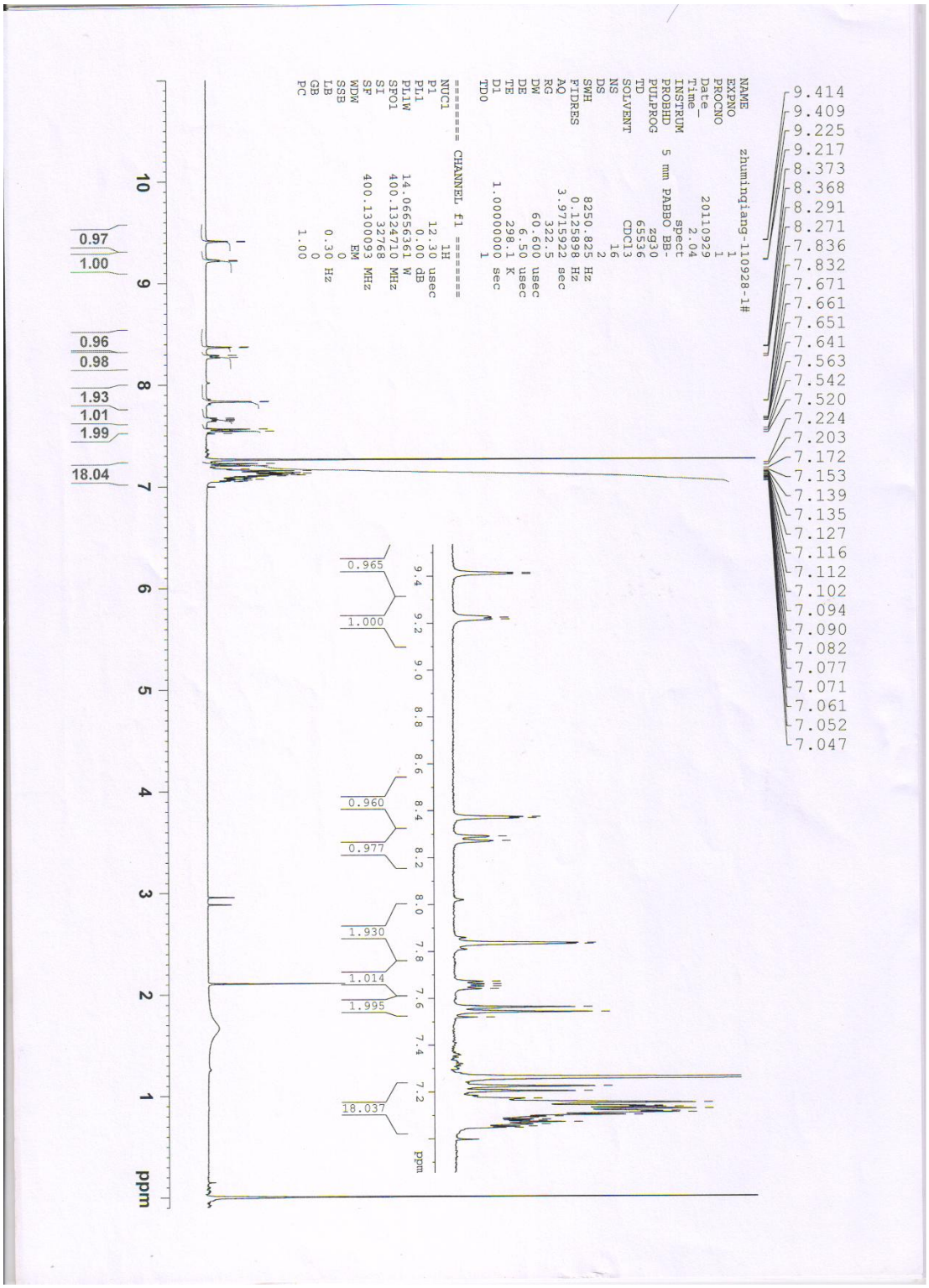


Fig S52. ^{1}H-NMR Spectrum of 3-tetraphenylethene-1, 10-phenanthroline in $CDCl_3$

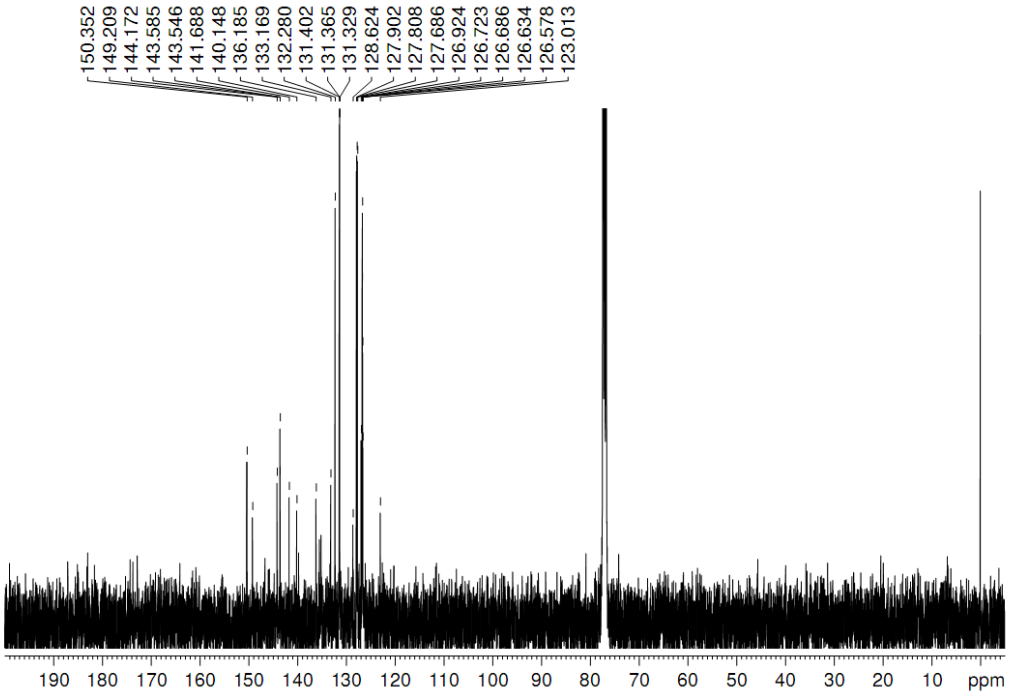


Fig S53. ^{13}C-NMR Spectrum of 3-tetraphenylethene-1, 10-phenanthroline in $CDCl_3$

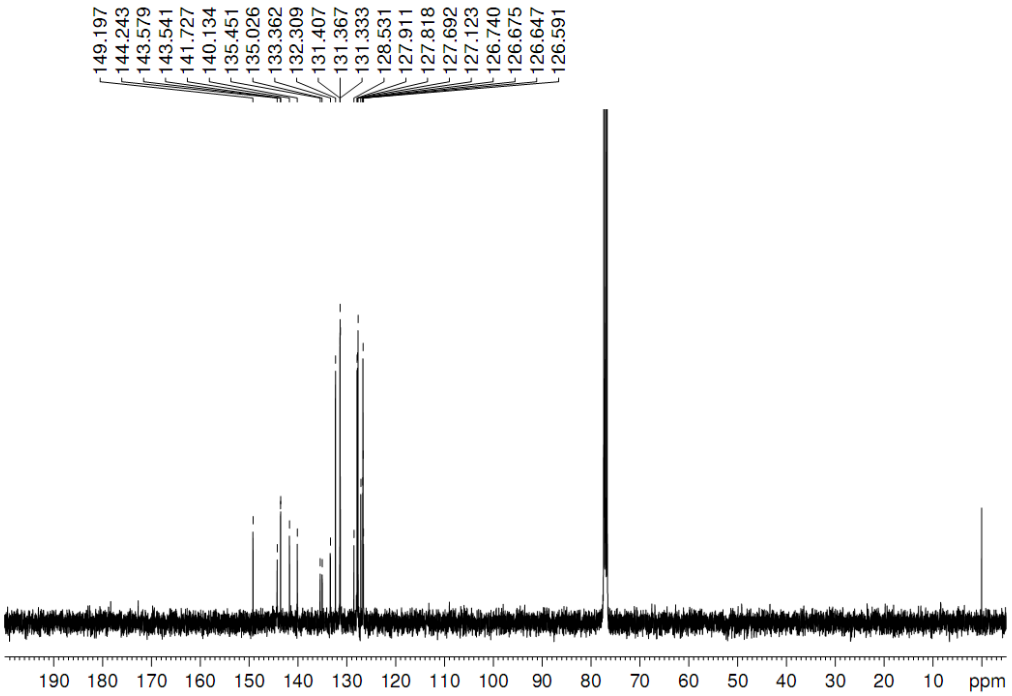


Fig S54. ^{13}C-NMR spectrum of 3, 8-ditetraphenylethene-1, 10-phenanthroline in $CDCl_3$

12. Mass Spectra

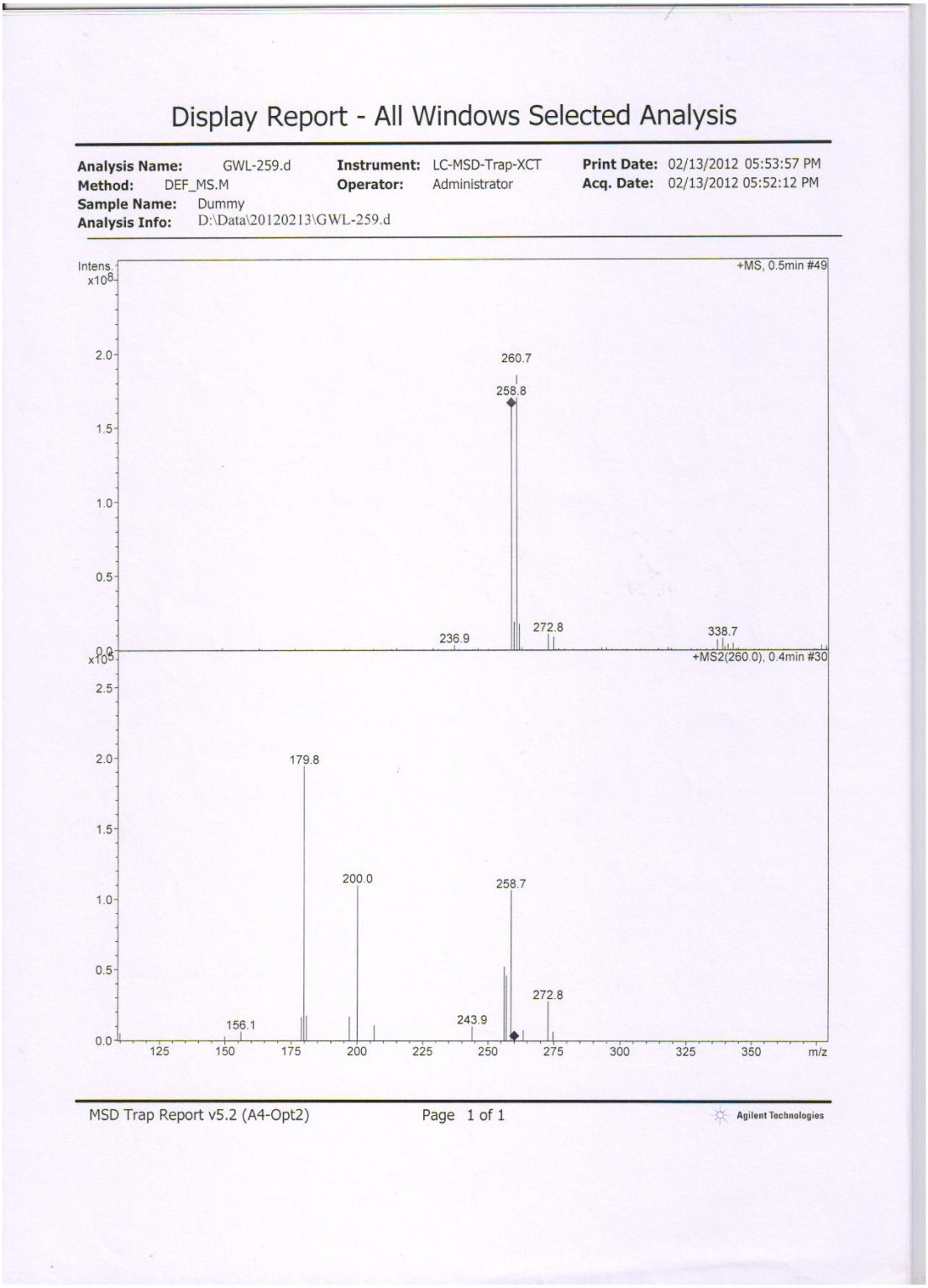


Fig.S55 Mass spectrum of 3-bromo-1, 10-phenanthroline

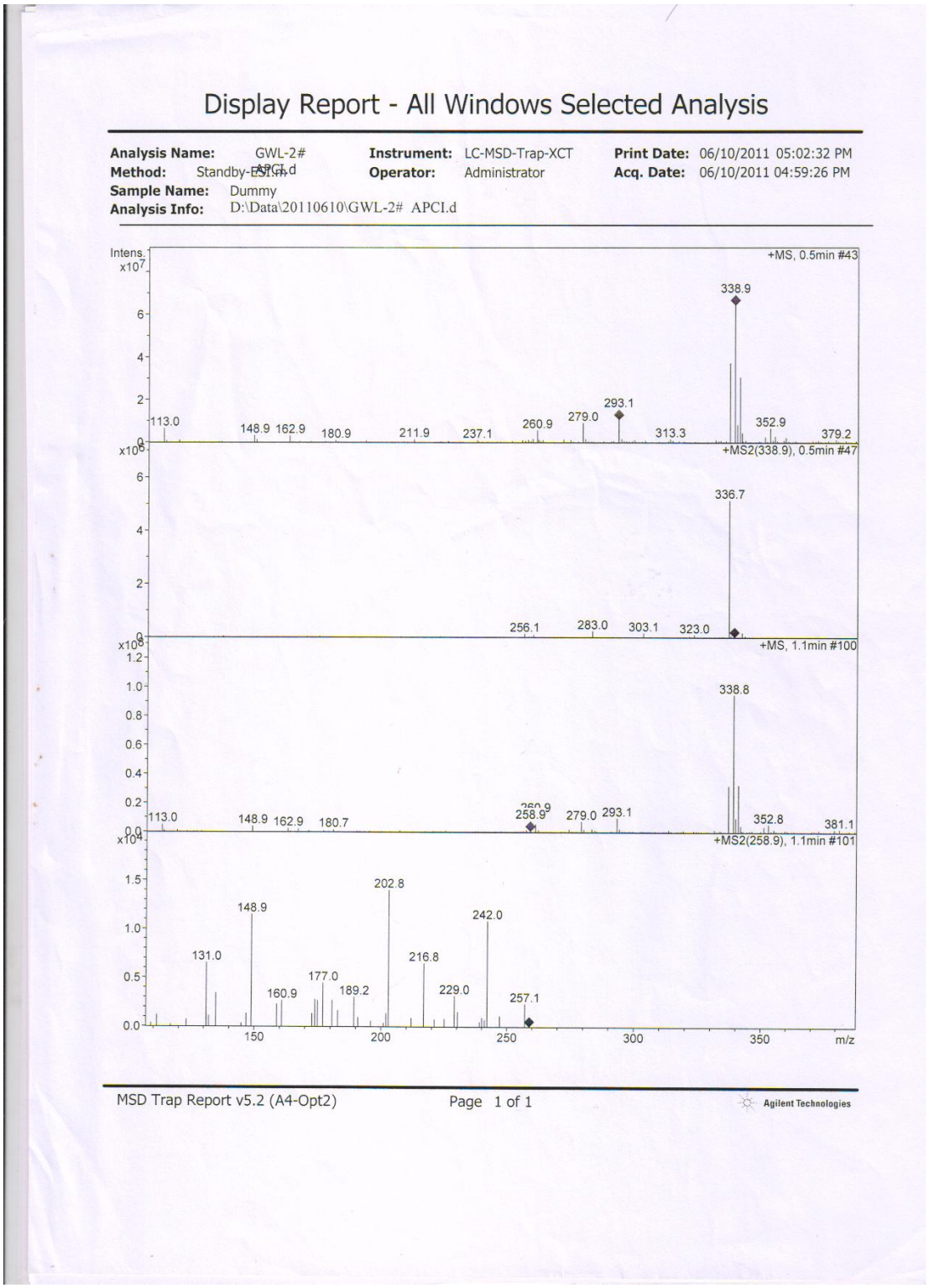


Fig S56. Mass spectrum of 3, 8-dibromo-1, 10-phenanthroline

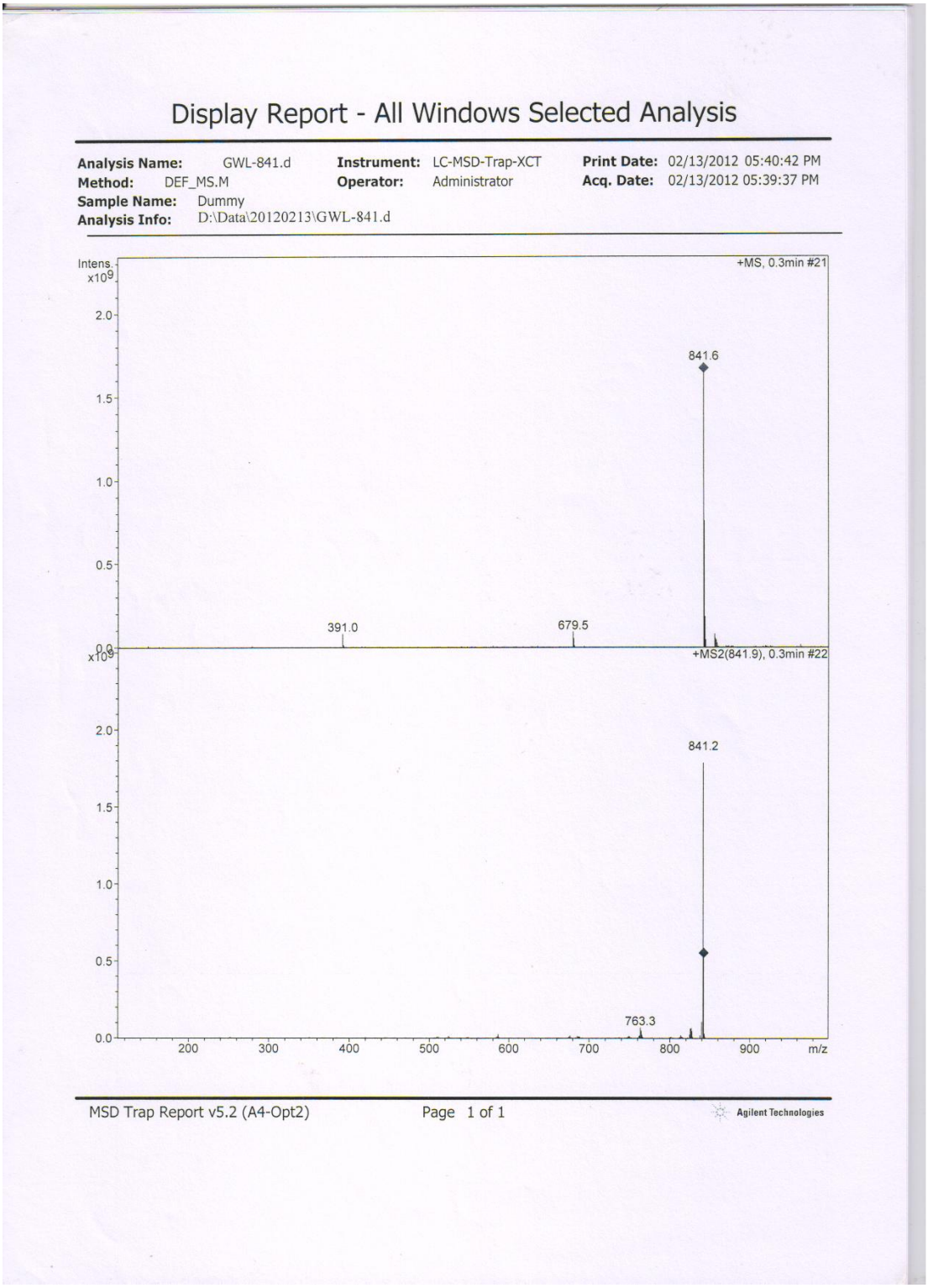


Fig S57. Mass spectrum of 3, 8-diTPE-1, 10-phenanthroline

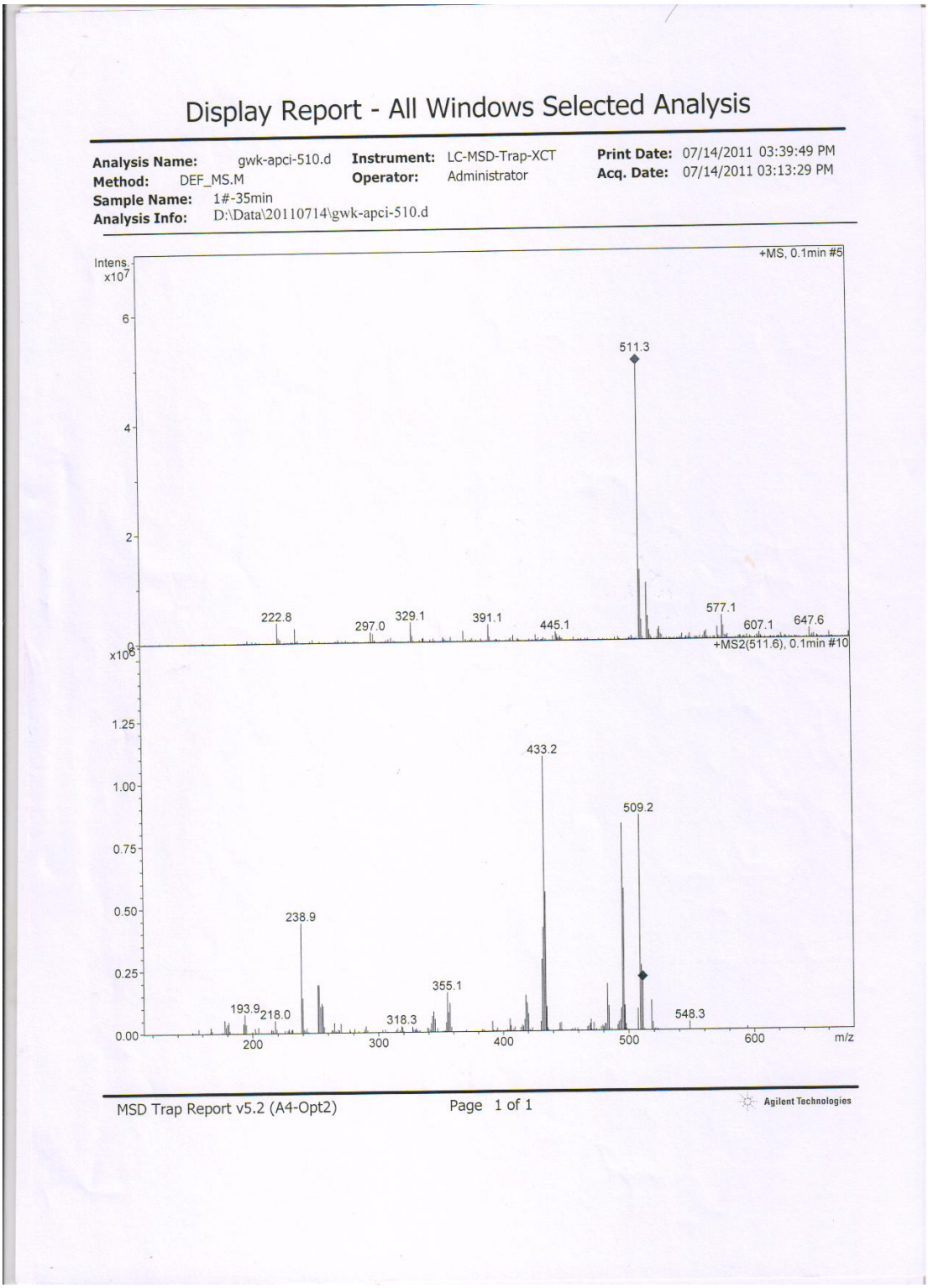


Fig S58. Mass spectrum of 3-tetraphenylethene-1, 10-phenanthroline