Supporting Information

Chiral and non-conjugated fluorescent Salen ligands: AIE, anion probes, chiral recognition of unprotected amino acids, and cell imaging applications

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Experimental Section

Materials and Instrumentation. All reagents were purchased from commercial suppliers and used without further purification. All the Salen ligands were prepared according to previous reports.[14] ^{1}HNMR (400MHz) spectra were recorded in $CDCl_3$ or DMSO-d_6. Chemical shifts are reported in ppm using tetramethylsilane as internal standard. UV/vis absorption spectra were recorded using a U5100 (Hitachi) spectrophotometer with quartz cuvettes of 1 cm pathlength. Fluorescence spectra were obtained using F-7000 Fluorescence spectrophotometer (Hitachi) at room temperature. The slit width was 5 nm and 2.5 nm for excitation and emission. The photon multiplier voltage was 400 V. CD spectra were recorded using a Chirascan plus qCD (Applied Photophysics) at room temperature. Samples in solution and powder were contained in 1 cm path length quartz cuvettes (3.5 mL volume) and quartz tube, respectively. The single-crystals of **3-F-Cy**, **3-F-(R,R)Cy**, **3-F-(S,S)Cy**, **3-F-diPh**, and **3-Cl-diPh**, were obtained by a slow diffusion/evaporation of CH_2Cl_2/ethyl acetate/hexane solution at room temperature during about two weeks.

Measurement of Fluorescence Quantum Yield (Φ). The quantum yield of a solution sample was measured by the optical dilute method of Demas and Crosby[18] with a standard of quinine sulfate (Φ_r = 0.55, quinine in 0.05 mol dm^{-3} sulfuric acid) calculated by: $\Phi_s = \Phi_r(B_r/B_s)(n_s/n_r)^2(D_s/D_r)$, where the subscripts s and r refer to the sample and reference standard solution respectively; *n* is the refractive index of the solvents; D is the integrated intensity. The excitation intensity *B* is calculated by: $B = 1 - 10^{-AL}$, where A is the absorbance at the excitation wavelength and L is the optical path length (L = 1 cm in all cases). The refractive indices of the solvents at room temperature are taken from standard source. Errors for Φ values (± 10%) are estimated. The quantum yield of a solid sample was measured by an integrating sphere.

Computational Details. Calculations were carried out using the Gaussian 09 software package (B3LYP 6-31G(d,p)). The geometry optimization and absorption transition and spectrum were carried out by DFT and TD-DFT, respectively. The theoretical modelling was performed in the isolated molecule approximation ignoring the effect of the aggregation state or solvent. For the atoms of **3-F-Cy**, the standard split-valence basis sets B3LYP 6-31G(d,p) augmented with polarization d-functions for the non-hydrogen atoms and p-functions for the hydrogen atoms were used. Full geometry optimization corresponding to the minima on the potential energy surface (PES) was conducted until a gradient of 10^{-5} at.u. The spin multiplicities and charges of the **3-F-Cy** were set equal to 1 and 0, respectively. The spin multiplicities and charges of the **3-F-Cy** (in form of O^-) were set equal to 1 and −2, respectively. The other parameters were set to default values.

Cell Culture Methods and Imaging. The imaging of HeLa cells was finished by Fluorescence Vertical Microscope LEICA DM2500. HeLa cells were cultured in dulbecco's modified eagle medium (DMEM) supplemented with 10 % fetal bovine serum, penicillin (100 units mL^{-1}), streptomycin (100 mg mL^{-1}) and 5 % CO_2 at 37 °C. After removing the incubating media and rinse with PBS for three times, the cells were incubated with the dye (1.0×10^{-5} mol dm^{-3}) in PBS for 2 h at room temperature. Then, the cells were washed three times with PBS and incubated with aqueous alkali for 20 min. At last, the cells were imaged with confocal microscope.

Measurement of Anion hosts/probes: Anion titration experiment was started with the dye (10 mL) of known concentration (1.0×10^{-5} mol dm^{-3} in DMSO). For the titration, various sodium or potassium salts (1.0–0.10 mol dm^{-3} in water) were added by a microsyringe. All types of absorption and fluorescence measurement were monitored at about 1 hours after the addition of the anion to the dye solution at room temperature.

Measurement of Chirality amino acid hosts/probes: Anion titration experiment was started with the dye (10 mL) of known concentration (1.0×10^{-5} mol dm^{-3} in DMSO). For the titration, various D- or L-amino acids (5.0×10^{-2}–5.0×10^{-1} mol dm^{-3} in water) were added by a microsyringe. In order to improve the solubility of amino acids in DMSO, ultrasonic treatment was adopted. All types of absorption and fluorescence measurement were monitored at about 1 hours after the addition of the anion to the dye solution at room temperature.

Synthesis of Organic Dyes: Salen ligands were prepared by a similar method according to previous reports.[14] The mixture of salicyaldehyde or salicylaldehyde derivatives (2.1 mmol) and the corresponding diamine (1.0 mmol) in 20 mL ethanol solution was refluxed at 78 °C for 5 h. After the reaction was complete, the mixture was cooled to 0 °C and then the product in crystal or powder was collected by filtration.

Cy (74% yield): ^1H NMR (400 MHz, $CDCl_3$) δ 13.40 (s, 2H), 8.26 (s, 2H), 7.36 – 6.73 (m, 8H), 3.45 – 3.11 (m, 2H), 2.18 – 1.25 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.44, 161.11, 132.23, 131.48, 118.64, 118.52, 116.77, 72.63, 33.10, 24.18. HRMS (ESI):Calculated for

$C_{20}H_{22}N_2O_2$ [[M+Na]$^+$] 345.1579, found 345.1588. Anal. Calcd. (Found): C, 74.51 (74.48); H, 6.88 (6.91); N, 8.69 (8.66).

(R,R)Cy (77% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.41 (s, 2H), 8.26 (s, 2H), 7.36 – 6.74 (m, 8H), 3.45 – 3.12 (m, 2H), 2.17 – 1.25 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.46, 161.11, 132.23, 131.47, 118.64, 118.52, 116.77, 72.63, 33.10, 24.18. HRMS (ESI):Calculated for $C_{20}H_{22}N_2O_2$ [[M+Na]$^+$] 345.1579, found 345.1588. Anal. Calcd. (Found): C, 74.51 (74.45); H, 6.88 (6.94); N, 8.69 (8.71).

(S,S)Cy (70% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.40 (s, 2H), 8.26 (s, 2H), 7.36 – 6.73 (m, 8H), 3.44 – 3.11 (m, 2H), 2.18 – 1.25 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.44, 161.10, 132.25, 131.48, 118.65, 118.52, 116.77, 72.63, 33.10, 24.18. HRMS (ESI):Calculated for $C_{20}H_{22}N_2O_2$ [[M+Na]$^+$] 345.1579, found 345.1588. Anal. Calcd. (Found): C, 74.51 (74.47); H, 6.88 (6.90); N, 8.69 (8.67).

3-F-Cy (75% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.67 (s, 2H), 8.26 (s, 2H), 7.07 (ddd, *J* = 10.9, 8.1, 1.4 Hz, 2H), 6.93 (dt, *J* = 7.9, 1.2 Hz, 2H), 6.71 (td, *J* = 7.8, 4.5 Hz, 2H), 3.37 – 3.30 (m, 2H), 2.03 – 1.38 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.54, 152.46, 149.95, 126.48, 120.15, 118.52, 117.81, 72.42, 32.97, 24.04. HRMS (ESI):Calculated for $C_{20}H_{20}F_2N_2O_2$ [[M+Na]$^+$] 381.1391, found 381.1366. Anal. Calcd. (Found): C, 67.03 (67.06); H, 5.62 (5.65); N, 7.82 (7.89).

3-F-(R,R)Cy (70% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.68 (s, 2H), 8.26 (s, 2H), 7.07 (ddd, *J* = 10.9, 8.1, 1.4 Hz, 2H), 6.94 (dt, *J* = 7.9, 1.2 Hz, 2H), 6.71 (td, *J* = 7.8, 4.5 Hz, 2H), 3.47 – 3.16 (m, 2H), 2.05 – 1.38 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.54, 152.46, 149.95, 126.47, 120.15, 118.52, 117.81, 72.42, 32.97, 24.04. HRMS (ESI):Calculated for $C_{20}H_{20}F_2N_2O_2$ [[M+Na]$^+$] 381.1391, found 381.1366. Anal. Calcd. (Found): C, 67.03 (67.07); H, 5.62 (5.64); N, 7.82 (7.76).

3-F-(S,S)Cy (68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.68 (s, 2H), 8.27 (s, 2H), 7.07 (ddd, *J* = 10.9, 8.1, 1.4 Hz, 2H), 6.95 (dt, *J* = 7.8, 1.2 Hz, 2H), 6.71 (td, *J* = 7.9, 4.5 Hz, 2H), 3.51 – 3.11 (m, 2H), 2.13 – 1.34 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.53, 152.46, 149.95, 126.46, 120.15, 118.52, 117.82, 72.42, 32.97, 24.05. HRMS (ESI):Calculated for $C_{20}H_{20}F_2N_2O_2$ [[M+Na]$^+$] 381.1391, found 381.1366. Anal. Calcd. (Found): C, 67.03 (67.05); H, 5.62 (5.67); N, 7.82 (7.84).

3-Cl-Cy (72% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.32 (s, 2H), 8.24 (s, 2H), 7.35 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.08 (dd, *J* = 7.7, 1.6 Hz, 2H), 6.74 (t, *J* = 7.5 Hz, 2H), 3.42 – 3.27 (m, 2H), 1.92 – 1.43 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.40, 157.55, 132.64, 130.02, 121.58, 118.64, 72.15, 68.46, 32.97, 24.01. HRMS (ESI):Calculated for $C_{20}H_{20}Cl_2N_2O_2$ [[M+Na]$^+$] 413.0799, found 413.0787. Anal. Calcd. (Found): C, 61.39 (61.34); H, 5.15 (5.18); N, 7.16 (7.13).

3-Cl-(R,R)Cy (69% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.32 (s, 2H), 8.24 (s, 2H), 7.35 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.09 (dd, *J* = 7.7, 1.6 Hz, 2H), 6.74 (t, *J* = 7.5 Hz, 2H), 3.39 – 3.26 (m, 2H), 1.92 – 1.43 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.40, 157.55, 132.64, 130.02, 121.59, 118.64, 72.15, 68.46, 32.97, 24.01. HRMS (ESI):Calculated for $C_{20}H_{20}Cl_2N_2O_2$ [[M+Na]$^+$] 413.0799, found 413.0787. Anal. Calcd. (Found): C, 61.39 (67.05); H, 5.15 (5.13); N, 7.16 (7.19).

3-Cl-(S,S)Cy (72% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.32 (s, 2H), 8.24 (s, 2H), 7.35 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.08 (dd, *J* = 7.7, 1.6 Hz, 2H), 6.74 (t, *J* = 7.5 Hz, 2H), 3.41 – 3.28 (m, 2H), 1.94 – 1.41 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.40, 157.55, 132.64, 130.01, 121.58, 118.64, 72.15, 68.46, 32.97, 24.01. HRMS (ESI):Calculated for $C_{20}H_{20}Cl_2N_2O_2$ [[M+Na]$^+$] 413.0799, found 413.0787. Anal. Calcd. (Found): C, 61.39 (61.43); H, 5.15 (5.17); N, 7.16 (7.15).

3-*t*-Bu-Cy (68% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.95 (s, 2H), 8.34 (s, 2H), 7.28 (dd, *J* = 7.7, 1.6 Hz, 2H), 7.07 (dd, *J* = 7.6, 1.6 Hz, 2H), 6.76 (t, *J* = 7.6 Hz, 2H), 3.58 (m, 2H), 2.11 – 1.53 (m, 8H), 1.39 (s, 18H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.92, 160.54, 137.33, 129.84, 129.25, 118.73, 117.60, 69.32, 34.84, 30.52, 29.34, 22.67. HRMS (ESI):Calculated for $C_{20}H_{38}N_2O_2$ [[M+Na]$^+$] 457.2831, found 457.2826. Anal. Calcd. (Found): C, 77.38 (77.43); H, 8.81 (8.79); N, 6.45 (6.49).

3-OMe-Cy (70% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.87 (s, 2H), 8.24 (s, 2H), 6.87 – 6.71 (m, 6H), 3.86 (s, 6H), 3.35 – 3.26 (m, 2H), 1.98 – 1.43 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.75, 151.57, 148.23, 123.15, 118.36, 117.88, 113.78, 72.40, 56.00, 33.02, 24.04. HRMS (ESI):Calculated for $C_{22}H_{26}N_2O_4$ [[M+Na]$^+$] 405.1790, found 405.1776. Anal. Calcd. (Found): C, 69.09 (69.12); H, 6.85 (6.91); N, 7.32 (7.28)

3-NO_2-Cy (45% yield): ^{1}H NMR (400 MHz, DMSO) δ 15.19 (s, 2H), 8.76 (s, 2H), 8.02 (dd, *J* = 8.0, 1.8 Hz, 2H), 7.67 (dd, *J* =7.7, 1.8 Hz, 2H), 6.69 (t, *J* = 7.8 Hz, 2H), 4.02 – 3.75 (m, 2H), 2.15 – 1.33 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 167.18, 163.12, 140.03, 131.71, 119.75, 113.94 66.50, 63.69, 31.98, 23.91. HRMS (ESI):Calculated for $C_{20}H_{20}N_4O_6$

[[M+Na]$^+$] 435.1281, found 435.1277. Anal. Calcd. (Found): C, 58.25 (58.23); H, 4.89 (4.87); N, 13.59 (13.57).

3-NO$_2$-(R,R)Cy (46% yield): ^{1}H NMR (400 MHz, DMSO) δ 15.19 (s, 2H), 8.76 (s, 2H), 8.01 (dd, *J* = 8.0, 1.8 Hz, 2H), 7.67 (dd, *J* =7.7, 1.8 Hz, 2H), 6.69 (t, *J* = 7.8 Hz, 2H), 4.01 – 3.75 (m, 2H), 2.16 – 1.33 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 167.18, 163.12, 140.03, 131.71, 119.76, 113.94 66.50, 63.69, 31.98, 23.91. HRMS (ESI):Calculated for $C_{20}H_{20}N_4O_6$ [[M+Na]$^+$] 435.1281, found 435.1277. Anal. Calcd. (Found): C, 58.25 (58.29); H, 4.89 (4.91); N, 13.59 (13.55).

3-NO$_2$-(S,S)Cy (48% yield): ^{1}H NMR (400 MHz, DMSO) δ 15.19 (s, 2H), 8.76 (s, 2H), 8.02 (dd, *J* = 8.0, 1.8 Hz, 2H), 7.67 (dd, *J* =7.7, 1.8 Hz, 2H), 6.69 (t, *J* = 7.8 Hz, 2H), 4.02 – 3.76 (m, 2H), 2.17 – 1.35 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 167.18, 163.12, 140.03, 131.71, 119.75, 113.93 66.50, 63.69, 31.98, 23.91. HRMS (ESI):Calculated for $C_{20}H_{20}N_4O_6$ [[M+Na]$^+$] 435.1281, found 435.1277. Anal. Calcd. (Found): C, 58.25 (58.28); H, 4.89 (4.93); N, 13.59 (13.54).

4-NEt$_2$-Cy(76% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.76 (s, 2H), 7.93 (s, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 6.14 – 5.97 (m, 4H), 3.32 (q, *J* = 6.9 Hz, 8H), 3.21 – 3.10 (m, 2H), 2.04 – 1.34 (m, 8H), 1.14 (t, *J* = 7.0 Hz, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.99, 162.74, 151.46, 133.13, 108.20, 102.95, 98.12, 70.79, 44.46, 33.26, 24.36, 12.72. HRMS (ESI):Calculated for $C_{28}H_{40}N_4O_2$ [[M+Na]$^+$] 487.3049, found 487.3054. Anal. Calcd. (Found): C, 72.38 (72.35); H, 8.68 (8.72); N, 12.06 (12.11)

5-OMe-Cy(62% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.81 (s, 2H), 8.19 (s, 2H), 6.88 – 6.79 (m, 4H), 6.64 (d, *J* = 2.7 Hz, 2H), 3.70 (s, 6H), 3.33 – 3.21 (m, 2H), 2.03 – 1.35 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.50, 155.08, 151.98, 119.41, 118.25, 117.48, 114.81, 72.76, 55.87, 33.05, 24.17. HRMS (ESI):Calculated for $C_{22}H_{26}N_2O_4$ [[M+Na]$^+$] 405.1790, found 405.1784. Anal. Calcd. (Found): C, 69.09 (69.11); H, 6.85 (6.89); N, 7.32 (7.35)

5-NO$_2$-Cy(35% yield): ^{1}H NMR (400 MHz, DMSO) δ 14.86 (s, 2H), 8.87 (s, 2H), 8.48 (d, *J* = 3.0 Hz, 2H), 8.14 (dd, *J* = 9.4, 3.0 Hz, 2H), 6.85 (d, *J* = 9.4 Hz, 2H), 4.01 (d, *J* = 4.4 Hz, 2H), 1.97 – 1.50 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 172.49, 166.62, 137.15, 130.43, 129.13, 120.67, 116.28, 64.83, 29.74, 21.90. HRMS (ESI):Calculated for $C_{20}H_{20}N_4O_6$ [[M+Na]$^+$] 435.1281, found 435.1268. Anal. Calcd. (Found): C, 58.25 (58.28); H, 4.89 (4.92); N, 13.59 (13.62)

Naph-Cy (72% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.65 (s, 2H), 8.76 (s, 2H), 7.74 (d, *J* = 8.3 Hz, 2H), 7.53 (d, *J* = 9.2 Hz, 2H), 7.46 (dd, *J* = 7.9, 1.1 Hz, 2H), 7.30 (ddd, *J* = 8.4, 7.0, 1.4 Hz, 2H), 7.13 (ddd, *J* = 8.0, 7.1, 1.0 Hz, 2H), 6.89 – 6.82 (m, 2H), 3.47 – 3.33 (m, 2H), 2.25 – 1.39 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.27, 159.24, 136.50, 133.21, 128.87, 127.84, 126.54, 122.84, 118.43, 107.16, 69.04, 32.68, 24.25. HRMS (ESI):Calculated for $C_{28}H_{26}N_2O_2$ [[M+Na]$^+$] 445.1892, found 445.1888. Anal. Calcd. (Found): C, 79.59 (79.56); H, 6.20 (6.22); N, 6.63 (6.65)

3,5-Cl-Cy (72% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.18 (s, 2H), 8.18 (s, 2H), 7.35 (d, *J* = 2.5 Hz, 2H), 7.08 (d, *J* = 2.5 Hz, 2H), 3.41 – 3.32 (m, 2H), 2.01 – 1.39 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.40, 156.32, 132.35, 129.23, 122.94, 122.64, 119.23, 72.20, 32.88, 23.91. HRMS (ESI):Calculated for $C_{20}H_{18}Cl_4N_2O_2$ [[M+Na]$^+$] 481.0020, found 481.0045. Anal. Calcd. (Found): C, 52.20 (52.24); H, 3.94 (3.91); N, 6.09 (6.07).

3,5-Cl-(R,R)Cy (70% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.18 (s, 2H), 8.18 (s, 2H), 7.36 (d, *J* = 2.5 Hz, 2H), 7.08 (d, *J* = 2.5 Hz, 2H), 3.39 – 3.32 (m, 2H), 2.01 – 1.39 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.40, 156.31, 132.36, 129.23, 122.94, 122.64, 119.23, 72.20, 32.88, 23.91. HRMS (ESI):Calculated for $C_{20}H_{18}Cl_4N_2O_2$ [[M+Na]$^+$] 481.0020, found 481.0066. Anal. Calcd. (Found): C, 52.20 (52.25); H, 3.94 (3.97); N, 6.09 (6.11).

3,5-Cl-(S,S)Cy (74% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.18 (s, 2H), 8.18 (s, 2H), 7.35 (d, *J* = 2.5 Hz, 2H), 7.09 (d, *J* = 2.5 Hz, 2H), 3.41 – 3.33 (m, 2H), 2.01 – 1.41 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.41, 156.33, 132.34, 129.24, 122.93, 122.63, 119.23, 72.19, 32.87, 23.91. HRMS (ESI):Calculated for $C_{20}H_{18}Cl_4N_2O_2$ [[M+Na]$^+$] 481.0020, found 481.0052. Anal. Calcd. (Found): C, 52.20 (52.18); H, 3.94 (3.95); N, 6.09 (6.12).

3,5-NO$_2$-Cy (38% yield): ^{1}H NMR (400 MHz, DMSO) δ 13.54 (s, 2H), 8.92 (s, 2H), 8.72 (d, *J* = 3.1 Hz, 2H), 8.67 (d, *J* = 3.1 Hz, 2H), 4.24 (d, *J* = 8.9 Hz, 2H), 2.22 – 1.35 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 170.16, 168.31, 141.19, 138.06, 130.47, 127.95, 117.55, 63.77, 31.18, 23.86. HRMS (ESI):Calculated for $C_{20}H_{18}N_6O_{10}$ [[M+Na]$^+$] 525.0982, found 525.0976. Anal. Calcd. (Found): C, 47.81 (47.79); H, 3.61 (3.59); N, 16.73 (16.70).

3,5-NO$_2$-(R,R)Cy (37% yield): ^{1}H NMR (400 MHz, DMSO) δ 13.54 (s, 2H), 8.92 (s, 2H), 8.72 (d, *J* = 3.1 Hz, 2H), 8.67 (d, *J* = 3.1 Hz, 2H), 4.24 (d, *J* = 8.9 Hz, 2H), 2.21 – 1.34 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 170.16, 168.31, 141.19, 138.06, 130.47, 127.95, 117.55, 63.78, 31.18, 23.86. HRMS (ESI):Calculated for $C_{20}H_{18}N_6O_{10}$ [[M+Na]$^+$] 525.0982, found 525.0976. Anal. Calcd. (Found): C, 47.81 (47.83); H, 3.61 (3.63); N, 16.73 (16.69).

3,5-NO_2-(S,S)Cy (38% yield): ^{1}H NMR (400 MHz, DMSO) δ 13.54 (s, 2H), 8.92 (s, 2H), 8.72 (d, *J* = 3.1 Hz, 2H), 8.67 (d, *J* = 3.1 Hz, 2H), 4.24 (d, *J* = 8.9 Hz, 2H), 2.22 – 1.34 (m, 8H). ^{13}C NMR (101 MHz, DMSO) δ 170.16, 168.31, 141.19, 138.06, 130.47, 127.95, 117.55, 63.77, 31.18, 23.86. HRMS (ESI):Calculated for $C_{20}H_{18}N_6O_{10}$ [[M+Na]$^+$] 525.0982, found 525.0976. Anal. Calcd. (Found): C, 47.81 (47.80); H, 3.61 (3.65); N, 16.73 (16.76).

diPh (75% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.31 (s, 2H), 8.32 (s, 2H), 7.30 – 7.13 (m, 14H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.80 (td, *J* = 7.5, 1.0 Hz, 2H), 4.75 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.20, 160.96, 139.34, 132.66, 131.80, 128.41, 127.87, 127.68, 118.79, 118.56, 116.93, 80.18. HRMS (ESI):Calculated for $C_{28}H_{24}N_2O_2$ [[M+Na]$^+$] 443.1735, found 443.1728. Anal. Calcd. (Found): C, 79.98 (80.02); H, 5.75 (5.72); N, 6.66 (6.59).

(R,R)diPh (74% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.31 (s, 2H), 8.32 (s, 2H), 7.30 – 7.11 (m, 14H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.80 (td, *J* = 7.5, 0.9 Hz, 2H), 4.75 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.21, 160.96, 139.34, 132.66, 131.80, 128.41, 127.87, 127.68, 118.79, 118.56, 116.93, 80.18. HRMS (ESI):Calculated for $C_{28}H_{24}N_2O_2$ [[M+Na]$^+$] 443.1735, found 443.1731. Anal. Calcd. (Found): C, 79.98 (79.95); H, 5.75 (5.72); N, 6.66 (6.71).

(S,S)diPh (71% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.31 (s, 2H), 8.32 (s, 2H), 7.30 – 7.12 (m, 14H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.80 (td, *J* = 7.5, 1.0 Hz, 2H), 4.75 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.21, 160.97, 139.34, 132.66, 131.80, 128.41, 127.87, 127.68, 118.79, 118.56, 116.93, 80.18. HRMS (ESI):Calculated for $C_{28}H_{24}N_2O_2$ [[M+Na]$^+$] 443.1735, found 443.1735. Anal. Calcd. (Found): C, 79.98 (80.0); H, 5.75 (5.69); N, 6.66 (6.68).

3-F-diPh (63% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.58 (s, 2H), 8.37 (s, 2H), 7.24 – 7.13 (m, 10H), 7.10 (ddd, *J* = 10.9, 8.1, 1.5 Hz, 2H), 6.94 (d, J = 7.9 Hz, 2H), 6.74 (td, *J* = 8.0, 4.5 Hz, 2H), 4.74 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.88, 152.38, 149.95, 149.47, 138.71, 128.55, 127.89, 126.77, 120.24, 118.94, 118.24, 80.13. HRMS (ESI): Calculated for $C_{28}H_{22}F_2N_2O_2$ [[M+Na]$^+$] 479.1547, found 479.1544. Anal. Calcd. (Found): C, 73.67 (73.65); H, 4.86 (4.88); N, 6.14 (6.16).

3-F-(R,R)diPh (65% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.59 (s, 2H), 8.37 (s, 2H), 7.27 – 7.13 (m, 10H), 7.09 (ddd, *J* = 9.8, 8.1, 1.3 Hz, 2H), 6.96 (d, *J* = 7.8 Hz, 2H), 6.73 (td, *J* = 7.9, 4.5 Hz, 2H), 4.74 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.89, 152.38, 149.95, 149.47, 138.72, 128.55, 127.89, 126.77, 120.24, 118.94, 118.24, 80.13. HRMS (ESI): Calculated for $C_{28}H_{22}F_2N_2O_2$ [[M+Na]$^+$] 479.1547, found 479.1565. Anal. Calcd. (Found): C, 73.67 (73.63); H, 4.86 (4.89); N, 6.14 (6.18).

3-F-(S,S)diPh (61% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.59 (s, 2H), 8.37 (s, 2H), 7.27 – 7.13 (m, 10H), 7.09 (ddd, *J* = 9.8, 8.1, 1.3 Hz, 2H), 6.96 (d, *J* = 7.8 Hz, 2H), 6.73 (td, *J* = 7.9, 4.5 Hz, 2H), 4.74 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.88, 152.38, 149.94, 149.47, 138.72, 128.55, 127.89, 126.77, 120.24, 118.94, 118.24, 80.13. HRMS (ESI): Calculated for $C_{28}H_{22}F_2N_2O_2$ [[M+Na]$^+$] 479.1547, found 479.1542. Anal. Calcd. (Found): C, 73.67 (73.67); H, 4.86 (4.92); N, 6.14 (6.10).

3-Cl-diPh (67% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.16 (s, 2H), 8.36 (s, 2H), 7.37 (dd, *J* = 7.9, 1.5 Hz, 2H), 7.25 – 7.13 (m, 2H), 7.11 (dd, *J* = 7.7, 1.5 Hz, 10H), 6.77 (t, *J* = 7.8 Hz, 2H), 4.73 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.65, 156.87, 138.51, 132.93, 130.30, 128.56, 127.94, 127.82, 121.42, 119.28, 119.11, 80.13. HRMS (ESI): Calculated for $C_{28}H_{22}Cl_2N_2O_2$ [[M+Na]$^+$] 511.0956, found 579.0156. Anal. Calcd. (Found): C, 68.91 (68.88); H, 5.18 (5.23); N, 5.54 (5.57).

3-Cl-(R,R)diPh (63% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.16 (s, 2H), 8.36 (s, 2H), 7.37 (dd, *J* = 7.9, 1.5 Hz, 2H), 7.25 – 7.13 (m, 2H), 7.11 (dd, *J* = 7.7, 1.5 Hz, 10H), 6.77 (t, *J* = 7.8 Hz, 2H), 4.73 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.65, 156.87, 138.50, 132.93, 130.30, 128.56, 127.95, 127.82, 121.42, 119.28, 119.11, 80.13. HRMS (ESI): Calculated for $C_{28}H_{22}Cl_2N_2O_2$ [[M+Na]$^+$] 511.0956, found 579.0177. Anal. Calcd. (Found): C, 68.91 (68.94); H, 5.18 (5.14); N, 5.54 (5.58).

3-Cl-(S,S)diPh (65% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.17 (s, 2H), 8.36 (s, 2H), 7.36 (dd, *J* = 7.9, 1.6 Hz, 2H), 7.24 – 7.13 (m, 10H), 7.11 (dd, *J* = 7.7, 1.5 Hz, 2H), 6.77 (t, *J* = 7.8 Hz, 2H), 4.73 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.66, 156.87, 138.50, 132.91, 130.31, 128.56, 127.94, 127.82, 121.42, 119.27, 119.11, 80.13. HRMS (ESI): Calculated for $C_{28}H_{22}Cl_2N_2O_2$ [[M+Na]$^+$] 511.0956, found 579.0978. Anal. Calcd. (Found): C, 68.91 (68.92); H, 5.18 (5.24); N, 5.54 (5.59).

3-NO$_2$-diPh (31% yield**):** ^{1}H NMR (400 MHz, DMSO) δ 15.32 (s, 2H), 8.80 (s, 2H), 8.03 (d, *J* = 7.9 Hz, 2H), 7.90 (d, *J* = 7.8 Hz, 2H), 7.75 – 7.23 (m, 10H), 6.93 (t, *J* = 7.8 Hz, 2H), 5.85 (s, 2H).HRMS (ESI): Calculated for $C_{28}H_{22}N_4O_6$ [[M+Na]$^+$] 533.1437, found 533.1439. Anal. Calcd. (Found): C, 65.88 (65.86); H, 4.34 (4.37); N, 10.97 (10.93).

4-NEt$_2$-diPh(81% yield): ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.85 (s, 2H), 8.07 (s, 2H), 7.23 – 6.04 (m, 16H), 4.60 (s, 2H), 3.34 (q, *J* = 7.1 Hz, 8H), 1.16 (t, *J* = 7.1 Hz, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.32, 163.99, 151.24, 140.54, 133.16, 128.12, 127.93, 127.18, 108.37, 103.07, 97.90, 79.41, 44.51, 12.71. HRMS (ESI): Calculated for $C_{36}H_{42}N_4O_2$ [[M+Na]$^+$]

585.3205, found 585.3199 Anal. Calcd. (Found): C, 76.84 (76.87); H, 7.52 (7.54); N, 9.96 (9.98).

5-NO$_2$-diPh (38% yield**):** ^{1}H NMR (400 MHz, DMSO) δ 14.50 (s, 2H), 8.75 (s, 2H), 8.42 (t, *J* = 7.9 Hz, 2H), 8.16 (dd, *J* = 9.3, 2.9 Hz, 2H), 7.42 – 7.19 (m, 10H), 6.99 (d, *J* = 9.3 Hz, 2H), 5.34 (s, 2H). ^{13}C NMR (101 MHz, DMSO) δ 168.83, 165.99, 139.02, 138.71, 129.03, 128.90, 128.85, 128.34, 128.18, 119.34, 117.69, 75.83. HRMS (ESI): Calculated for $C_{28}H_{22}N_4O_6$ [[M+Na]$^+$] 533.1437, found 533.1446. Anal. Calcd. (Found): C, 65.88 (65.90); H, 4.34 (4.37); N, 10.97 (11.02).

Naph-diPh (67% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 15.24 (s, 2H), 9.00 (s, 2H), 7.82 (d, *J* = 8.4 Hz, 2H), 7.62 (d, *J* = 9.1 Hz, 2H), 7.57 (d, *J* = 7.7 Hz, 2H), 7.40 – 7.18 (m, 14H), 7.03 (d, *J* = 9.1 Hz, 2H), 4.94 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.11, 161.25, 138.62, 135.64, 132.89, 128.97, 128.70, 128.02, 127.69, 127.67, 127.07, 123.07, 121.48, 118.83, 108.04, 78.01. HRMS (ESI):Calculated for $C_{36}H_{28}N_2O_2$ [[M+Na]$^+$] 543.2048, found 543.2042. Anal. Calcd. (Found): C, 83.05 (83.08); H, 5.42 (5.45); N, 5.38 (5.35)

3,5-Cl-diPh (62% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.06 (s, 2H), 8.27 (s, 2H), 7.39 (d, *J* = 2.5 Hz, 2H), 7.24 – 7.12 (m, 10H), 7.10 (d, *J* = 2.5 Hz, 2H), 4.76 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.69, 155.72, 138.06, 132.64, 129.49, 128.70, 128.19, 127.73, 123.40, 122.57, 119.44, 79.98. HRMS (ESI): Calculated for $C_{28}H_{20}Cl_4N_2O_2$ [[M+Na]$^+$] 579.0102, found 579.0156. Anal. Calcd. (Found): C, 60.24 (60.28); H, 3.61 (3.59); N, 5.02 (5.05).

3,5-Cl-(R,R)diPh (64% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.06 (s, 2H), 8.27 (s, 2H), 7.38 (d, *J* = 2.4 Hz, 2H), 7.25 – 7.11 (m, 10H), 7.09 (d, *J* = 2.5 Hz, 2H), 4.76 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.69 (s), 155.72, 138.05, 132.64, 129.49, 128.70, 128.19, 127.73, 123.40, 122.55, 119.44, 79.98. HRMS (ESI): Calculated for $C_{28}H_{20}C_{l4}N_2O_2$ [[M+Na]$^+$] 579.0177, found 579.0165. Anal. Calcd. (Found): C, 60.24 (60.20); H, 3.61 (3.64); N, 5.02 (5.08).

3,5-Cl-(S,S)diPh (60% yield**):** ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.06 (s, 2H), 8.27 (s, 2H), 7.38 (d, *J* = 2.5 Hz, 2H), 7.25 – 7.12 (m, 10H), 7.09 (d, *J* = 2.5 Hz, 2H), 4.76 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.69, 155.72, 138.05, 132.64, 129.49, 128.70, 128.19, 127.73, 123.40, 122.55, 119.44, 79.98. HRMS (ESI): Calculated for $C_{28}H_{20}C_{l4}N_2O_2$ [[M+Na]$^+$] 579.0177, found 579.0165. Anal. Calcd. (Found): C, 60.24 (60.26); H, 3.61 (3.58); N, 5.02 (5.06).

3,5-NO_2-diPh (31% yield**):** ^{1}H NMR (400 MHz, DMSO) δ 14.13 (s, 2H), 8.80 (s, 2H), 8.67 – 8.54 (m,12H), 8.28 (d, *J* = 3.3 Hz, 2H), 5.72 (s, 2H).HRMS (ESI): Calculated for $C_{28}H_{20}N_6O_{10}$ [[M+Na]$^+$] 623.1139, found 623.1151. Anal. Calcd. (Found): C, 56.00 (56.05); H, 3.36 (3.40); N, 14.00 (14.03).

Table. S1 Photophysical data of **Cys**. Sample without emission data mean that it is non-emissive.

	medium	λ_{abs} /nm (ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Stokes shift /nm	Φ	f /%
Cy	MeCN	253(2.37×10^4); 316(9.00×10^3)	457		0.0023	
	Water	338; 394	495	101	0.015	90
	Solid		502		0.018	
(R,R)Cy	MeCN	253(2.31×10^4); 316(9.10×10^3)	457		0.0021	
	Water	338; 394	496	102	0.014	90
	Solid		503		0.016	
(S,S)Cy	MeCN	253(2.42×10^4); 316(8.90×10^3)	457		0.0026	
	Water	338; 394	495	101	0.016	90
	Solid		502		0.018	
3-F-Cy	MeCN	253(2.51×10^4); 316(5.30×10^3)	458		0.0088	
	Water	372	501	129	0.020	90
	Solid		509		0.060	
3-F-(R,R)Cy	MeCN	253(2.54×10^4); 316(5.30×10^3)	458		0.0090	
	Water	372	502	130	0.022	90
	Solid		509		0.059	
3-F-(S,S)Cy	MeCN	253(2.47×10^4); 316(5.20×10^3)	458		0.0093	
	Water	373	502	129	0.023	90
	Solid		508		0.062	
3-Cl-Cy	MeCN	256(2.15×10^4); 323(6.60×10^3)	461		0.0086	
	Water	383	506	123	0.025	
	Solid		520		0.14	90
3-Cl-(R,R)Cy	MeCN	257(2.09×10^4); 323(6.80×10^3)	460		0.0088	
	Water	383	506	123	0.025	
	Solid		520		0.14	90
3-Cl-(S,S)Cy	MeCN	256(2.14×10^4); 323(6.20×10^3)	461		0.0090	

	Water	384	506	122	0.028	
	Solid		520		0.15	90
3-NO$_2$-Cy	MeCN	346(6.60×10^3); 436(1.17×10^4)	502		0.00092	
	Solid		565		0.051	
3-NO$_2$-(R,R)Cy	MeCN	346(6.40×10^3); 436(1.12×10^4)	502		0.00087	
	Solid		565		0.049	
3-NO$_2$-(S,S)Cy	MeCN	346(6.20×10^3); 436(1.09×10^4)	502		0.0010	
	Solid		565		0.053	
3-OMe-Cy	MeCN	259(2.59×10^4); 332(5.70×10^3)	458		0.0053	
	Solid		523		0.0085	
3-*t*-Bu-Cy	MeCN	258(2.46×10^4); 323(1.11×10^4)	457		0.00099	
	Solid		495		0.0012	
4-NEt$_2$-Cy	MeCN	323(5.97×10^4)	458		0.00056	
	Solid		519		0.038	
5-OMe-Cy	MeCN	255(1.53×10^4); 345(9.30×10^3)	457		0.0023	
	Water	257; 359	510	151	0.0037	80
	Solid		548		0.026	
5-NO$_2$-Cy	MeCN	323(1.75×10^4); 403(1.24×10^4)	482		0.0013	
	Water	325; 401	516	115	0.0015	90
	Solid		502		0.14	
Naph-Cy	MeCN	307(1.93×10^4); 401(1.07×10^4); 422(1.06×10^4)	457		0.00018	
	Solid		486		0.0053	
3,5-Cl-Cy	MeCN	257(1.97×10^4); 336(7.40×10^3)	486		0.010	
	Water	394	511	117	0.064	90
	Solid		532		0.32	
3,5-Cl-(R,R)Cy	MeCN	254(2.18×10^4); 336(7.80×10^3)	486		0.011	
	Water	395	511	116	0.066	90

	Solid		532		0.33	
3,5-Cl-(S,S)Cy	MeCN	257(1.84×10^4); 336(6.70×10^3)	486		0.010	
	Water	394	511	117	0.068	90
	Solid		532		0.35	
3,5-NO_2-Cy	MeCN	396(3.39×10^4)	487		0.00039	
	Solid		550		0.0016	
3,5-NO_2-(R,R)Cy	MeCN	396(3.60×10^4)	487		0.00043	
	Solid		549		0.0016	
3,5-NO_2-(S,S)Cy	MeCN	396(3.63×10^4)	487		0.00040	
	Solid		550		0.0019	

Table. S2 Photophysical data of **diPhs**. Sample without emission data mean that it is non-emissive.

	medium	λ_{abs} /nm (ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Stokes shift /nm	Φ	*f* /%
diPh	MeCN	255(4.06×10^4); 318(1.29×10^4)	433		0.00035	
	Water	256; 318	437	119	0.0058	60
	Solid		507		0.10	
(R,R)diPh	MeCN	257(3.44×10^4); 316(1.17×10^4)	433		0.00032	
	Water	257; 316	437	121	0.0056	60
	Solid		507		0.12	
(S,S)diPh	MeCN	256(3.61×10^4); 318(1.03×10^4)	432		0.00027	
	Water	257; 318	436	118	0.0058	70
	Solid		506		0.098	
3-F-diPh	MeCN	255(4.10×10^4); 318(8.16×10^3)	434		0.00059	
	Water	255; 318	504	186	0.0089	60
	Solid		512		0.12	
3-F-(R,R)diPh	MeCN	254(4.59×10^4); 319(7.76×10^3)	434		0.00072	
	Water	254; 318	504	186	0.0088	70
	Solid		511		0.12	
3-F-(S,S)diPh	MeCN	254(4.10×10^4); 319(6.84×10^3)	432		0.00051	
	Water	254; 319	504	185	0.0096	60
	Solid		512		0.14	
3-Cl-diPh	MeCN	257(3.71×10^4); 323(9.11×10^3)	447		0.00094	
	Water	258; 323	506	183	0.0113	70
	Solid		524		0.16	
3-Cl-(R,R)diPh	MeCN	257(3.77×10^4); 323(9.24×10^3)	446		0.0011	
	Water	258; 323	506	183	0.011	60
	Solid		524		0.16	
3-Cl-(S,S)diPh	MeCN	258(3.73×10^4); 325(9.00×10^3)	447		0.00081	

	Water	259; 323	506	181	0.013	70
	Solid		522		0.18	
3-NO$_2$-diPh	MeCN	348(1.22×10^4); 434(6.20×10^3)	503		<0.00010	
	Water	429	511	182	0.00011	95
	Solid		522		0.020	
4-NEt$_2$-diPh	MeCN	331(6.23×10^4)	432		0.00018	
	Solid		492		0.032	
5-NO$_2$-diPh	MeCN	318(2.31×10^4); 391(1.18×10^3)	457		0.00022	
	Solid		500; 555		0.074	
Naph-diPh	MeCN	312(6.35×10^4); 359(3.60×10^4); 402(2.05×10^4); 423(1.86×10^4)	454		0.00021	
	Solid		474		0.021	
3,5-Cl-diPh	MeCN	258(3.30×10^4); 335(9.21×10^3)	490		0.0016	
	Water	258; 335	508	173	0.072	40
	Solid		534		0.20	
3,5-Cl-(R,R)diPh	MeCN	257(3.33×10^4); 335(9.08×10^3)	487		0.0016	
	Water	258; 335	509	174	0.070	40
	Solid		534		0.20	
3,5-Cl-(S,S)diPh	MeCN	259(3.26×10^4); 335(9.24×10^3)	491		0.0018	
	Water	259; 335	508	173	0.075	30
	Solid		536		0.22	
3,5-NO$_2$-diPh	MeCN	369(2.15×10^4)	430		<0.00010	
	Solid		556		0.012	

Table. S3 Photophysical data of anion probes. The amount of X^- is 100 equivalent to Salen ligands. Solvent is DMSO. Sample without emission data means that it is non-emissive.

diPhs+X^-	λ_{abs} /nm(ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Φ	I/I_0
Cy	318(5.50×10^3); 415(1.80×10^3)	440	0.0043	
Cy+F^-	316(1.71×10^4); 390(8.80×10^3)	454	0.077	199
Cy+PO_4^{3-}	391(3.40×10^3)	457	0.077	177
Cy+OH^-	262(2.18×10^4); 370(1.41×10^4)	456	0.088	199
Cy+CO_3^{2-}	271(2.40×10^4); 373(1.23×10^4)	467	0.065	132
Cy+SO_4^{2-}	262(1.67×10^4); 357(5.30×10^3)	439	0.053	89.2
3-F-Cy	318(5.50×10^3); 415(1.70×10^3)	467	0.0073	
3-F-Cy+F^-	262(4.40×10^4); 390(3.26×10^4)	475	0.25	1087
3-F-Cy+$P_2O_7^{4-}$	260(1.26×10^4); 389(1.82×10^4)	469	0.14	686
3-F-Cy+PO_4^{3-}	258(8.45×10^4); 387(1.14×10^4)	449	0.24	1219
3-F-Cy+OH^-	262(6.32×10^4); 376(2.22×10^4)	442	0.27	1535
3-F-Cy+CO_3^{2-}	271(3.97×10^4); 378(1.46×10^4)	464	0.083	397
3-F-Cy+S^{2-}	268(3.02×10^5)	444	0.23	1287
3-Cl-Cy	260(5.22×10^3); 421(3.90×10^3)	458	0.0080	
3-Cl-Cy+F^-	264(7.24×10^4); 394(2.20×10^4)	448	0.14	68.6
3-Cl-Cy+PO_4^{3-}	258(9.49×10^4); 395(1.75×10^4)	449	0.13	48.6
3-Cl-Cy+OH^-	264(5.83×10^4); 386(2.08×10^4)	447	0.13	67.6
3-Cl-Cy+CO_3^{2-}	264(6.68×10^4); 395(2.42×10^4)	458	0.044	33.3
3-Cl-Cy+S^{2-}	265(3.42×10^5)	450	0.099	61.8
3,5-Cl-Cy	263(5.24×10^4); 425(7.20×10^3)	482	0.011	

3,5-Cl-Cy+F^-	262(7.55×10^4); 403(2.31×10^4)	456	0.15	27.8
3,5-Cl-Cy+PO_4^{3-}	262(6.98×10^4); 407(1.93×10^4)	459	0.18	26.3
3,5-Cl-Cy+OH^-	259(8.42×10^4); 399(2.07×10^4)	456	0.16	28.6
3,5-Cl-Cy+CO_3^{2-}	272(6.15×10^4); 404(2.57×10^4)	460	0.12	23.2
3,5-Cl-Cy+S^{2-}	274(2.49×10^5)	458	0.14	30.7
diPh	260(4.12×10^3); 408(1.20×10^3)	430	0.0014	
diPh+F^-	264(3.71×10^4); 376(1.82×10^4)	475	0.0025	36.2
diPh+OH^-	261(3.95×10^4); 364(1.23×10^4)	462	0.026	163
diPh+CO_3^{2-}	264(5.21×10^4); 365(1.85×10^4)	470	0.0056	33.6
diPh+PO_4^{3-}	266(3.83×10^4); 379(1.76×10^4)	450	0.014	96.7
diPh+S^{2-}	273(2.78×10^5)	455	0.0096	72.2
3-F-diPh	260(5.22×10^3); 420(1.00×10^3)	430	0.0072	
3-F-diPh+F^-	265(4.69×10^4); 385(2.62×10^4)	451	0.19	1676
3-F-diPh+OH^-	260(4.36×10^4); 372(1.45×10^4)	452	0.23	1077
3-F-diPh+CO_3^{2-}	264(5.53×10^4); 372(2.04×10^4)	460	0.075	450
3-F-diPh+PO_4^{3-}	266(4.24×10^4); 385(1.97×10^4)	452	0.24	1733
3-F-diPh+S^{2-}	270(3.54×10^5)	451	0.082	612
3-Cl-diPh	318(5.50×10^3); 415(1.70×10^3)	450	0.021	
3-Cl-diPh+F^-	265(5.03×10^4); 392(3.41×10^4)	456	0.20	421
3-Cl-diPh+$P_2O_7^{4-}$	259(4.74×10^4); 390(9.70×10^3)	442	0.25	424
3-Cl-diPh+OH^-	260(4.58×10^4); 378(1.66×10^4)	453	0.16	283
3-Cl-diPh+CO_3^{2-}	264(5.46×10^4); 379(2.32×10^4)	466	0.099	156
3-Cl-diPh+PO_4^{3-}	265(4.66×10^4); 393(2.12×10^4)	459	0.22	366

3,5-Cl-diPh	264(2.31×10^4); 336 (6.90×10^3); 433(3.20×10^3)	485	0.064	
3,5-Cl-diPh+F^-	264(5.23×10^4); 402(3.14×10^4)	416; 466	0.24	41.1
3,5-Cl-diPh+OH^-	260(4.85×10^4); 389(1.87×10^4)	411; 458	0.29	36.4
3,5-Cl-diPh+CO_3^{2-}	264(5.88×10^4); 388(3.12×10^4)	477	0.23	27.6
3,5-Cl-diPh+PO_4^{3-}	265(5.02×10^4); 404(2.43×10^4)	416; 457	0.28	29.4
3,5-Cl-diPh+S^{2-}	288(3.67×10^5)	464	0.22	36.9

Table. S4 Photophysical data of C_2, **Cy** and **diPh** DMSO solution upon the addition of 100 equivalent of different L-amino acids. Sample without emission data means that it is non-emissive. I_0 and I represents the fluorescence emission intensity of receptor in the absence and presence of L-amino acid, respectively.

Dyes+amino acid	λ_{abs} /nm(ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Φ	I/I_0
C_2	263(2.43×10^4); 320(8.00×10^3)	432	0.0068	
C_2+L-Ala	261(1.11×10^4); 319(7.50×10^3); 411(4.40×10^3)	434	0.025	27.8
C_2+L-Pro	261(1.24×10^4); 323(5.40×10^3); 409(7.10×10^3)	430	0.0071	1.56
C_2+L-Glu	263(7.10×10^3); 322(7.30×10^3)	441	0.048	12.5
C_2+L-Gln	264(9.90×10^3); 319(7.40×10^3); 410(3.00×10^3)	430	0.042	31.5
C_2+L-Val	265(1.03×10^4); 321(5.80×10^3)	432	0.0075	2.15
C_2+L-Arg	324(6.00×10^3)	431	0.0092	5.15
C_2+L-Leu	264(1.36×10^4); 316(8.40×10^3); 410(5.00×10^3)	429	0.032	39.9
C_2+L-Trp	258(>1.00×10^6); 410(2.20×10^3)	432	0.034	26.0
C_2+L-Ser	267(8.3×10^3); 318(7.20×10^3);410(1.90×10^3)	433	0.048	26.5
C_2+L-His	268(2.80×10^3); 320(4.80×10^3)	432	0.028	13.9
Cy	318(5.50×10^3); 415(5.80×10^3)	440	0.0011	
Cy+L-Ala	267(3.42×10^4); 424(5.80×10^3)	438	0.0088	13.4
Cy+L-Pro	271(3.47×10^4); 424(6.10×10^3)	489	0.0048	15.3
Cy+L-Glu	272(3.42×10^4); 428(2.00×10^3)	442	0.014	21.6
Cy+L-Gln	266(3.76×10^3); 423(6.20×10^3)	439	0.0068	19.4
Cy+L-Val	267(3.58×10^4); 419(5.70×10^3)	433	0.012	22.7
Cy+L-Arg	265(4.21×10^4); 402(6.70×10^3)	445	0.013	27.1
Cy+L-Leu	266(3.43×10^4); 424(4.30×10^3)	438	0.01	17.6
Cy+L-Trp	297(>1.00×10^6); 380(7.30×10^3)	433	0.0075	16.7
Cy+L-Ser	269(3.38×10^4); 426(4.60×10^3)	447	0.0078	32.5

Cy+L-His	266(3.32×10^3); 424(3.60×10^3)	433	0.0088	16.8
diPh	260(4.12×10^3); 408(1.20×10^3)	430	0.0014	
diPh+L-Ala	260(3.79×10^4)	431	0.0068	24.8
diPh+L-Pro	271(3.54×10^4)	435	0.0013	1.11
diPh+L-Glu	268(3.72×10^4)	449	0.0057	20.7
diPh+L-Gln	266(3.69×10^4)	428	0.0060	21.9
diPh+L-Val	265(4.01×10^4); 397(1.30×10^3)	433	0.0054	19.7
diPh+L-Arg	266(3.68×10^4); 373(1.40×10^3)	430	0.045	116
diPh+L-Leu	266(3.21×10^4)	429	0.0065	23.4
diPh+L-Trp	288(>1.00×10^6); 297(2.72×10^5)	431	0.016	56.1
diPh+L-Ser	266(4.11×10^4)	426	0.0015	0.97
diPh+L-His	266(4.53×10^4)	428	0.0012	1.03

Table. S5 Photophysical data of **3-F-(R,R)Cy** and **3-F-(S,S)Cy** DMSO solution upon the addition of 100 equivalent of different D- or L-amino acids. Sample without emission data means that it is non-emissive. *ef* = (I_D-I_0)/(I_L-I_0).

3-F-Cy+amino acid	λ_{abs} /nm(ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Φ	I/I_0	*ef*
3-F-(R,R)Cy	318(5.50×10^{3}); 415(1.70×10^{3})	457	0.0061		
3-F-(R,R)Cy+L-Ala	282(9.40×10^{3}); 409(6.50×10^{3})	462	0.031	70.5	0.94
3-F-(R,R)Cy+D-Ala	281(9.40×10^{3}); 409(6.40×10^{3})	462	0.047	66.3	
3-F-(R,R)Cy+L-Pro	264(1.07×10^{4}); 324(5.40×10^{3}); 409(2.60×10^{3})	478	0.11	104	0.89
3-F-(R,R)Cy+D-Pro	264(1.48×10^{4}); 322(5.10×10^{3}); 409(2.00×10^{3})	479	0.12	92.6	
3-F-(R,R)Cy+L-Glu	265(1.63×10^{4}); 322(5.50×10^{3}); 409(3.40×10^{3})	433	0.043	48.0	1.17
3-F-(R,R)Cy+D-Glu	264(1.92×10^{4}); 321(5.50×10^{3}); 409(3.20×10^{3})	435	0.049	56.2	
3-F-(R,R)Cy+L-Gln	276(1.24×10^{4}); 409(9.80×10^{3})	429	0.038	84.1	1.58
3-F-(R,R)Cy+D-Gln	279(1.68×10^{4}); 413(1.07×10^{4})	430	0.050	132	
3-F-(R,R)Cy+L-Val	284(1.33×10^{4}); 410(8.40×10^{3})	434	0.035	85.1	0.66
3-F-(R,R)Cy+D-Val	279(1.19×10^{4}); 408(8.40×10^{3})	431	0.024	56.2	
3-F-(R,R)Cy+L-Arg	278(1.98×10^{4}); 406(1.13×10^{4})	439	0.14	407	1.07
3-F-(R,R)Cy+D-Arg	278(1.58×10^{4}); 409(1.04×10^{4})	454	0.16	436	
3-F-(R,R)Cy+L-Leu	278(1.62×10^{4}); 412(1.06×10^{4})	436	0.038	102	1.29
3-F-(R,R)Cy+D-Leu	279(1.19×10^{4}); 414(8.20×10^{3})	427	0.055	131	
3-F-(R,R)Cy+L-Trp	286(>1.00×10^{6})	430	0.037	83.6	1.19
3-F-(R,R)Cy+D-Trp	283(>1.00×10^{6})	432	0.038	99.5	
3-F-(R,R)Cy+L-Ser	278(1.14×10^{4}); 412(7.70×10^{3})	447	0.039	72.9	1.27
3-F-(R,R)Cy+D-Ser	279(1.28×10^{4}); 413(9.30×10^{3})	437	0.036	92.6	
3-F-(R,R)Cy+L-His	278(1.19×10^{4}); 412(8.20×10^{3})	437	0.062	124	1.06
3-F-(R,R)Cy+D-His	279(1.45×10^{4}); 410(1.10×10^{4})	436	0.048	132	
3-F-(S,S)Cy	318(5.60×10^{3}); 415(1.90×10^{3})	456	0.0067		

3-F-(S,S)Cy+L-Ala	281(1.51×10^{4}); 409(1.24×10^{4})	461	0.039	62.8	1.45
3-F-(S,S)Cy+D-Ala	281(1.27×10^{4}); 408(1.08×10^{4})	462	0.061	90.4	
3-F-(S,S)Cy+L-Pro	264(1.21×10^{4}); 324(5.20×10^{3}); 409(2.50×10^{3})	475	0.20	113	0.83
3-F-(S,S)Cy+D-Pro	264(1.05×10^{4}); 319(4.50×10^{3}); 409(2.90×10^{3})	476	0.16	94.0	
3-F-(S,S)Cy+L-Glu	266(1.11×10^{4}); 321(5.20×10^{3}); 411(3.80×10^{3})	437	0.033	21.0	0.67
3-F-(S,S)Cy+D-Glu	265(9.70×10^{3}); 319(4.70×10^{3}); 413 (3.10×10^{3})	436	0.023	14.3	
3-F-(S,S)Cy+L-Gln	281(1.30×10^{4}); 412(1.02×10^{4})	431	0.052	104	1.14
3-F-(S,S)Cy+D-Gln	279(1.09×10^{4}); 413(8.20×10^{3})	429	0.080	119	
3-F-(S,S)Cy+L-Val	284(1.33×104); 412(1.02×10^{4})	435	0.016	26.7	1.48
3-F-(S,S)Cy+D-Val	276(1.03×104); 411(7.60×10^{3})	431	0.028	39.1	
3-F-(S,S)Cy+L-Arg	276(6.42×10^{4}); 407(3.31×10^{4})	470	0.018	86.8	1.09
3-F-(S,S)Cy+D-Arg	279(5.95×10^{4}); 409(3.18×10^{4})	464	0.015	94.6	
3-F-(S,S)Cy+L-Leu	270(1.33×10^{4}); 412(3.20×10^{3})	428	0.17	65.1	0.44
3-F-(S,S)Cy+D-Leu	279(1.18×10^{4}); 411(9.60×10^{3})	430	0.060	29.3	
3-F-(S,S)Cy+L-Trp	290($>1.00\times10^{6}$)	431	0.021	21.6	1.59
3-F-(S,S)Cy+D-Trp	291($>1.00\times10^{6}$)	437	0.040	33.7	
3-F-(S,S)Cy+L-Ser	277(1.02×10^{4}); 413(7.80×10^{3})	450	0.022	27.9	0.80
3-F-(S,S)Cy+D-Ser	277(1.19×10^{4}); 412(9.20×10^{3})	437	0.015	22.6	
3-F-(S,S)Cy+L-His	282(1.17×10^{4}); 411(9.20×10^{3})	435	0.041	58.2	0.88
3-F-(S,S)Cy+D-His	281(1.42×10^{4}); 414(1.10×10^{4})	427	0.031	51.2	

Table. S6 Photophysical data of **3-Cl-(R,R)Cy** and **3-Cl-(S,S)Cy** DMSO solution upon the addition of 100 equivalent of different D- or L-amino acids. Sample without emission data means that it is non-emissive. *ef* = $(I_D-I_0)/(I_L-I_0)$.

3-Cl-Cy+amino acid	λ_{abs} /nm(ε/dm³ mol⁻¹cm⁻¹)	λ_{em} /nm	Φ	I/I_0	*ef*
3-Cl-(R,R)Cy	260(5.22×10³); 421(3.90×10³)	458	0.0076		
3-Cl-(R,R)Cy+L-Ala	271(3.57×10⁴); 417(6.90×10³)	429	0.11	19.1	0.75
3-Cl-(R,R)Cy+D-Ala	269(3.75×10⁴); 416(1.22×10⁴)	434	0.051	14.5	
3-Cl-(R,R)Cy+L-Pro	268(3.25×10⁴); 413(6.20×10³)	479	0.13	15.7	0.95
3-Cl-(R,R)Cy+D-Pro	265(3.70×10⁴); 411(6.10×10³)	475	0.11	14.9	
3-Cl-(R,R)Cy+L-Glu	267(1.38×10⁴); 324(7.60×10³); 413(7.30×10³)	432	0.023	6.51	0.82
3-Cl-(R,R)Cy+D-Glu	268(3.83×10⁴); 421(6.00×10³)	431	0.035	5.54	
3-Cl-(R,R)Cy+L-Gln	285(1.28×10⁴); 417(1.28×10⁴)	427	0.081	22.7	0.54
3-Cl-(R,R)Cy+D-Gln	276(3.60×10⁴); 414(1.23×10⁴)	432	0.049	12.5	
3-Cl-(R,R)Cy+L-Val	283(1.55×10⁴); 417(1.69×10⁴)	433	0.015	5.15	0.97
3-Cl-(R,R)Cy+D-Val	283(1.47×10⁴); 418(1.64×10⁴)	434	0.015	5.05	
3-Cl-(R,R)Cy+L-Arg	274(3.86×10⁴); 411(1.25×10⁴)	434	0.091	30.4	0.89
3-Cl-(R,R)Cy+D-Arg	275(3.87×10⁴); 417(1.14×10⁴)	437	0.090	27.1	
3-Cl-(R,R)Cy+L-Leu	277(3.80×10⁴); 417(1.45×10⁴)	434	0.020	5.99	1.03
3-Cl-(R,R)Cy+D-Leu	274(3.72×10⁴); 418(1.33×10⁴)	430	0.024	6.14	
3-Cl-(R,R)Cy+L-Trp	272(>1.00×10⁶); 294(>1.00×10⁶)	424	0.017	5.65	1.25
3-Cl-(R,R)Cy+D-Trp	273(>1.00×10⁶); 288(>1.00×10⁶)	423	0.025	6.79	
3-Cl-(R,R)Cy+L-Ser	280(4.20×10⁴); 418(1.44×10⁴)	435	0.015	4.81	1.80
3-Cl-(R,R)Cy+D-Ser	282(3.96×10⁴); 418(1.56×10⁴)	430	0.023	7.78	
3-Cl-(R,R)Cy+L-His	278(3.71×10⁴); 419(1.22×10⁴)	426	0.035	7.99	0.87
3-Cl-(R,R)Cy+D-His	281(4.16×10⁴); 418(1.61×10⁴)	428	0.020	7.10	
3-Cl-(S,S)Cy	260(5.43×10³); 421(4.10×10³)	458	0.0081		

3-Cl-(S,S)Cy+L-Ala	282(8.50×10^3); 417(1.09×10^4)	424	0.043	6.83	1.18
3-Cl-(S,S)Cy+D-Ala	285(1.07×10^4); 417(1.24×10^4)	434	0.037	7.88	
3-Cl-(S,S)Cy+L-Pro	262(1.24×10^4); 327(6.00×10^3); 411(8.20×10^3)	478	0.066	11.0	1.37
3-Cl-(S,S)Cy+D-Pro	265(9.40×10^3); 327(5.10×10^3); 406(4.40×10^3)	476	0.14	14.6	
3-Cl-(S,S)Cy+L-Glu	261(1.25×10^4); 331(4.50×10^3); 417(5.00×10^3)	430	0.041	5.60	1.01
3-Cl-(S,S)Cy+D-Glu	265(1.13×10^4); 326(7.00×10^3); 414(6.80×10^3)	428	0.032	5.65	
3-Cl-(S,S)Cy+L-Gln	285(1.06×10^4); 418(1.28×10^4)	430	0.033	6.19	1.23
3-Cl-(S,S)Cy+D-Gln	283(8.20×10^3); 418(1.09×10^4)	426	0.041	7.42	
3-Cl-(S,S)Cy+L-Val	286(1.62×10^4); 411(1.54×10^4)	435	0.020	5.94	1.57
3-Cl-(S,S)Cy+D-Val	285(8.80×10^3); 418(1.08×10^3)	436	0.046	8.78	
3-Cl-(S,S)Cy+L-Arg	278(8.10×10^3); 412(9.90×10^3)	430	0.12	25.2	1.03
3-Cl-(S,S)Cy+D-Arg	283(1.55×10^4); 417(1.35×10^4)	441	0.090	25.8	
3-Cl-(S,S)Cy+L-Leu	285(1.42×10^4); 411(1.54×10^4)	430	0.022	6.27	0.74
3-Cl-(S,S)Cy+D-Leu	281(1.18×10^4); 411(1.28×10^4)	428	0.019	4.90	
3-Cl-(S,S)Cy+L-Trp	264(>1.00×10^6); 293(>1.00×10^6)	431	0.015	2.93	0.68
3-Cl-(S,S)Cy+D-Trp	267(>1.00×10^6); 273(>1.00×10^6)	432	0.012	2.33	
3-Cl-(S,S)Cy+L-Ser	282(8.50×10^3); 412(1.05×10^4)	434	0.050	8.53	0.55
3-Cl-(S,S)Cy+D-Ser	285(1.58×10^4); 413(1.68×10^4)	439	0.021	5.11	
3-Cl-(S,S)Cy+L-His	286(8.80×10^3); 418(1.08×10^4)	432	0.013	2.77	2.95
3-Cl-(S,S)Cy+D-His	285(1.43×10^4); 418(1.26×10^4)	431	0.027	6.24	

Table. S7 Photophysical data of **3,5-Cl-(R,R)Cy** and **3,5-Cl-(S,S)Cy** DMSO solution upon the addition of 100 equivalent of different D- or L-amino acids. Sample without emission data means that it is non-emissive. *ef* = (I_D-I_0)/(I_L-I_0).

3,5-Cl-Cy+amino acid	λ_{abs} /nm(ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Φ	I/I_0	*ef*
3,5-Cl-(R,R)Cy	263(5.24×10^4); 425(7.20×10^3)	482	0.011		
3,5-Cl-(R,R)Cy+L-Ala	274(3.25×10^4); 425(1.71×10^4)	488	0.035	3.17	1.02
3,5-Cl-(R,R)Cy+D-Ala	271(3.71×10^4); 427(1.59×10^4)	486	0.045	3.21	
3,5-Cl-(R,R)Cy+L-Pro	270(3.68×10^4); 427(1.42×10^4)	488	0.12	7.99	0.95
3,5-Cl-(R,R)Cy+D-Pro	270(3.79×10^4); 426(1.32×10^4)	483	0.13	7.61	
3,5-Cl-(R,R)Cy+L-Glu	270(3.68×10^4); 425(1.46×10^4)	436	0.035	2.43	0.96
3,5-Cl-(R,R)Cy+D-Glu	270(3.79×10^4); 427(1.32×10^4)	435	0.036	2.36	
3,5-Cl-(R,R)Cy+L-Gln	268(3.88×10^4); 425(1.71×10^4)	434	0.066	4.48	1.63
3,5-Cl-(R,R)Cy+D-Gln	270(3.97×10^4); 425(1.60×10^4)	442	0.075	7.16	
3,5-Cl-(R,R)Cy+L-Val	270(4.30×10^4); 427(1.97×10^4)	430	0.035	2.81	1.75
3,5-Cl-(R,R)Cy+D-Val	277(3.96×10^4); 427(1.96×10^4)	436	0.044	4.71	
3,5-Cl-(R,R)Cy+L-Arg	270(3.78×10^4); 428(1.44×10^4)	482	0.10	6.67	0.97
3,5-Cl-(R,R)Cy+D-Arg	269(4.08×10^4); 425(1.39×10^4)	482	0.072	6.56	
3,5-Cl-(R,R)Cy+L-Leu	275(4.00×10^4); 426(1.62×10^4)	435	0.059	5.70	0.55
3,5-Cl-(R,R)Cy+D-Leu	275(3.71×10^4); 428(1.65×10^4)	436	0.051	3.28	
3,5-Cl-(R,R)Cy+L-Trp	263(>1.00×10^6); 267(>1.00×10^6)	425	0.041	4.33	0.53
3,5-Cl-(R,R)Cy+D-Trp	275(>1.00×10^6); 280(>1.00×10^6)	432	0.031	2.80	
3,5-Cl-(R,R)Cy+L-Ser	271(3.61×10^4); 425(1.56×10^4)	440	0.087	6.65	0.96
3,5-Cl-(R,R)Cy+D-Ser	270(4.10×10^4); 427(1.74×10^4)	436	0.068	6.40	
3,5-Cl-(R,R)Cy+L-His	268(4.11×10^4); 427(1.31×10^4)	430	0.034	2.22	3.16
3,5-Cl-(R,R)Cy+D-His	270(4.06×10^4); 425(9.80×10^3)	434	0.071	6.80	
3,5-Cl-(S,S)Cy	264(5.35×10^4); 425(7.30×10^3)	482			

3,5-Cl-(S,S)Cy+L-Ala	270(3.91×10^4); 425(1.84×10^4)	433	0.055	2.82	1.09
3,5-Cl-(S,S)Cy+D-Ala	274(4.07×10^4); 425(1.53×10^4)	436	0.068	3.05	
3,5-Cl-(S,S)Cy+L-Pro	267(3.68×10^4); 426(1.99×10^4)	488	0.068	8.43	0.80
3,5-Cl-(S,S)Cy+D-Pro	266(3.91×10^4); 426(1.38×10^4)	488	0.099	6.87	
3,5-Cl-(S,S)Cy+L-Glu	268(3.68×10^4); 425(1.28×10^4)	437	0.036	2.01	0.95
3,5-Cl-(S,S)Cy+D-Glu	276(3.78×10^4); 426(1.13×10^4)	432	0.044	1.93	
3,5-Cl-(S,S)Cy+L-Gln	265(3.91×10^4); 427(1.72×10^4)	434	0.059	3.09	1.63
3,5-Cl-(S,S)Cy+D-Gln	264(4.22×10^4); 428(1.76×10^4)	434	0.059	4.88	
3,5-Cl-(S,S)Cy+L-Val	270(4.03×10^4); 425(1.76×10^4)	430	0.061	5.09	0.55
3,5-Cl-(S,S)Cy+D-Val	272(3.89×10^4); 426(1.82×10^4)	439	0.035	2.89	
3,5-Cl-(S,S)Cy+L-Arg	270(3.78×10^4); 428(1.44×10^4)	478	0.072	6.99	1.26
3,5-Cl-(S,S)Cy+D-Arg	270(4.25×10^4); 402(1.47×10^4)	447	0.12	8.77	
3,5-Cl-(S,S)Cy+L-Leu	266(4.16×10^4); 426(1.56×10^4)	430	0.099	8.46	0.72
3,5-Cl-(S,S)Cy+D-Leu	267(4.26×10^4); 426(1.63×10^4)	433	0.079	6.17	
3,5-Cl-(S,S)Cy+L-Trp	279(>1.00×10^6); 287(>1.00×10^6)	434	0.015	1.49	1.39
3,5-Cl-(S,S)Cy+D-Trp	275(>1.00×10^6); 285(>1.00×10^6)	431	0.031	2.02	
3,5-Cl-(S,S)Cy+L-Ser	274(4.06×10^4); 425(1.79×10^4)	482	0.041	2.92	1.18
3,5-Cl-(S,S)Cy+D-Ser	272(4.04×10^4); 427(1.82×10^4)	439	0.048	3.41	
3,5-Cl-(S,S)Cy+L-His	264(3.94×10^4); 426(1.18×10^4)	433	0.052	3.55	2.06
3,5-Cl-(S,S)Cy+D-His	267(4.38×10^4); 426(1.16×10^4)	432	0.072	7.11	

Table. S8 Photophysical data of **3-F-(R,R)diPh** and **3-F-(S,S)diPh** DMSO solution upon the addition of 100 equivalent of different D- or L-amino acids. Sample without emission data means that it is non-emissive. *ef* = (I_D-I_0)/(I_L-I_0).

3-F-diPh+amino acid	λ_{abs} /nm(ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Φ	I/I_0	*ef*
3-F-(R,R)diPh	260(5.22×10^3); 420(1.00×10^3)	430	0.0077		
3-F-(R,R)diPh+L-Ala	323(4.10×10^3); 412(5.10×10^3)	431	0.042	40.6	0.84
3-F-(R,R)diPh+D-Ala	323(3.70×10^3); 414(4.80×10^3)	430	0.040	34.1	
3-F-(R,R)diPh+L-Pro	333(4.90×10^3); 413(3.00×10^3)	473	0.11	143	1.43
3-F-(R,R)diPh+D-Pro	324(4.50×10^3); 405(3.70×10^3)	472	0.17	205	
3-F-(R,R)diPh+L-Glu	323(5.30×10^3); 414(2.90×10^3)	431	0.030	32.3	1.45
3-F-(R,R)diPh+D-Glu	327(8.50×10^3); 415(6.50×10^3)	431	0.016	46.8	
3-F-(R,R)diPh+L-Gln	323(2.70×10^3); 413(7.60×10^3)	428	0.056	99.2	1.32
3-F-(R,R)diPh+D-Gln	330(3.30×10^3); 413(6.50×10^3)	431	0.074	131	
3-F-(R,R)diPh+L-Val	330(3.80×10^3); 413(6.50×10^3)	429	0.073	119	0.40
3-F-(R,R)diPh+D-Val	333(3.30×10^3); 413(5.90×10^3)	430	0.060	47.6	
3-F-(R,R)diPh+L-Arg	263(4.31×10^4); 412(2.29×10^3)	435	0.0097	74.4	4.89
3-F-(R,R)diPh+D-Arg	268(4.59×10^4); 413(2.29×10^4)	436	0.054	364	
3-F-(R,R)diPh+L-Leu	267(1.89×10^4); 414(5.80×10^3)	429	0.097	118	0.81
3-F-(R,R)diPh+D-Leu	267(2.07×10^4); 414(5.00×10^3)	433	0.080	95.6	
3-F-(R,R)diPh+L-Trp	294(>1.00×10^6); 414(1.02×10^4)	421	0.063	127	0.73
3-F-(R,R)diPh+D-Trp	273(>1.00×10^6); 414(6.70×10^3)	430	0.060	92.7	
3-F-(R,R)diPh+L-Ser	327(3.60×10^3); 415(5.50×10^3)	432	0.097	136	0.90
3-F-(R,R)diPh+D-Ser	328(3.00×10^3); 413(6.80×10^3)	432	0.075	122	
3-F-(R,R)diPh+L-His	268(1.56×10^4); 416(5.80×10^3)	431	0.098	79.4	0.61
3-F-(R,R)diPh+D-His	267(1.74×10^4); 413(1.01×10^4)	430	0.062	48.4	
3-F-(S,S)diPh	260(5.32×10^3); 420(1.10×10^3)	430	0.0082		

3-F-(S,S)diPh+L-Ala	324(3.80×10^3); 412(4.90×10^3)	434	0.040	67.3	1.53
3-F-(S,S)diPh+D-Ala	323(4.10×10^3); 412(5.80×10^3)	430	0.062	103	
3-F-(S,S)diPh+L-Pro	332(2.90×10^3); 411(1.10×10^3)	473	0.040	79.1	2.11
3-F-(S,S)diPh+D-Pro	325(3.60×10^3); 406(2.40×10^3)	471	0.082	167	
3-F-(S,S)diPh+L-Glu	356(5.50×10^3); 412(1.20×10^3)	432	0.021	33.5	0.91
3-F-(S,S)diPh+D-Glu	332(4.90×10^3); 416(2.50×10^3)	436	0.020	30.5	
3-F-(S,S)diPh+L-Gln	276(8.20×10^3); 416(5.80×10^3)	431	0.096	125	0.86
3-F-(S,S)diPh+D-Gln	275(4.30×10^3); 413(3.50×10^3)	427	0.041	108	
3-F-(S,S)diPh+L-Val	330(3.50×10^3); 414(4.50×10^3)	427	0.36	703	0.11
3-F-(S,S)diPh+D-Val	332(3.70×10^3); 413(6.80×10^3)	433	0.030	77.3	
3-F-(S,S)diPh+L-Arg	276(1.20×10^4); 365(2.99×10^4)	432	0.012	249	0.33
3-F-(S,S)diPh+D-Arg	276(3.41×10^4); 365(2.05×10^4)	440	0.035	82.2	
3-F-(S,S)diPh+L-Leu	267(2.19×10^4); 415(5.60×10^3)	426	0.059	93.7	5.25
3-F-(S,S)diPh+D-Leu	267(2.27×10^4); 414(5.20×10^3)	423	0.21	492	
3-F-(S,S)diPh+L-Trp	285(>1.00×10^6); 355(9.00×10^3)	424	0.060	151	0.73
3-F-(S,S)diPh+D-Trp	294 (>1.00×10^6); 359(1.09×10^4)	430	0.042	110	
3-F-(S,S)diPh+L-Ser	326(3.10×10^3); 415(6.50×10^3)	429	0.074	133	0.97
3-F-(S,S)diPh+D-Ser	327(3.30×10^3); 413(5.80×10^3)	430	0.069	129	
3-F-(S,S)diPh+L-His	294(7.59×10^4); 365(1.01×10^4)	433	0.034	117	1.23
3-F-(S,S)diPh+D-His	293(7.71×10^4); 365(1.21×10^4)	432	0.062	144	

Table. S9 Photophysical data of **3-Cl-(R,R)diPh** and **3-Cl-(S,S)diPh** DMSO solution upon the addition of 100 equivalent of different D- or L-amino acids. Sample without emission data means that it is non-emissive. *ef* = (I_D-I_0)/(I_L-I_0).

3-Cl-diPh+amino acid	λ_{abs} /nm(ε/dm³ mol⁻¹cm⁻¹)	λ_{em} /nm	Φ	I/I_0	*ef*
3-Cl-(R,R)diPh	318(5.50×10³); 415(1.70×10³)	456	0.021		
3-Cl-(R,R)diPh+L-Ala	281(1.09×10⁴); 418(1.04×10⁴)	435	0.047	14.8	1.53
3-Cl-(R,R)diPh+D-Ala	282 (1.00×10⁴); 418(6.70×10³)	432	0.072	22.6	
3-Cl-(R,R)diPh+L-Pro	330(4.00×10³); 417(3.10×10³)	480	0.23	35.9	1.30
3-Cl-(R,R)diPh+D-Pro	326(4.00×10³); 416(3.50×10³)	483	0.31	46.6	
3-Cl-(R,R)diPh+L-Glu	327(5.10×10³); 421(2.00×10³)	434	0.16	10.8	0.91
3-Cl-(R,R)diPh+D-Glu	330(6.30×10³); 419(2.20×10³)	433	0.11	9.83	
3-Cl-(R,R)diPh+L-Gln	326(2.50×10³); 417(1.04×10⁴)	431	0.050	20.7	0.68
3-Cl-(R,R)diPh+D-Gln	326(2.80×10³); 417(5.80×10³)	432	0.063	14.1	
3-Cl-(R,R)diPh+L-Val	293(3.90×10³); 419(1.12×10⁴)	426	0.030	10.5	2.78
3-Cl-(R,R)diPh+D-Val	290(2.30×10³); 414(9.50×10³)	429	0.068	29.2	
3-Cl-(R,R)diPh+L-Arg	326(3.50×10³); 417(4.90×10³)	431	0.062	12.8	4.43
3-Cl-(R,R)diPh+D-Arg	289(3.30×10³); 416(1.35×10⁴)	472	0.12	56.7	
3-Cl-(R,R)diPh+L-Leu	329(3.90×10³); 419(6.10×10³)	432	0.040	9.04	1.15
3-Cl-(R,R)diPh+D-Leu	326(4.60×10³); 417(3.60×10³)	430	0.066	10.4	
3-Cl-(R,R)diPh+L-Trp	262(>1.00×10⁶); 417(1.35×10⁴)	426	0.076	53.4	0.18
3-Cl-(R,R)diPh+D-Trp	273(>1.00×10⁶); 418(6.40×10³)	431	0.035	9.61	
3-Cl-(R,R)diPh+L-Ser	326(3.20×10³); 417(3.70×10³)	438	0.040	6.23	0.92
3-Cl-(R,R)diPh+D-Ser	327(3.40×10³); 417(3.60×10³)	432	0.037	5.73	
3-Cl-(R,R)diPh+L-His	327(2.40×10³); 417(7.60×10³)	423	0.098	38.9	0.56
3-Cl-(R,R)diPh+D-His	327(2.10×10³); 420(8.50×10³)	432	0.062	21.8	
3-Cl-(S,S)diPh	318(5.60×10³); 415(1.80×10³)	456	0.023		

3-Cl-(S,S)diPh+L-Ala	281(1.10×10^4); 418(1.04×10^4)	461	0.031	11.2	1.04
3-Cl-(S,S)diPh+D-Ala	282 (1.00×10^4); 420(8.60×10^3)	474	0.041	11.6	
3-Cl-(S,S)diPh+L-Pro	326(5.40×10^3); 418(4.10×10^3)	474	0.14	35.3	1.52
3-Cl-(S,S)diPh+D-Pro	327(5.60×10^3); 414(6.00×10^3)	476	0.15	53.6	
3-Cl-(S,S)diPh+L-Glu	326(5.40×10^3); 421(2.10×10^3)	429	0.11	10.9	0.60
3-Cl-(S,S)diPh+D-Glu	330(5.90×10^3); 420(2.00×10^3)	431	0.048	6.54	
3-Cl-(S,S)diPh+L-Gln	326(5.60×10^3); 417(1.10×10^4)	439	0.18	57.9	0.21
3-Cl-(S,S)diPh+D-Gln	327(5.20×10^3); 422(3.60×10^3)	432	0.050	12.2	
3-Cl-(S,S)diPh+L-Val	282(1.38×10^4); 419(1.39×10^4)	431	0.032	9.66	3.48
3-Cl-(S,S)diPh+D-Val	284(1.29×10^3); 416(1.22×10^4)	428	0.12	33.6	
3-Cl-(S,S)diPh+L-Arg	326(4.10×10^3); 420(6.90×10^3)	434	0.030	8.67	5.48
3-Cl-(S,S)diPh+D-Arg	266(1.36×10^4); 416(1.37×10^4)	474	0.090	47.5	
3-Cl-(S,S)diPh+L-Leu	287(1.27×10^4); 421(1.19×10^4)	426	0.057	29.7	0.28
3-Cl-(S,S)diPh+D-Leu	275(1.02×10^4); 421(8.10×10^3)	431	0.030	8.32	
3-Cl-(S,S)diPh+L-Trp	266($>1.00\times10^6$); 421(1.28×10^4)	424	0.071	47.9	0.37
3-Cl-(S,S)diPh+D-Trp	268($>1.00\times10^6$); 414(5.80×10^3)	432	0.052	17.7	
3-Cl-(S,S)diPh+L-Ser	327(3.80×10^3); 417(5.90×10^3)	431	0.037	9.02	2.87
3-Cl-(S,S)diPh+D-Ser	327(3.60×10^3); 419(6.70×10^3)	434	0.091	25.9	
3-Cl-(S,S)diPh+L-His	327(3.00×10^3); 420(7.90×10^3)	432	0.068	26.7	0.60
3-Cl-(S,S)diPh+D-His	327(4.50×10^3); 420(1.12×10^4)	433	0.031	16.1	

Table. S10 Photophysical data of **3,5-Cl-(R,R)diPh** and **3,5-Cl-(S,S)diPh** DMSO solution upon the addition of 100 equivalent of different D- or L-amino acids. Sample without emission data means that it is non-emissive. *ef* = $(I_D-I_0)/(I_L-I_0)$.

3,5-Cl-diPh+amino acid	λ_{abs} /nm(ε/dm^3 mol^{-1}cm^{-1})	λ_{em} /nm	Φ	I/I_0	*ef*
3,5-Cl-(R,R)diPh	266(2.46×10^4); 334 (9.70×10^3); 420(4.90×10^3)	475	0.061		
3,5-Cl-(R,R)diPh+L-Ala	275(1.04×10^4); 287(8.20×10^3); 430(1.37×10^4)	485	0.16	11.3	0.74
3,5-Cl-(R,R)diPh+D-Ala	276(7.60×10^3); 286(7.30×10^3); 427(1.22×10^4)	489	0.097	8.36	
3,5-Cl-(R,R)diPh+L-Pro	269(7.80×10^3); 429(9.10×10^3)	495	0.19	13.5	0.78
3,5-Cl-(R,R)diPh+D-Pro	269(8.20×10^3); 430(8.90×10^3)	491	0.19	10.5	
3,5-Cl-(R,R)diPh+L-Glu	267(7.90×10^3); 285(6.90×10^3); 431(7.80×10^3)	442	0.095	5.3	0.78
3,5-Cl-(R,R)diPh+D-Glu	267(9.20×10^3); 285(7.60×10^3); 431(7.80×10^3)	450	0.084	4.13	
3,5-Cl-(R,R)diPh+L-Gln	267(8.90×10^3); 285(8.40×10^3; 431(1.21×10^4)	486	0.11	6.89	2.93
3,5-Cl-(R,R)diPh+D-Gln	264(1.07×10^4); 282(8.20×10^3); 426(1.16×10^4)	436	0.22	20.2	
3,5-Cl-(R,R)diPh+L-Val	267(1.08×10^4); 285(1.08×10^4); 428(1.54×10^4)	430	0.090	6.65	0.70
3,5-Cl-(R,R)diPh+D-Val	268(7.70×10^3); 289(8.50×10^3); 425(1.21×10^4)	430	0.10	4.66	
3,5-Cl-(R,R)diPh+L-Arg	267(1.10×10^4); 425(1.30×10^4)	444	0.26	20.3	0.91
3,5-Cl-(R,R)diPh+D-Arg	269(1.09×10^4); 417(1.44×10^4)	482	0.27	18.5	
3,5-Cl-(R,R)diPh+L-Leu	269(1.53×10^4); 429(1.91×10^4)	429	0.054	4.93	0.79
3,5-Cl-(R,R)diPh+D-Leu	268(1.42×10^4); 430(1.08×10^4)	482	0.092	3.89	
3,5-Cl-(R,R)diPh+L-Trp	283(>1.00×10^6);429(1.26×10^4)	435	0.091	6.46	0.84
3,5-Cl-(R,R)diPh+D-Trp	288(>1.00×10^6); 431(1.39×10^4)	434	0.067	5.43	
3,5-Cl-(R,R)diPh+L-Ser	274(1.01×10^4); 429(1.20×10^4)	447	0.099	6.34	0.98
3,5-Cl-(R,R)diPh+D-Ser	282(9.80×10^4); 430(1.31×10^4)	483	0.12	6.21	
3,5-Cl-(R,R)diPh+L-His	288(6.40×10^3); 430(6.70×10^3) 0.027	431	0.13	4.67	2.46
3,5-Cl-(R,R)diPh+D-His	288(6.50×10^4); 430(6.70×10^3) 0.027	432	0.25	11.5	
3,5-Cl-(S,S)diPh	264(2.31×10^4); 336 (6.90×10^3); 433(3.20×10^3)	475	0.068		

3,5-Cl-(S,S)diPh+L-Ala	274(1.03×10^{4}); 282(9.30×10^{3}); 427(1.44×10^{4})	437	0.18	18.2	0.45
3,5-Cl-(S,S)diPh+D-Ala	274(8.70×10^{3}); 284(8.00×10^{3}); 424(1.24×10^{4})	444	0.13	8.19	
3,5-Cl-(S,S)diPh+L-Pro	267(7.70×10^{3}); 425(9.10×10^{3})	473	0.15	10.7	0.99
3,5-Cl-(S,S)diPh+D-Pro	267(8.30×10^{3}); 427(8.70×10^{3})	471	0.16	10.6	
3,5-Cl-(S,S)diPh+L-Glu	267(9.10×10^{3}); 285(7.50×10^{3}); 430(8.50×10^{3})	438	0.092	4.33	1.29
3,5-Cl-(S,S)diPh+D-Glu	267(8.90×10^{3}); 285(6.80×10^{3}); 430(8.40×10^{3})	437	0.090	5.59	
3,5-Cl-(S,S)diPh+L-Gln	267(1.11×10^{4}); 284(1.01×10^{4}); 430(1.39×10^{4})	438	0.081	5.82	6.22
3,5-Cl-(S,S)diPh+D-Gln	267(1.18×10^{4}); 285(9.20×10^{3}); 427(1.12×10^{4})	435	0.24	36.2	
3,5-Cl-(S,S)diPh+L-Val	267(1.35×10^{4}); 285(1.32×10^{4}); 426(1.69×10^{4})	432	0.075	7.84	0.56
3,5-Cl-(S,S)diPh+D-Val	267(1.32×10^{4}); 285(1.16×10^{4}); 425(1.45×10^{4})	433	0.050	4.39	
3,5-Cl-(S,S)diPh+L-Arg	267(2.35×10^{4}); 429(1.86×10^{4})	482	0.19	15.8	2.827
3,5-Cl-(S,S)diPh+D-Arg	272(1.25×10^{4}); 424(1.13×10^{4})	447	0.15	35.8	
3,5-Cl-(S,S)diPh+L-Leu	268(1.05×10^{4}); 426(1.35×10^{4})	430	0.10	4.2	0.53
3,5-Cl-(S,S)diPh+D-Leu	267(1.17×10^{4}); 427(1.26×10^{4})	430	0.071	2.23	
3,5-Cl-(S,S)diPh+L-Trp	275($>1.00\times10^{6}$);431(1.35×10^{4})	436	0.051	2.95	2.13
3,5-Cl-(S,S)diPh+D-Trp	286($>1.00\times10^{6}$); 433(1.33×10^{4})	434	0.067	6.31	
3,5-Cl-(S,S)diPh+L-Ser	272(8.40×10^{3}); 430(1.23×10^{4})	438	0.080	5.42	0.91
3,5-Cl-(S,S)diPh+D-Ser	285(9.40×10^{4}); 431(1.23×10^{4})	440	0.084	4.93	
3,5-Cl-(S,S)diPh+L-His	287(8.40×10^{3}); 430(9.20×10^{3}) 0.047	439	0.070	4.46	2.71
3,5-Cl-(S,S)diPh+D-His	287(9.80×10^{4}); 429(1.01×10^{4}) 0.060	431	0.12	12.3	

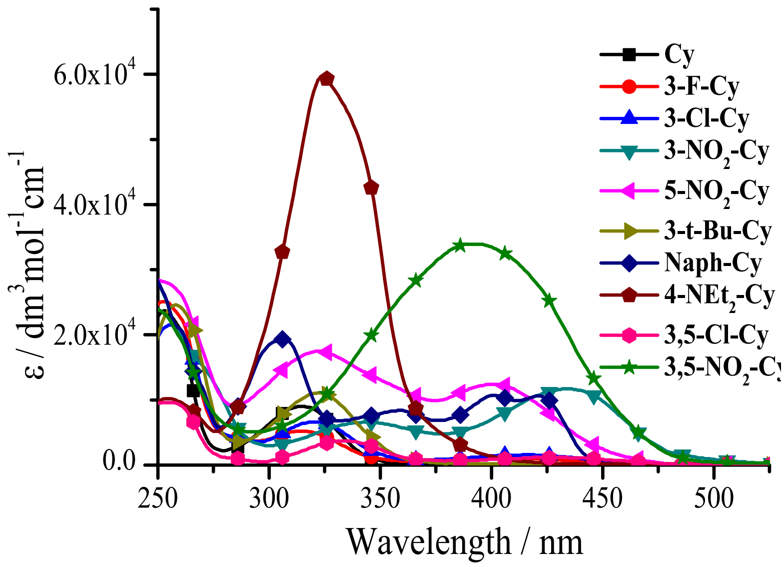

Fig. S1 Absorption spectra of some selected **Cys** in MeCN.

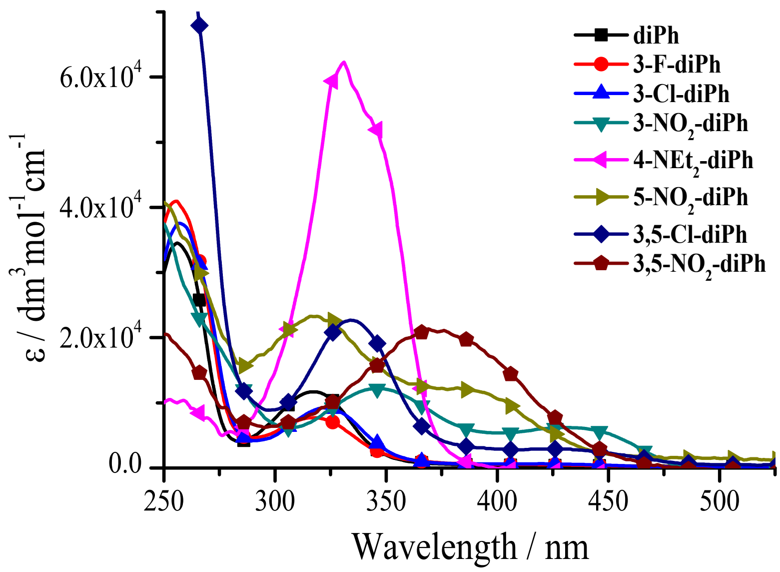

Fig. S2 Absorption spectra of some selected **diPhs** in MeCN.

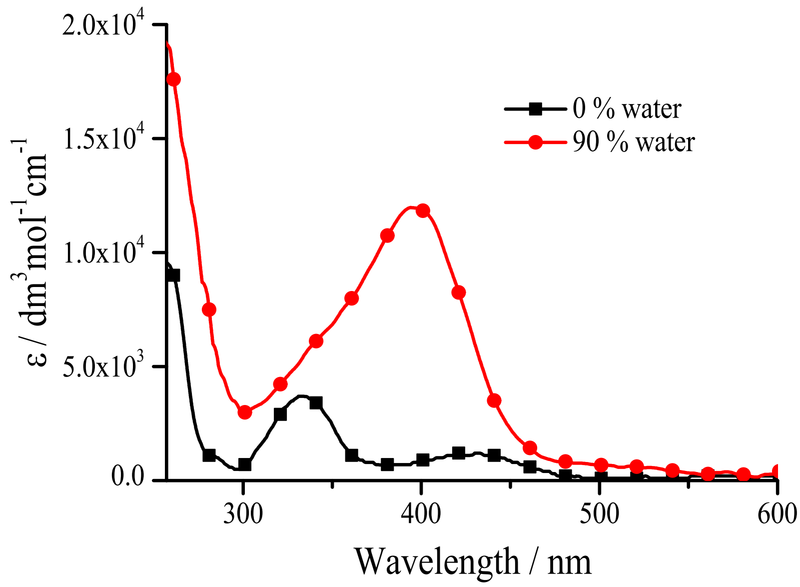

Fig. S3 Absorption spectra of **3,5-Cl-Cy** in MeCN-H_2O with different *f* values (1.0×10^{-5} mol dm^{-3}).

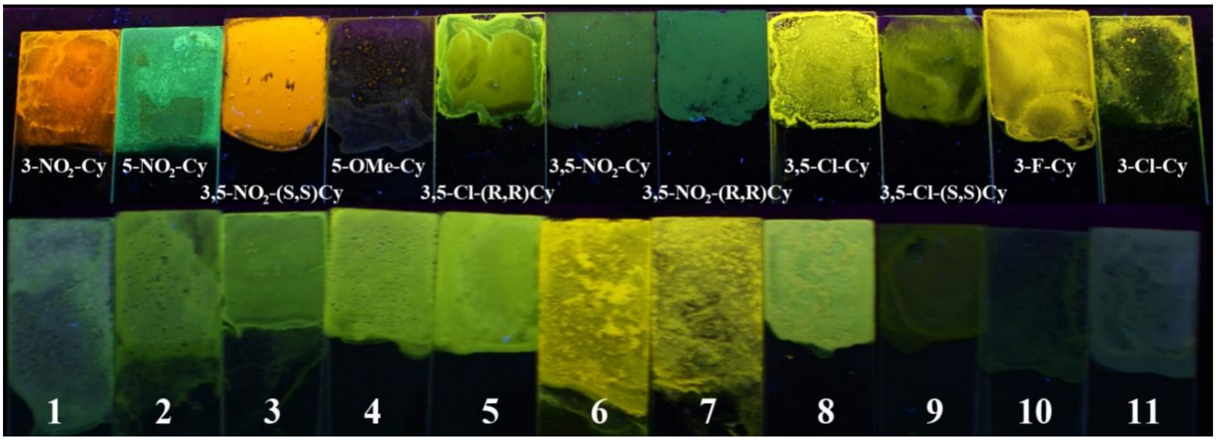

Fig. S4 Photographs (under 360 nm UV light) of casting films of **Cys** (top) and **diPhs** (bottom: 1 = **diPh**; 2 = **3-F-(R,R)diPh**; 3 = **3-Cl-(S,S)diPh**; 5 = **3-Cl-(R,R)diPh**; 6 = **3,5-Cl-(R,R)diPh**; 7 = **3-Cl-(S,S)diPh**; 8 = **5-NO$_2$-diPh**; 9 = **3-NO$_2$-diPh**; 10 = **Naph-diPh**; 11 = **4-NEt$_2$-diPh**;).

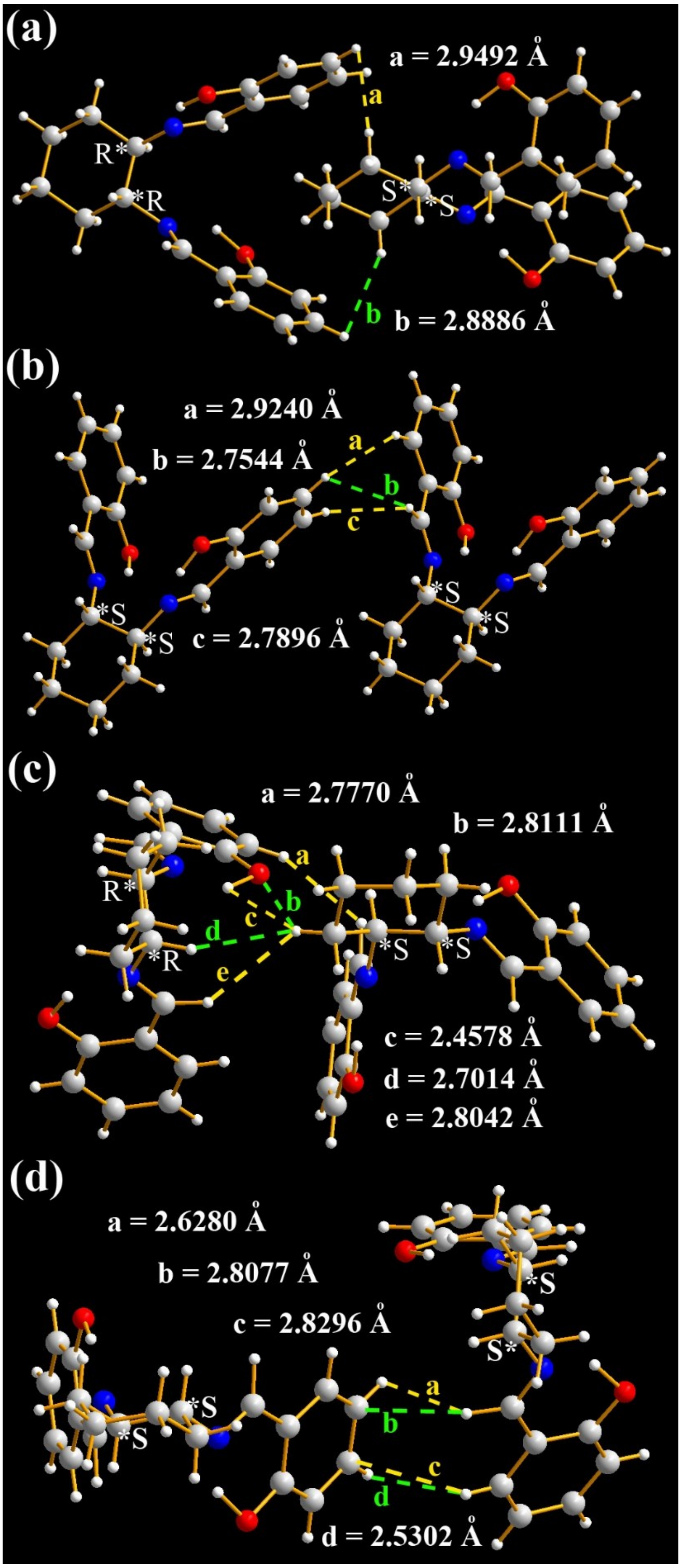

Fig. S5 X-ray single crystal structures and packing of **Cy** molecules: (a) and (b), intermolecular interactions of the two closest molecules; (c) enantiomers.

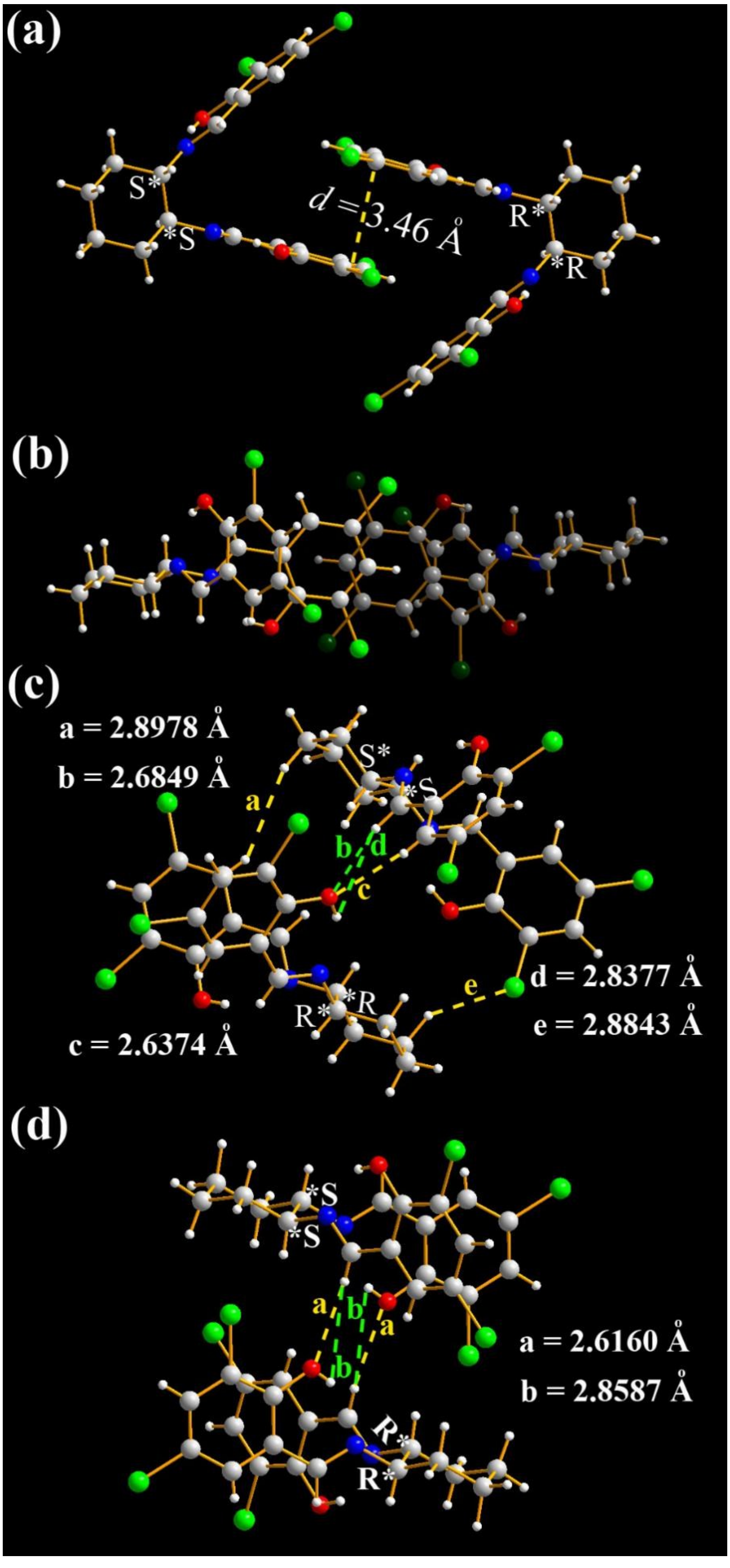

Fig. S6 X-ray single crystal structures and packing of **3,5-Cl-Cy** molecules (a: side view of face-to-face π–π interactions; b: top view of face-to-face π–π interactions).

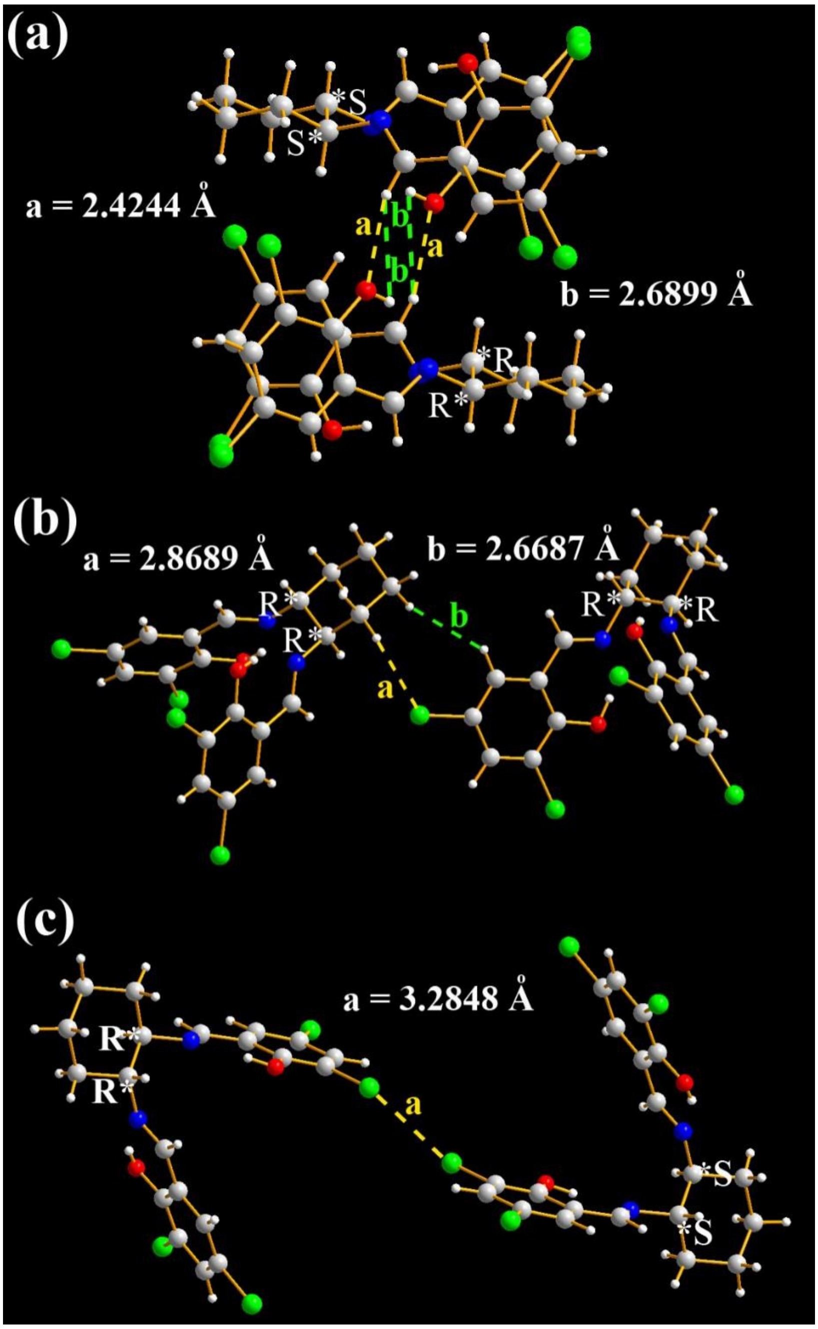

Fig. S7 X-ray single crystal structures and packing of **3,5-Cl-Cy** molecules.

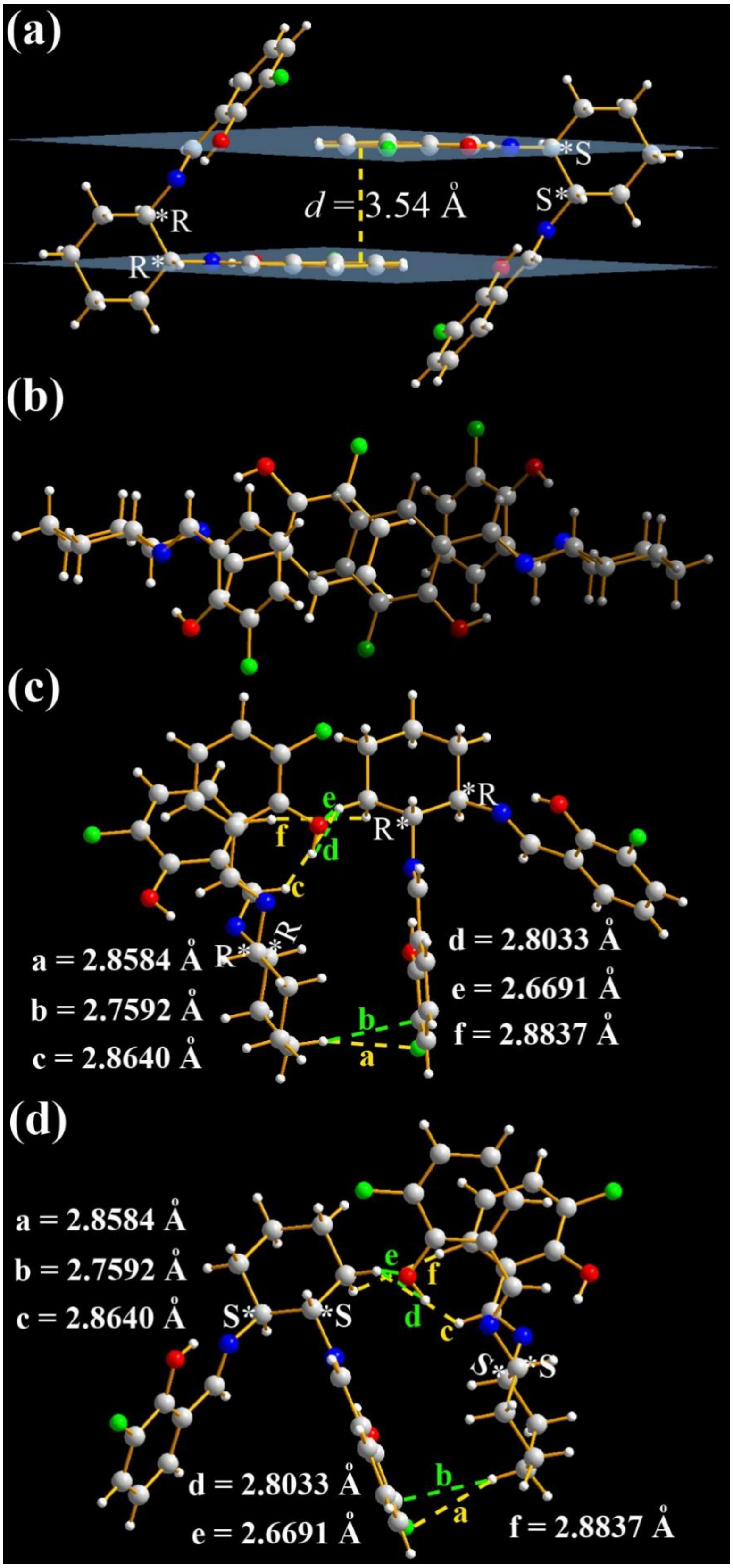

Fig. S8 X-ray single crystal structures and packing of **3-F-Cy** molecules (a: side view of face-to-face π–π interactions; b: top view of face-to-face π–π interactions).

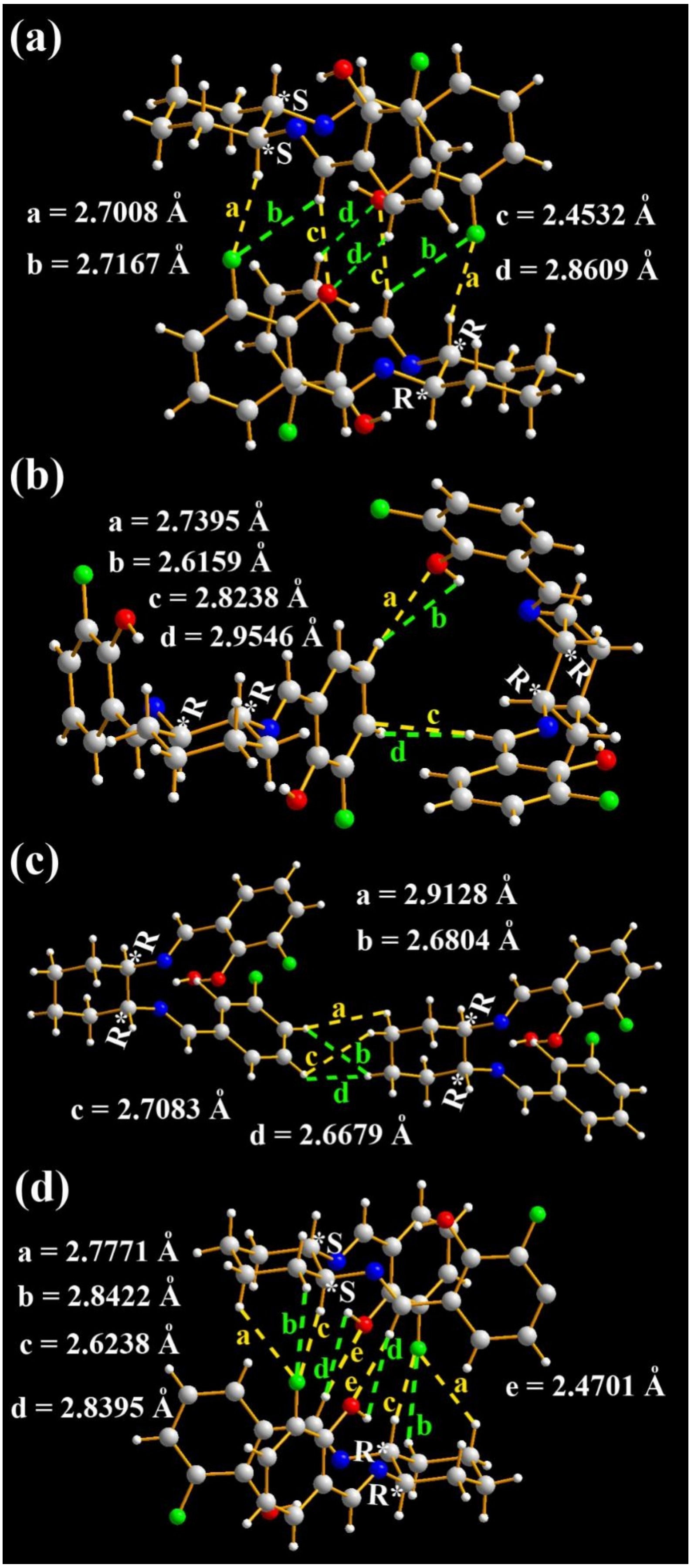

Fig. S9 X-ray single crystal structures and packing of **3-F-Cy** molecules.

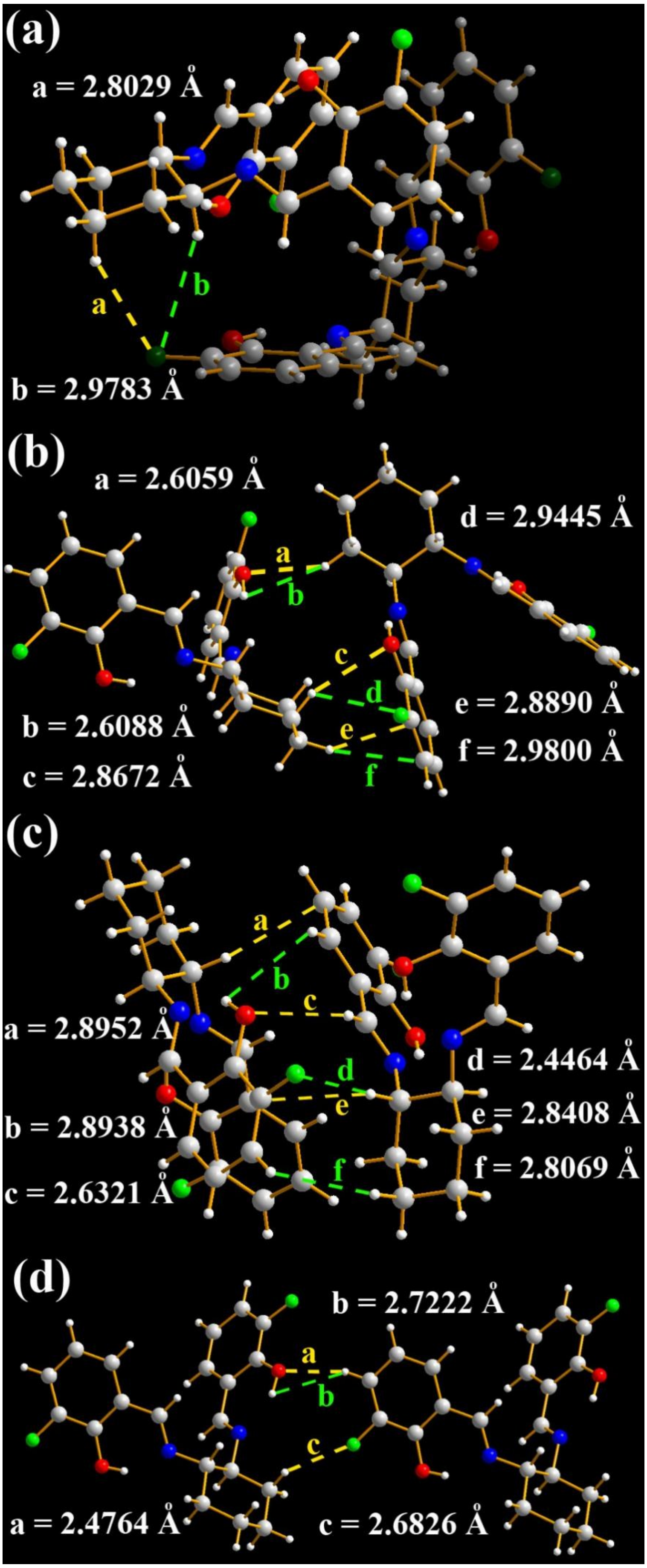

Fig. S10 X-ray single crystal structures and packing of **3-F-(S,S)Cy** molecules.

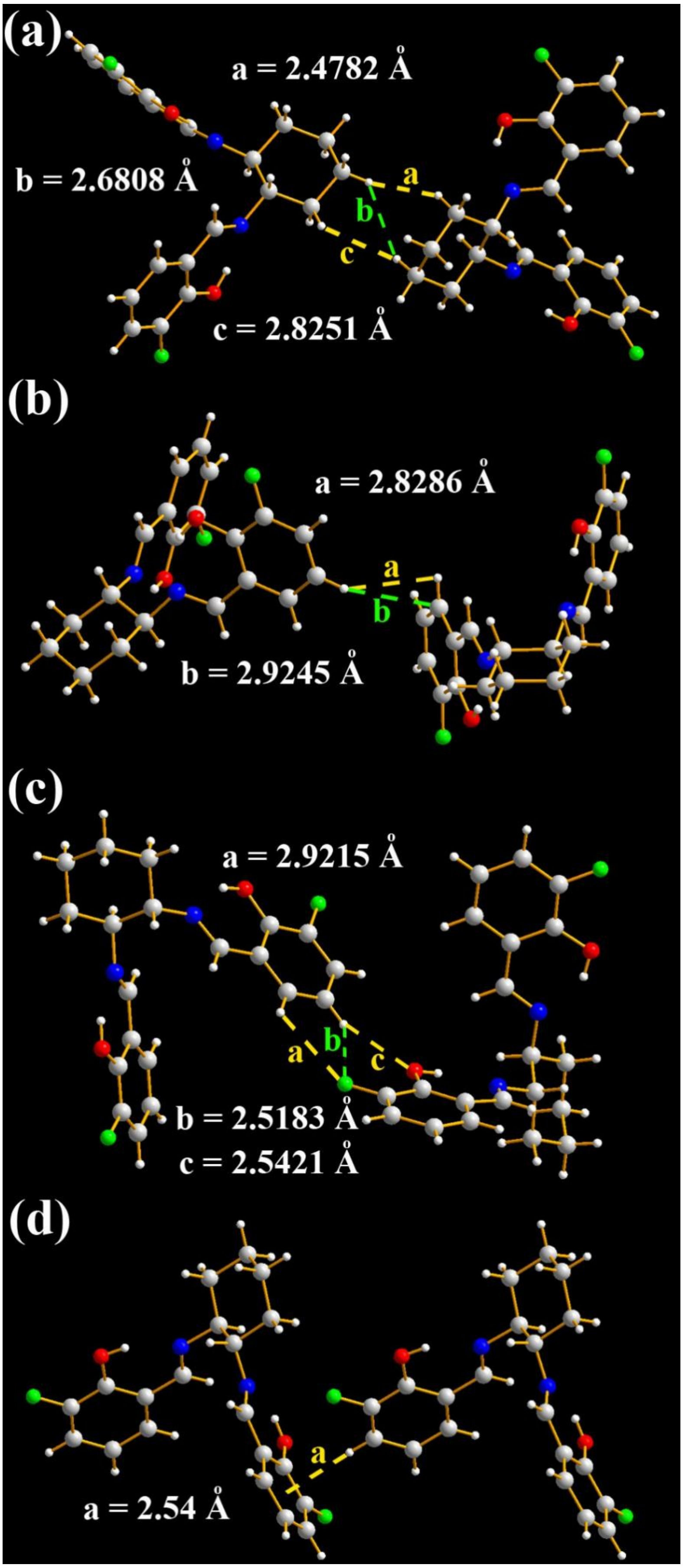

Fig. S11 X-ray single crystal structures and packing of **3-F-(S,S)Cy** molecules.

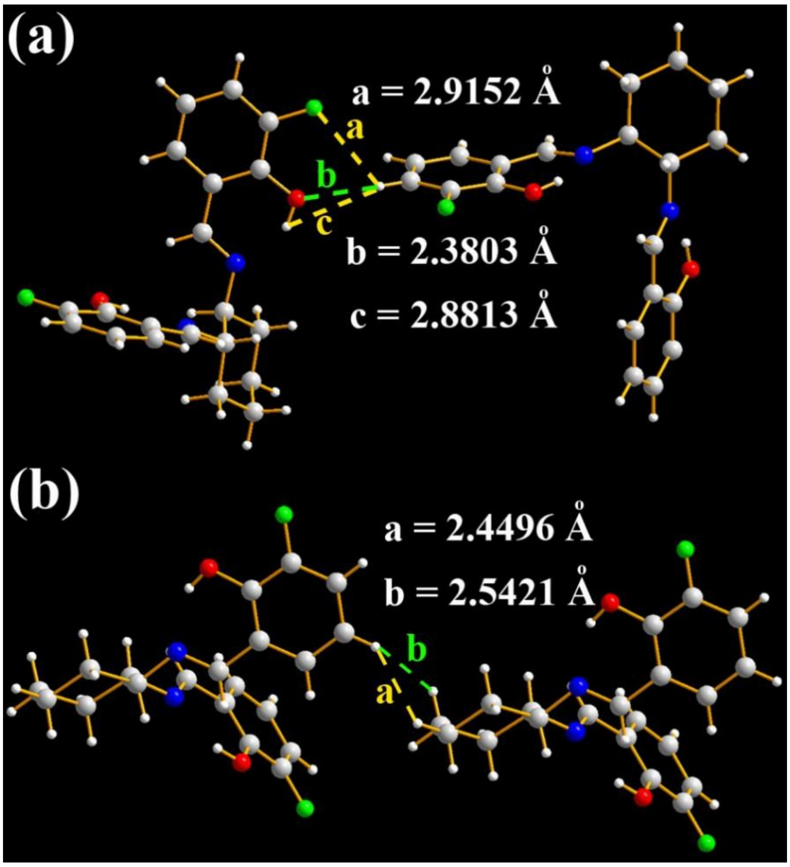

Fig. S12 X-ray single crystal structures and packing of **3-F-(S,S)Cy** molecules.

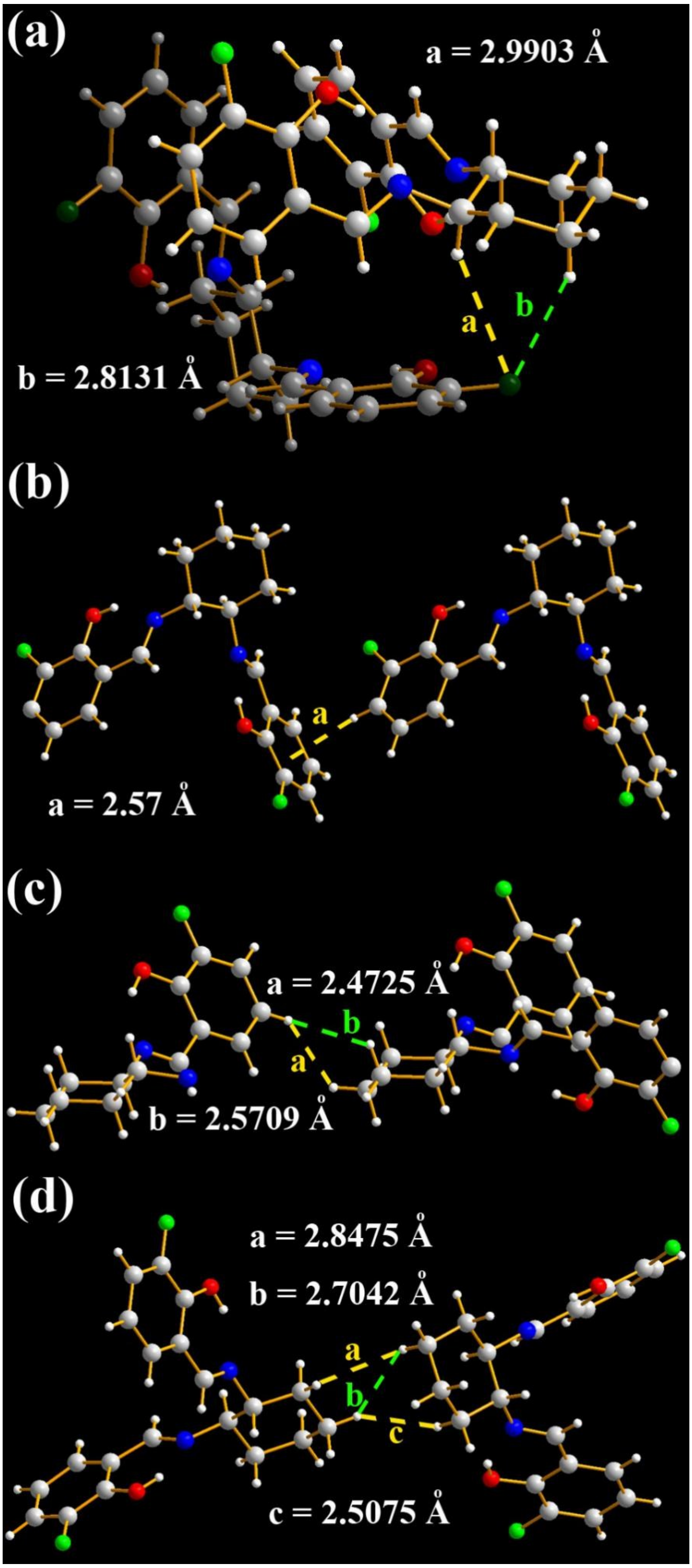

Fig. S13 X-ray single crystal structures and packing of **3-F-(R,R)Cy** molecules.

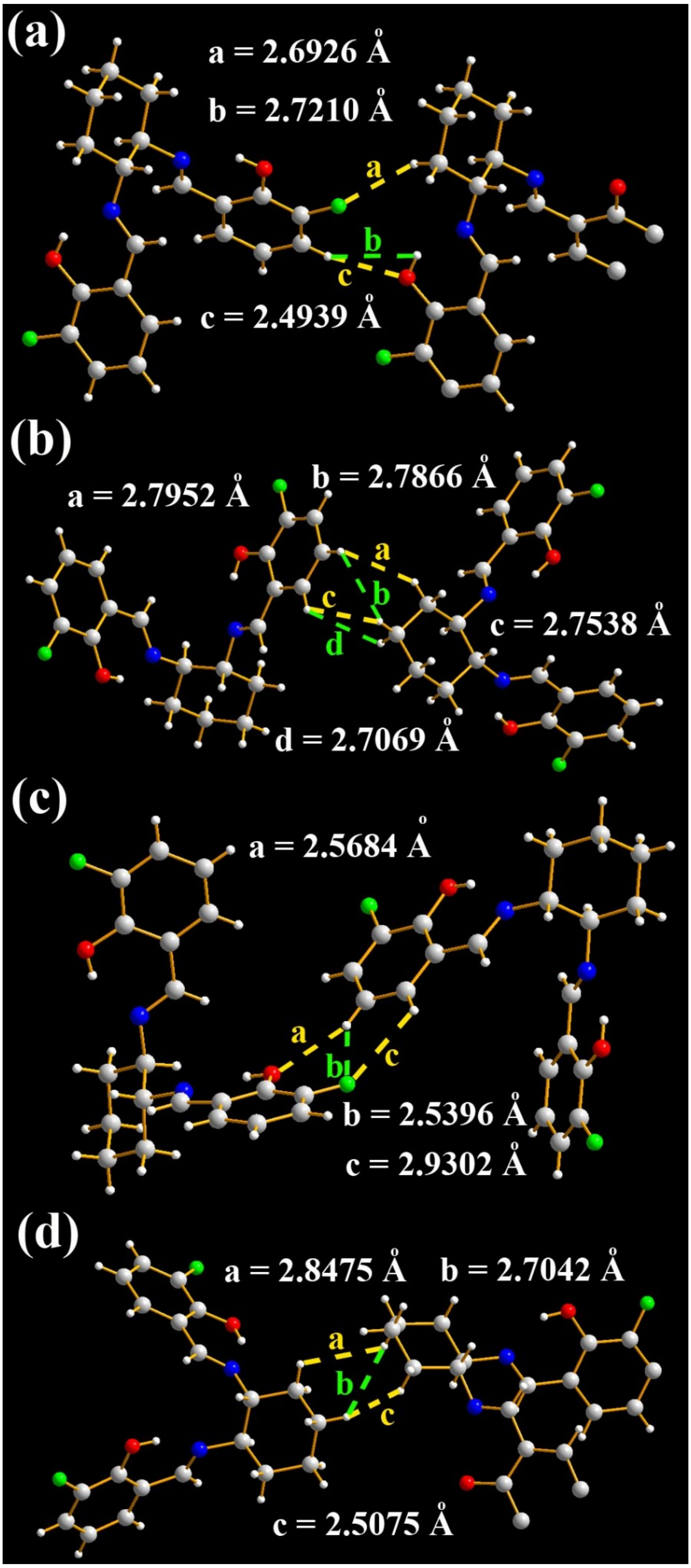

Fig. S14 X-ray single crystal structures and packing of **3-F-(R,R)Cy** molecules.

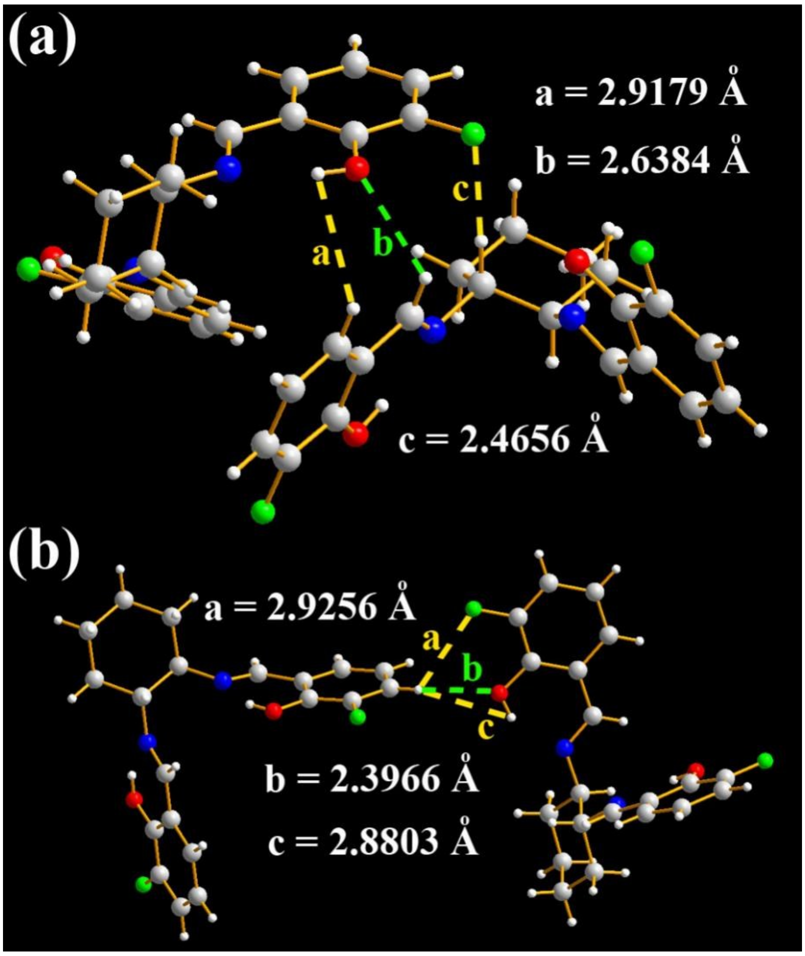

Fig. S15 X-ray single crystal structures and packing of **3-F-(R,R)Cy** molecules.

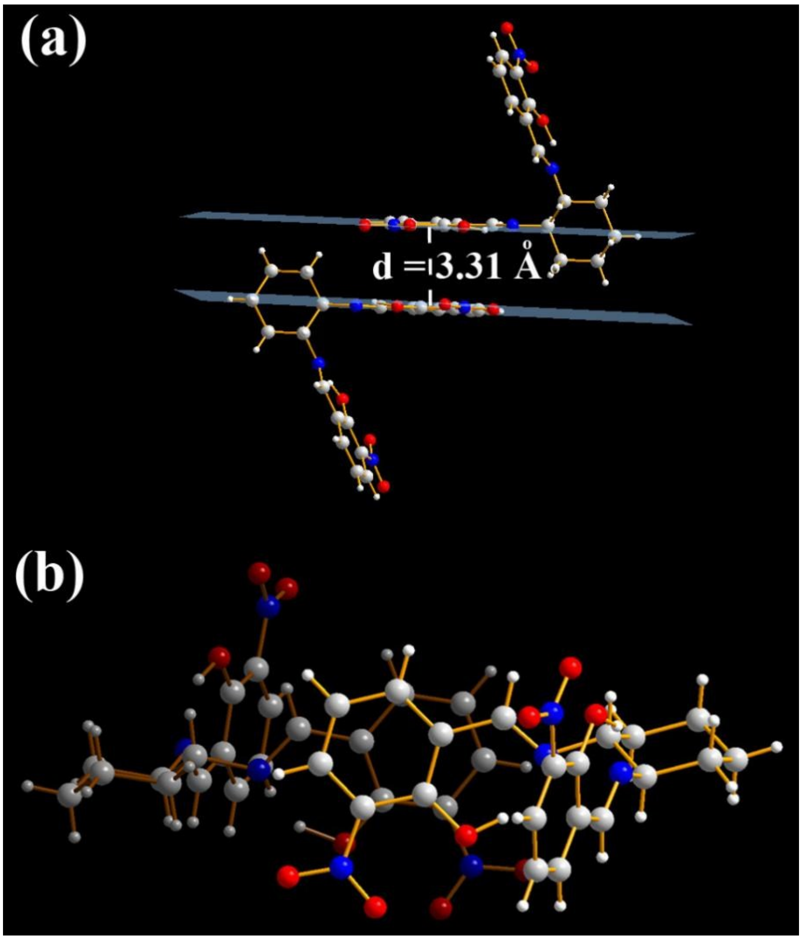

Fig. S16 X-ray single crystal structures and packing of **3-NO_2-(R,R)Cy** molecules (a: side view of face-to-face π–π interactions; b: top view of face-to-face π–π interactions; solvent molecules are omitted).

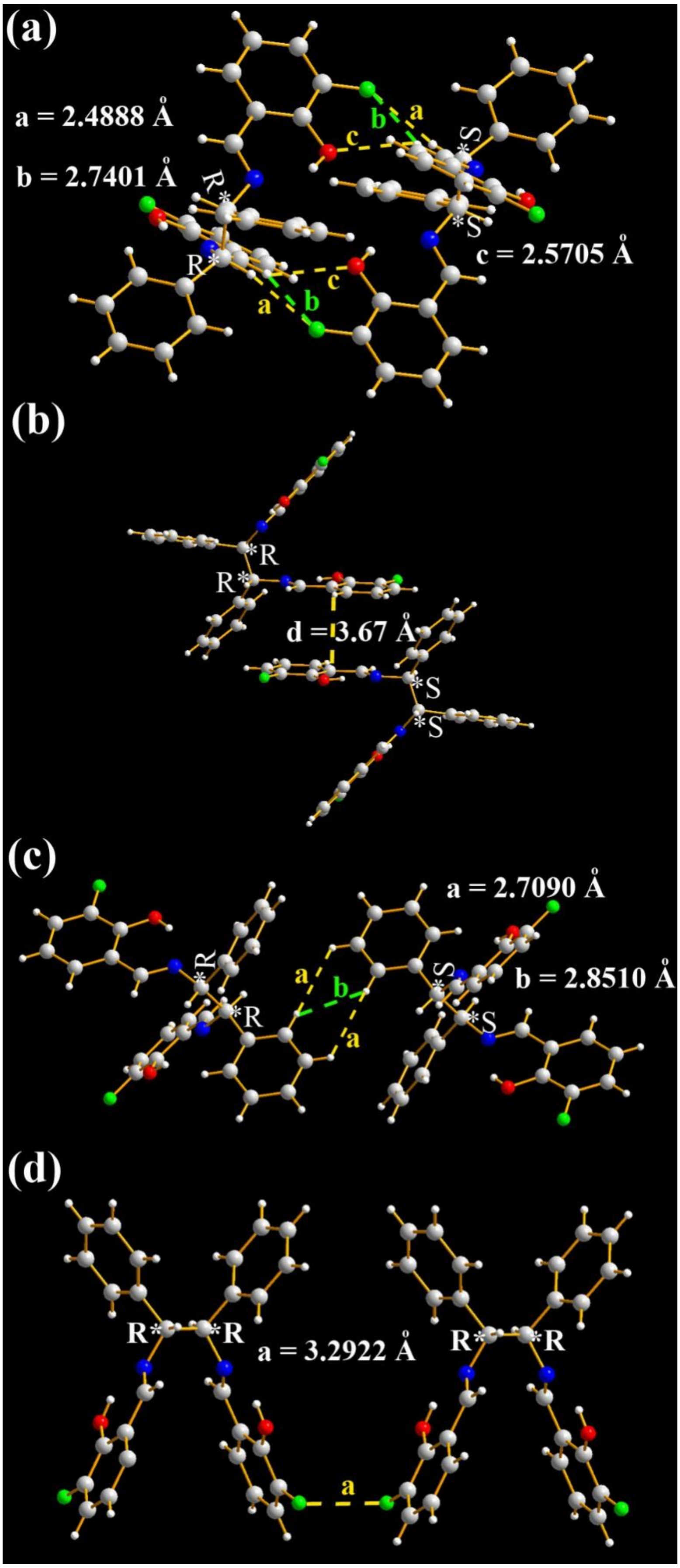

Fig. S17 X-ray single crystal structures and packing of **3-F-diPh** molecules.

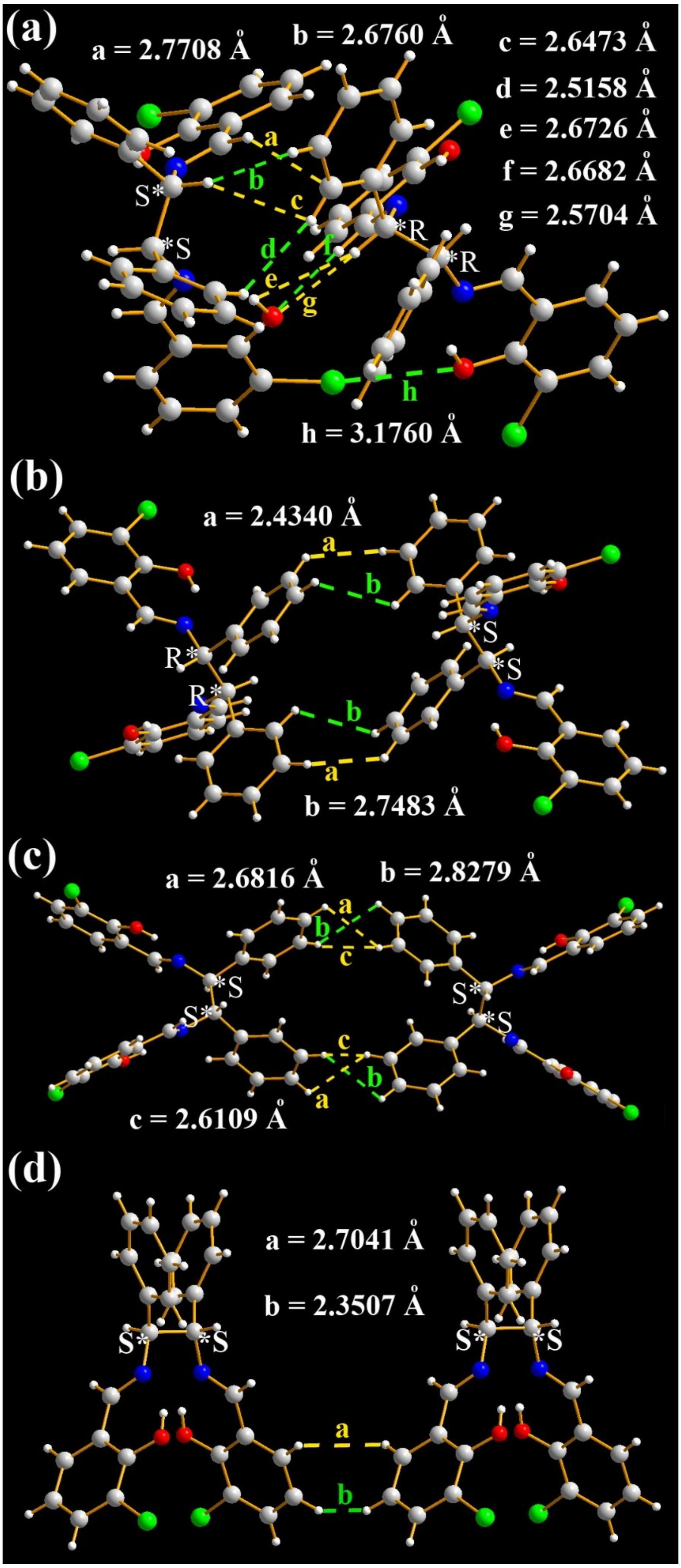

Fig. S18 X-ray single crystal structures and packing of **3-Cl-diPh** molecules.

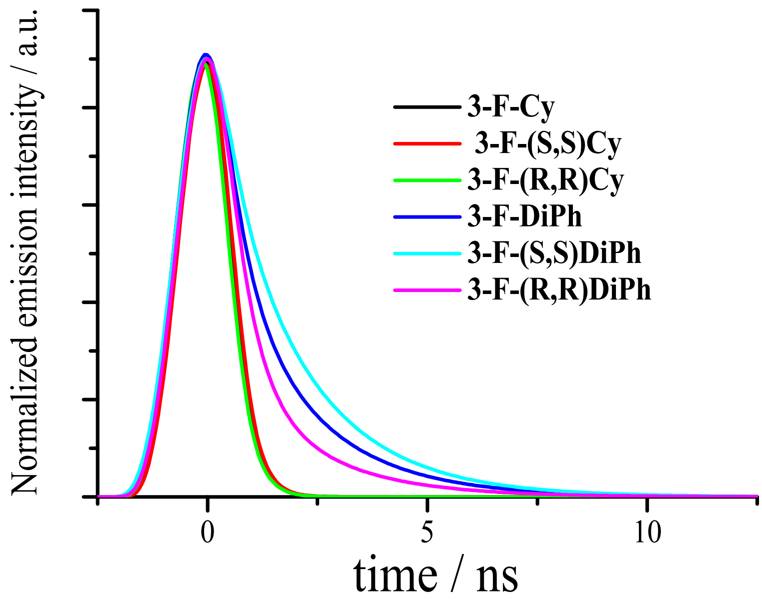

Fig. S19 Time-resolved emission decay spectra (excited at 370 nm) of powder samples.

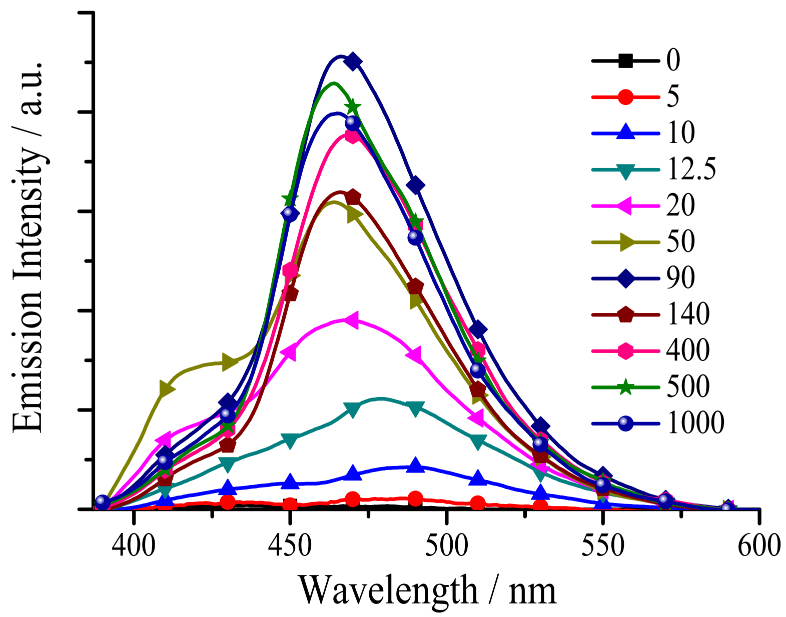

Fig. S20 Emission spectra of **3,5-Cl-diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of different equivalents of OH^{-}.

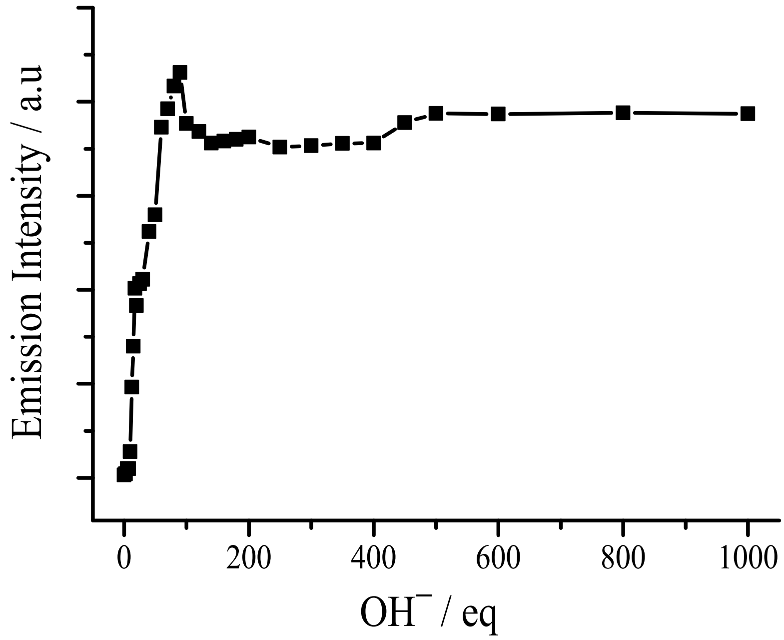

Fig. S21 Plot of emission intensity of **3,5-Cl-diPh** (1.0×10^{-5} mol dm^{-3} in DMSO) at 470 nm (excited at 380 nm) as a function of OH^{-} concentration.

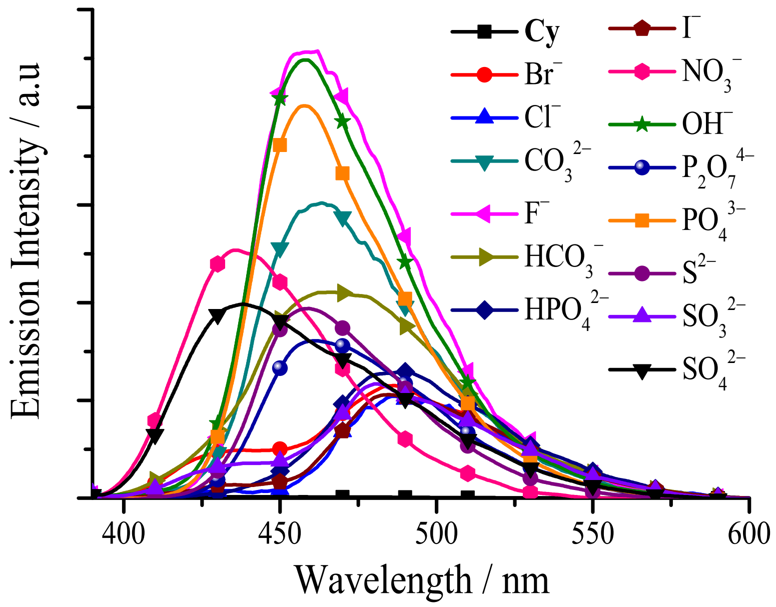

Fig. S22 Emission spectra of **Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

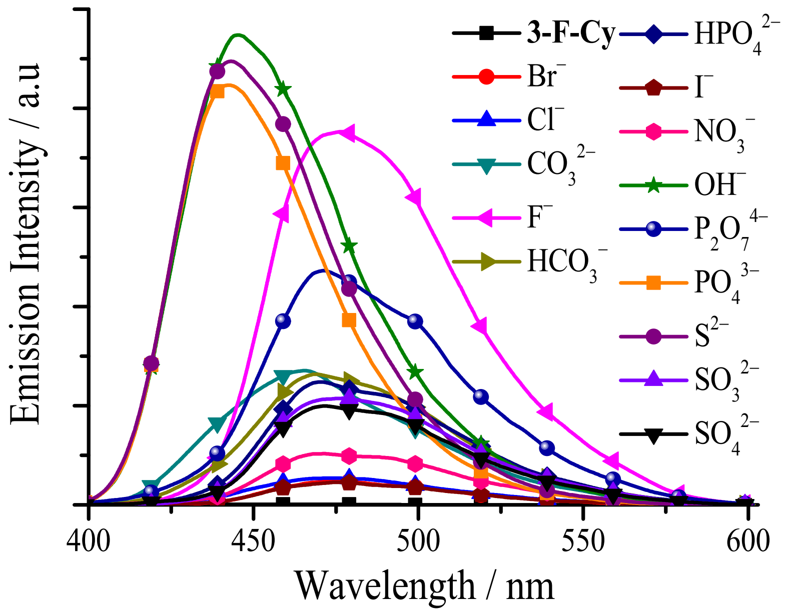

Fig. S23 Emission spectra of **3-F-Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

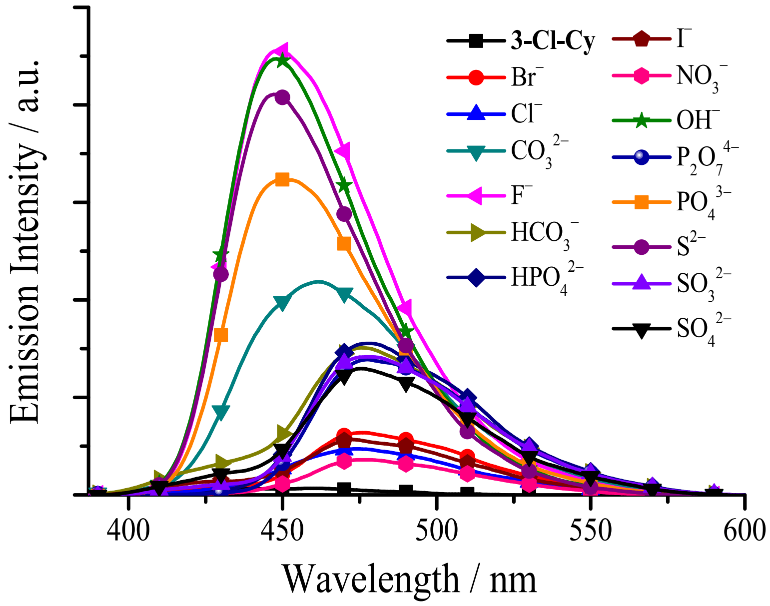

Fig. S24 Emission spectra of **3-Cl-Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

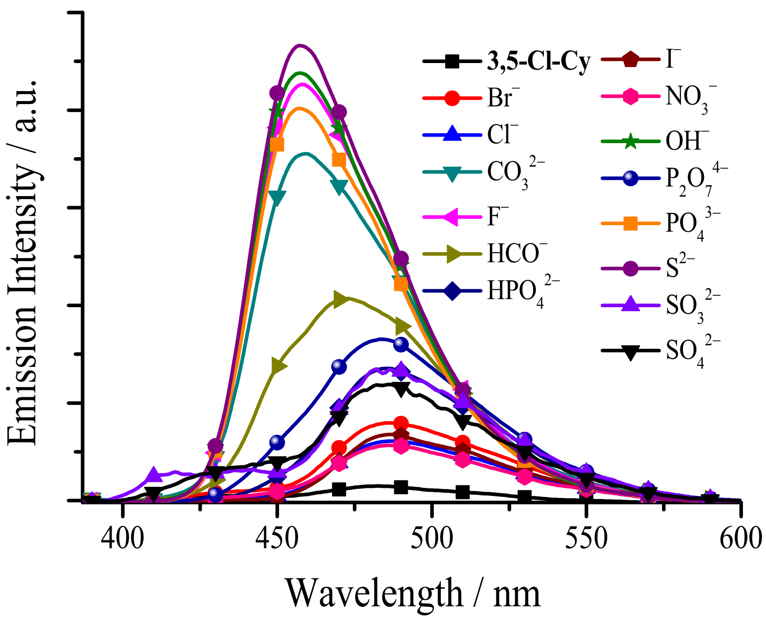

Fig. S25 Emission spectra of **3,5-Cl-Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

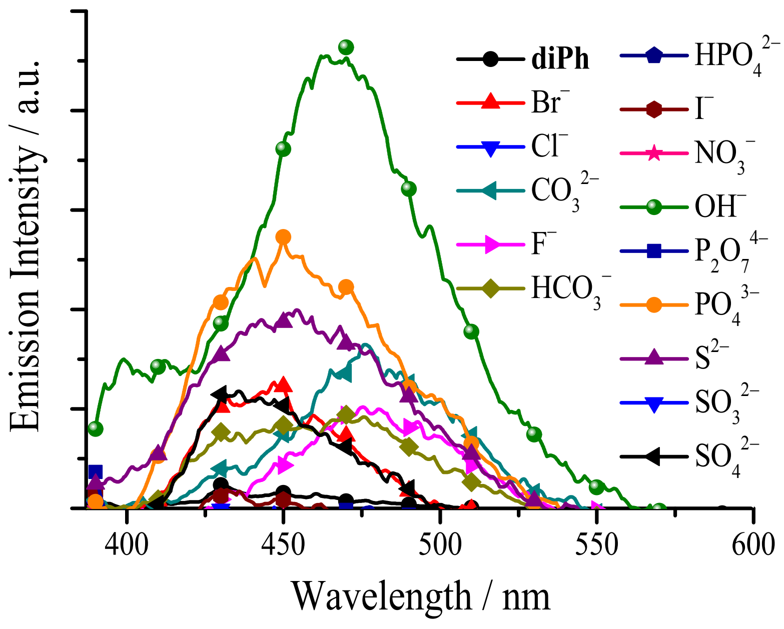

Fig. S26 Emission spectra of **diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

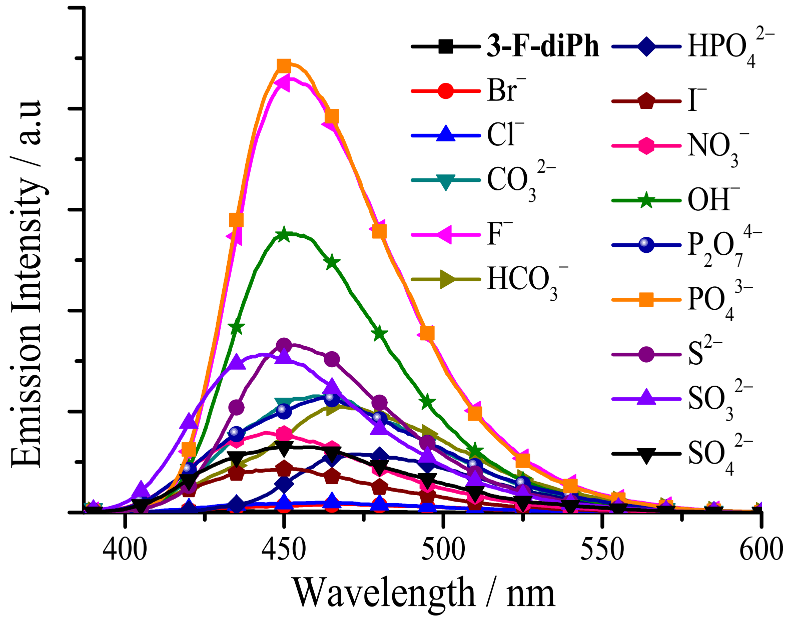

Fig. S27 Emission spectra of **3-F-diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

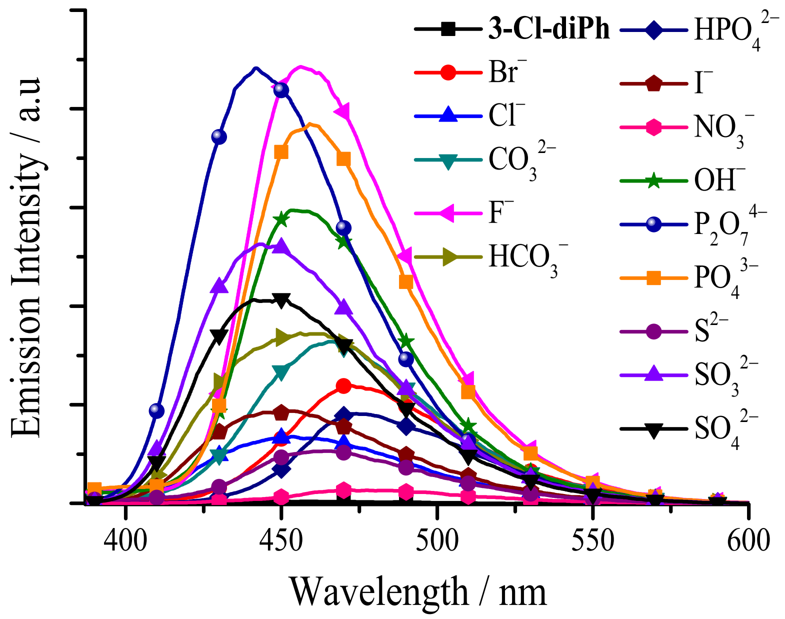

Fig. S28 Emission spectra of **3-Cl-diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions.

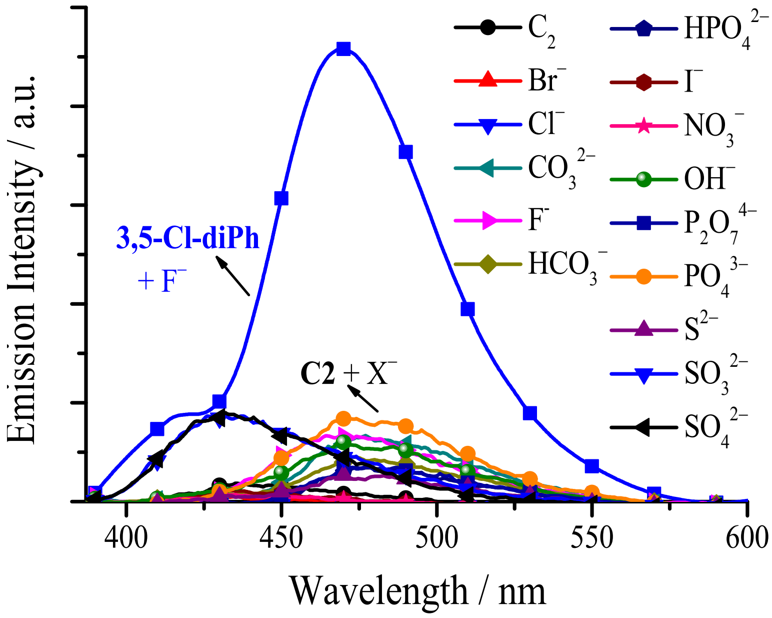

Fig. 29 Emission spectra of C_2 and **3,5-Cl-diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different anions and upon the addition of 100 equivalent of F^-, respectively.

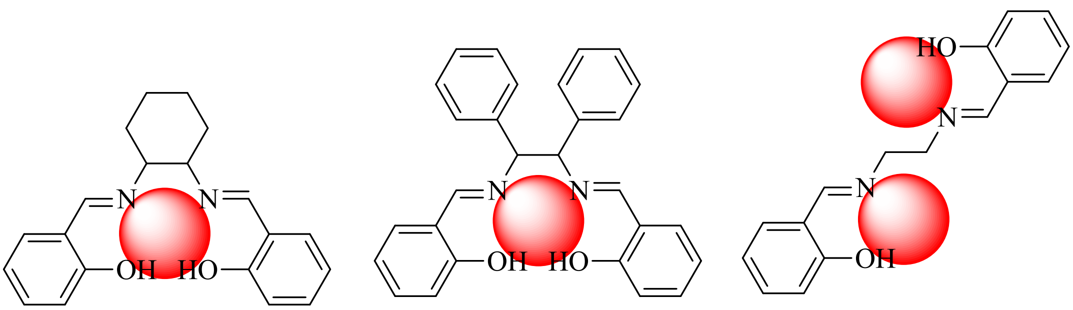

Fig. 30 Possible interaction mechanism between the dye and anion.

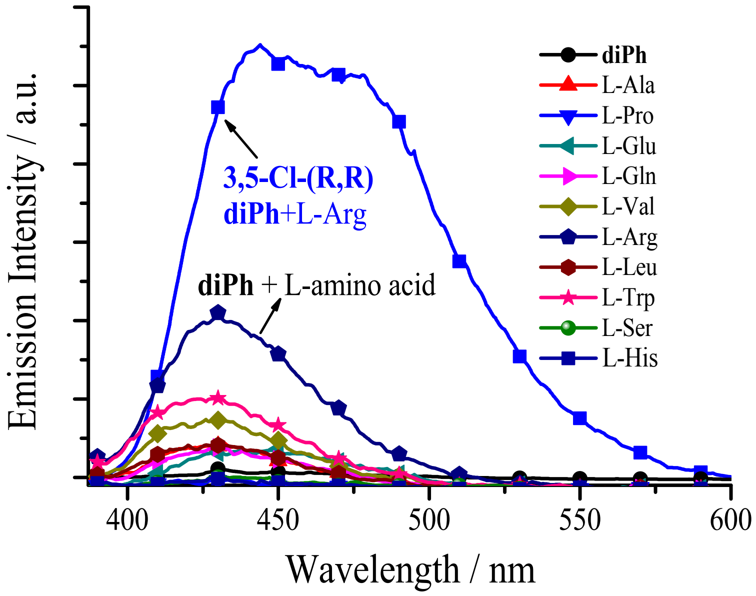

Fig. 31 Emission spectra of **diPh** and **3,5-Cl-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of different L- amino acid and upon the addition of 100 equivalent of L-Arg, respectively

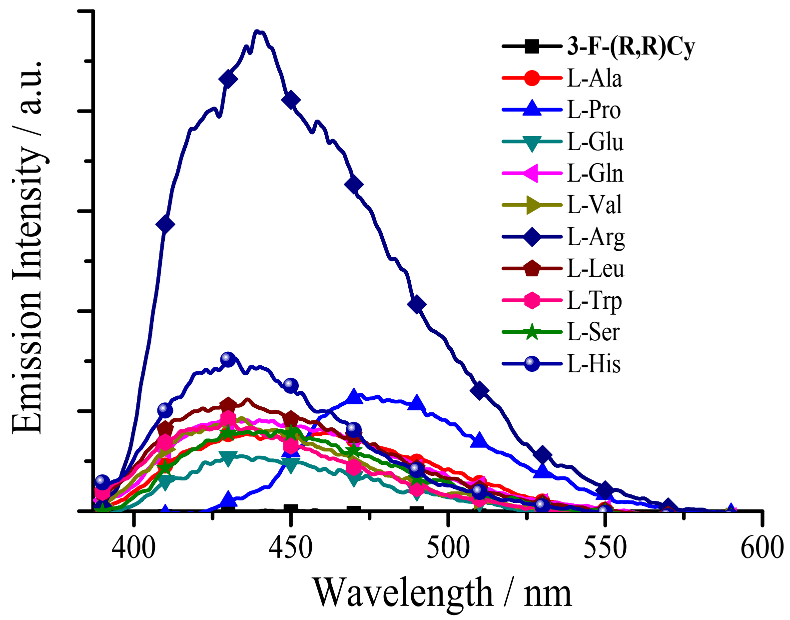

Fig. S32 Emission spectra of **3-F-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of different 100 equivalent of L-amino acid.

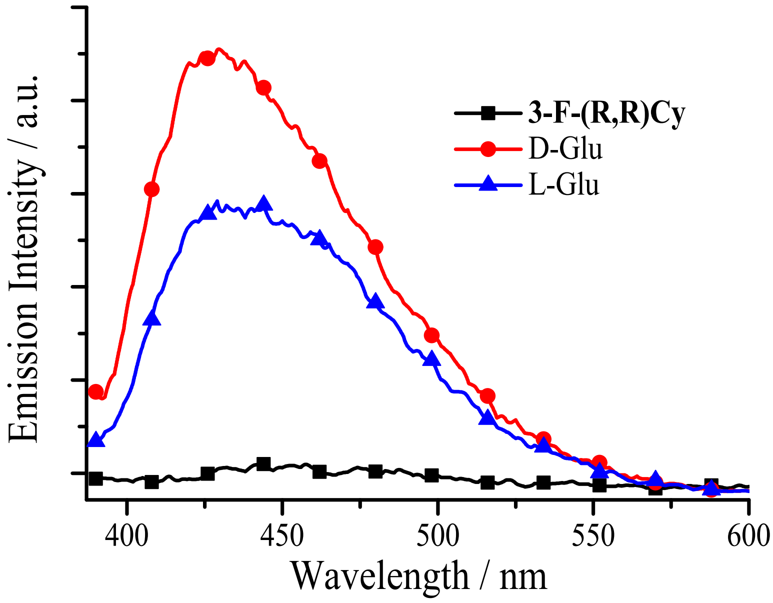

Fig. S33 Emission spectra of **3-F-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Glu.

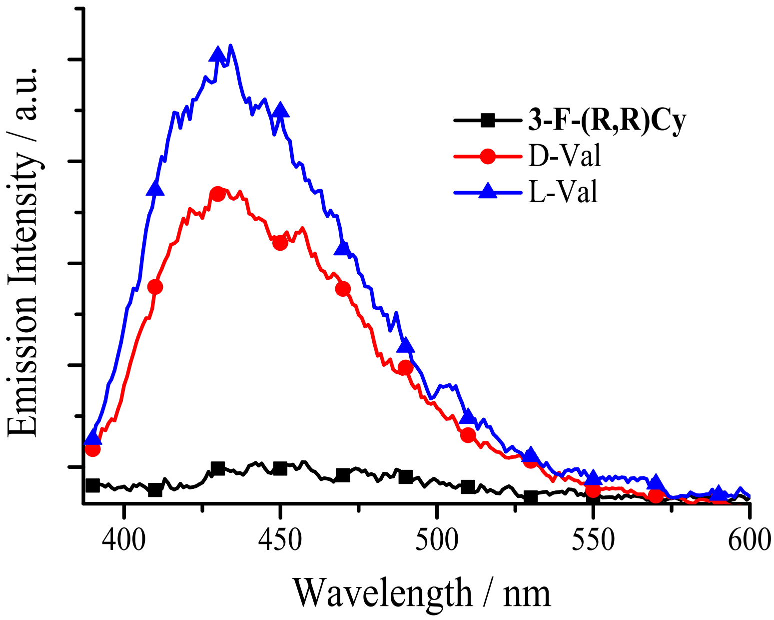

Fig. S34 Emission spectra of **3-F-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Val.

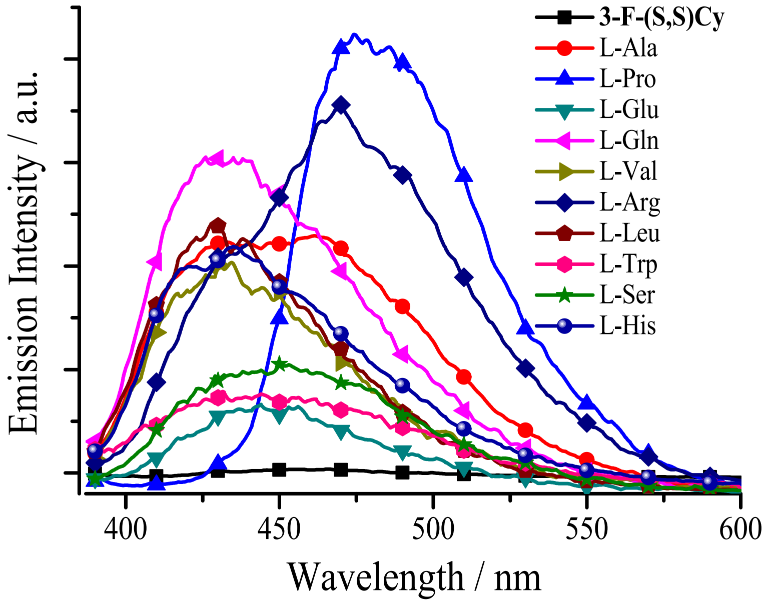

Fig. S35 Emission spectra of **3-F-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of different 100 equivalent of L-amino acid.

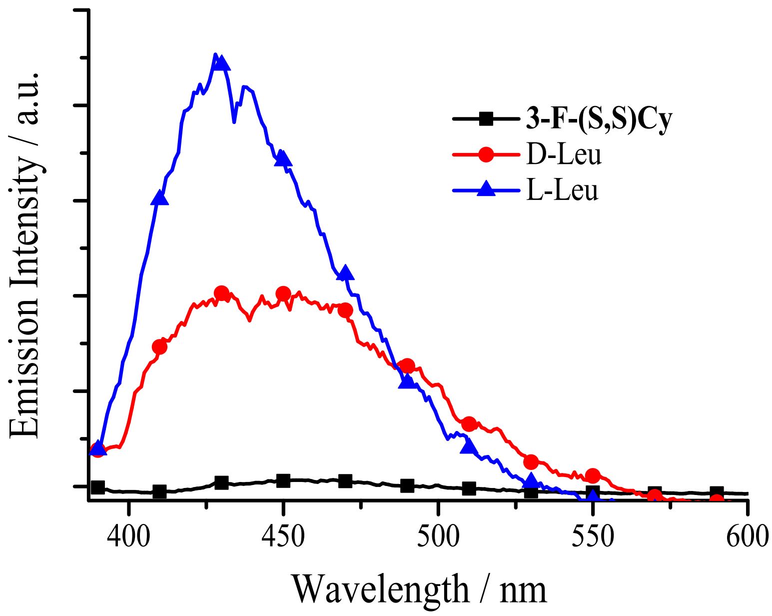

Fig. S36 Emission spectra of **3-F-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-Leu or D-Leu.

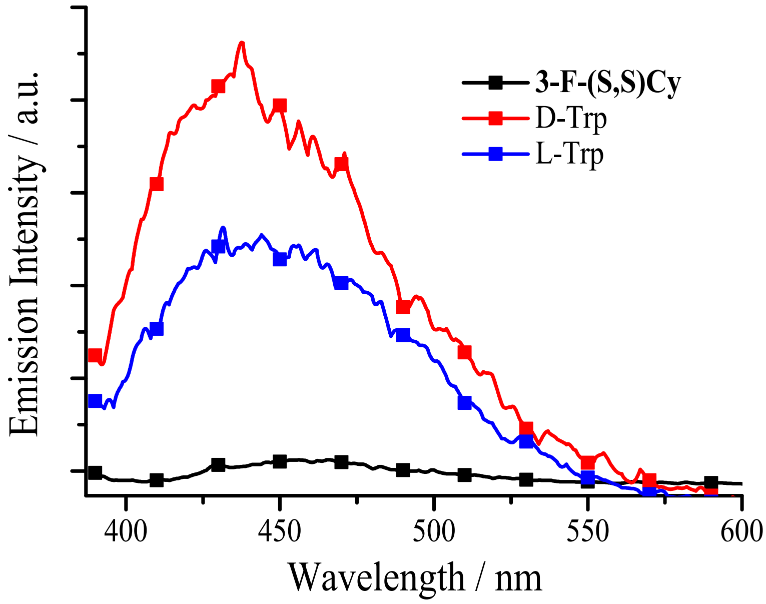

Fig. S37 Emission spectra of **3-F-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Trp.

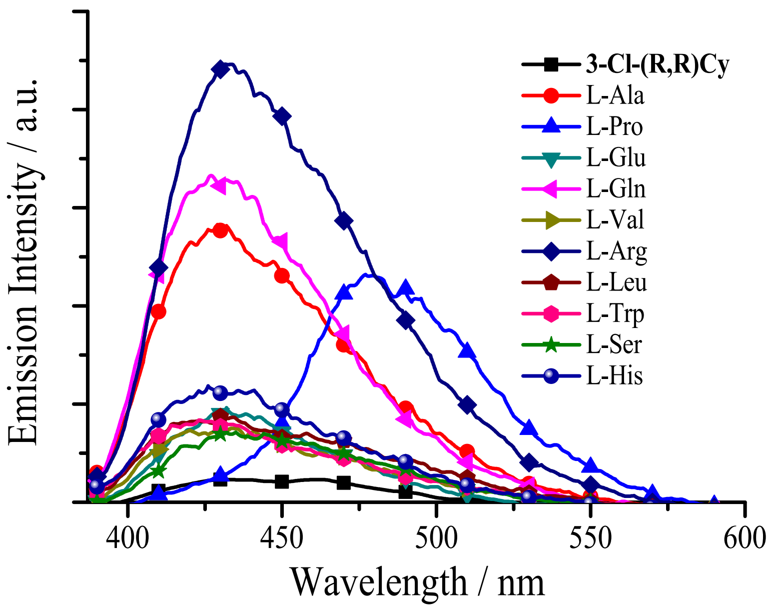

Fig. S38 Emission spectra of **3-Cl-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-amino acid.

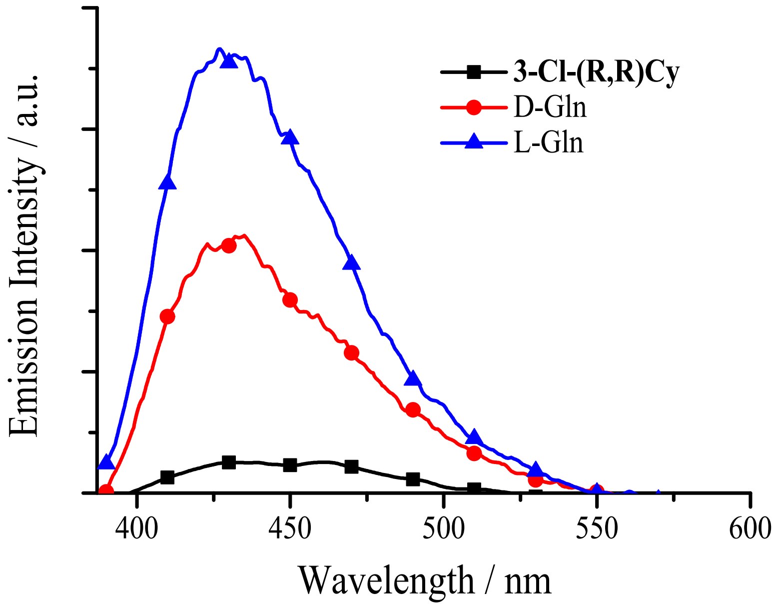

Fig. S39 Emission spectra of **3-Cl-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Trp.

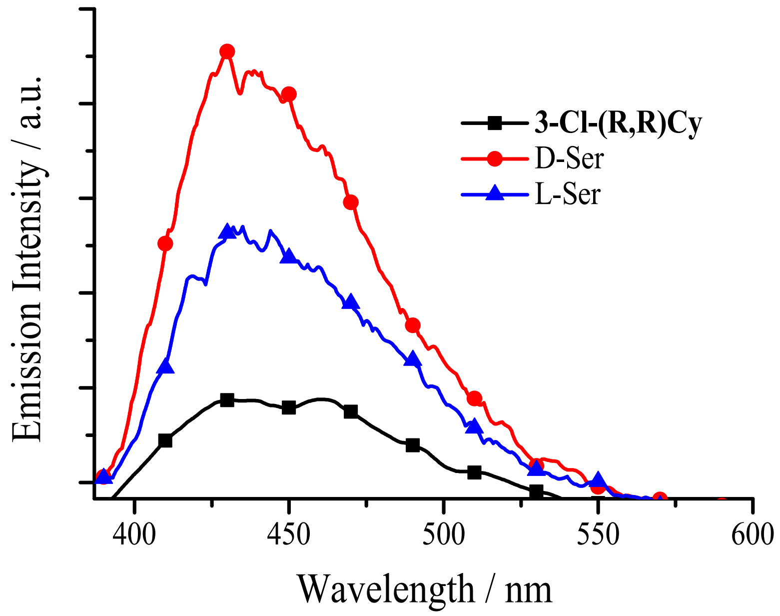

Fig. S40 Emission spectra of **3-Cl-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Ser.

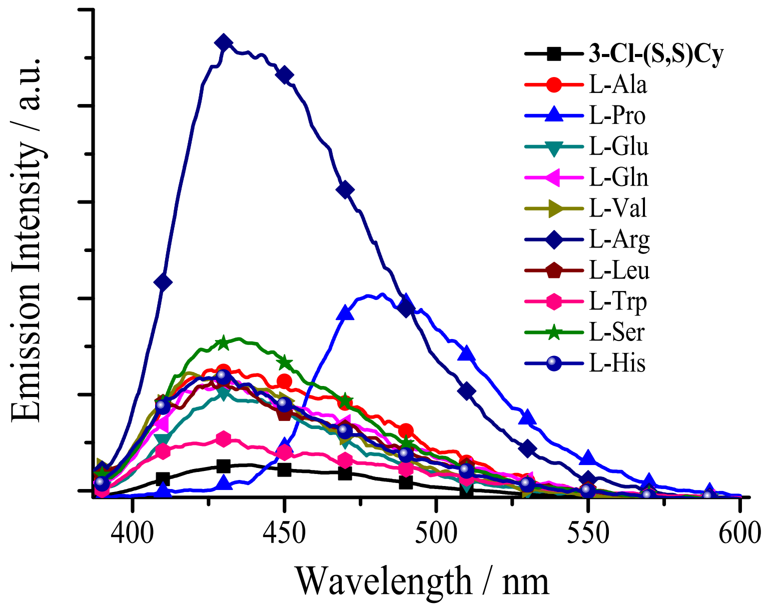

Fig. S41 Emission spectra of **3-Cl-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-amino acid.

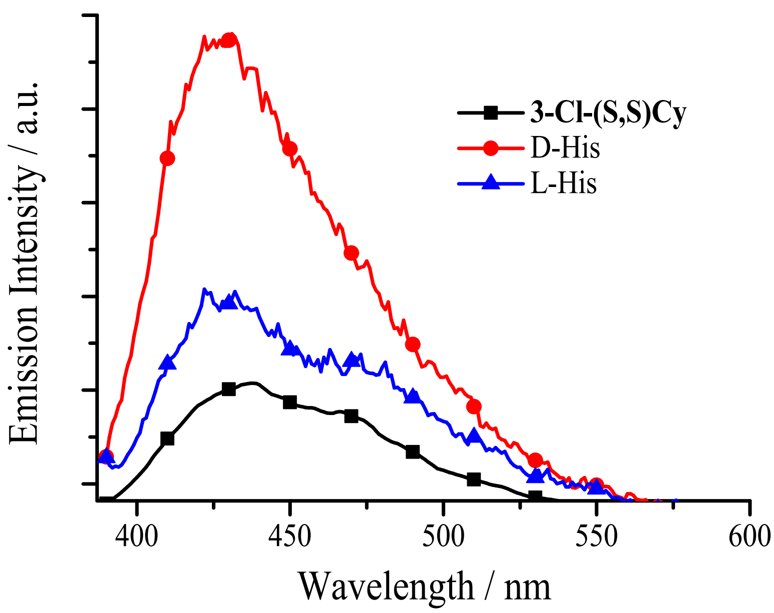

Fig. S42 Emission spectra of **3-Cl-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-His.

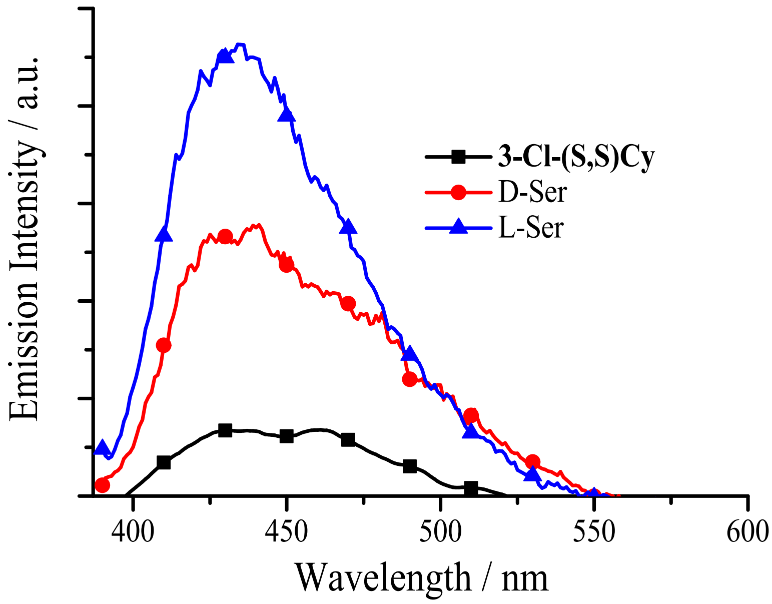

Fig. S43 Emission spectra of **3-Cl-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Ser.

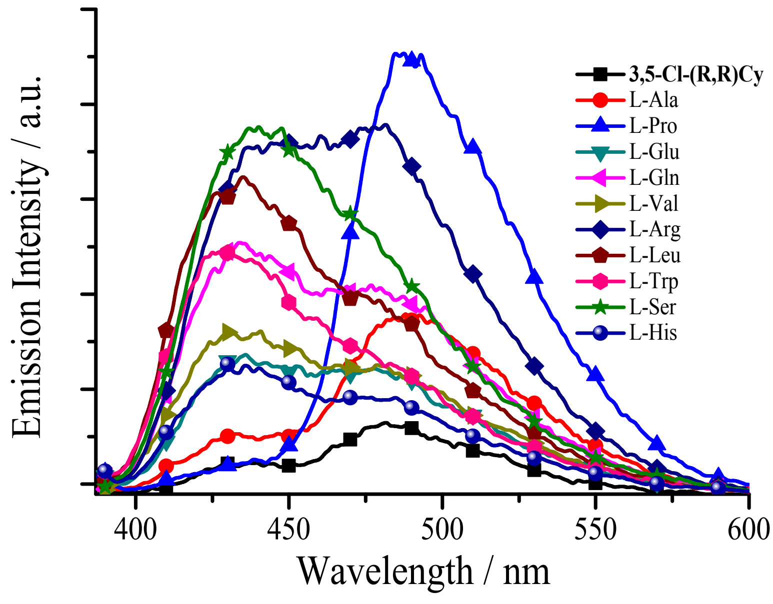

Fig. S44 Emission spectra of **3,5-Cl-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-amino acid.

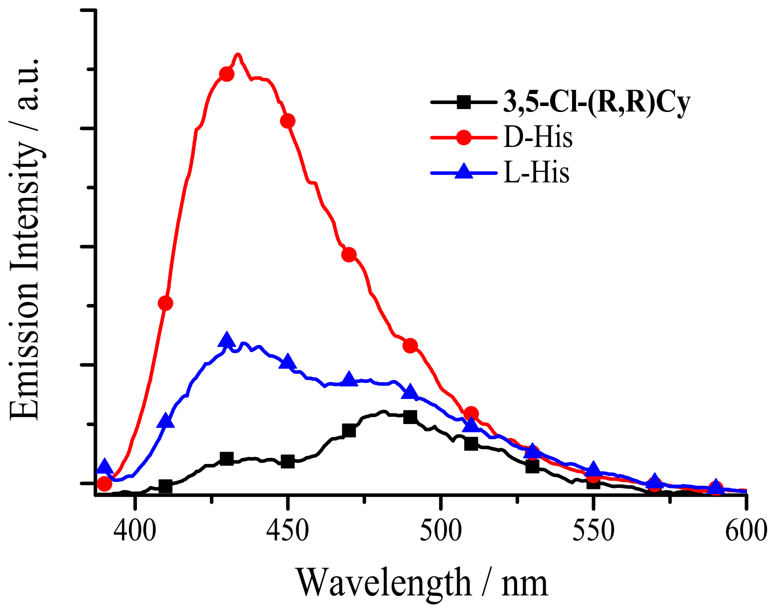

Fig. S45 Emission spectra of **3,5-Cl-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-His.

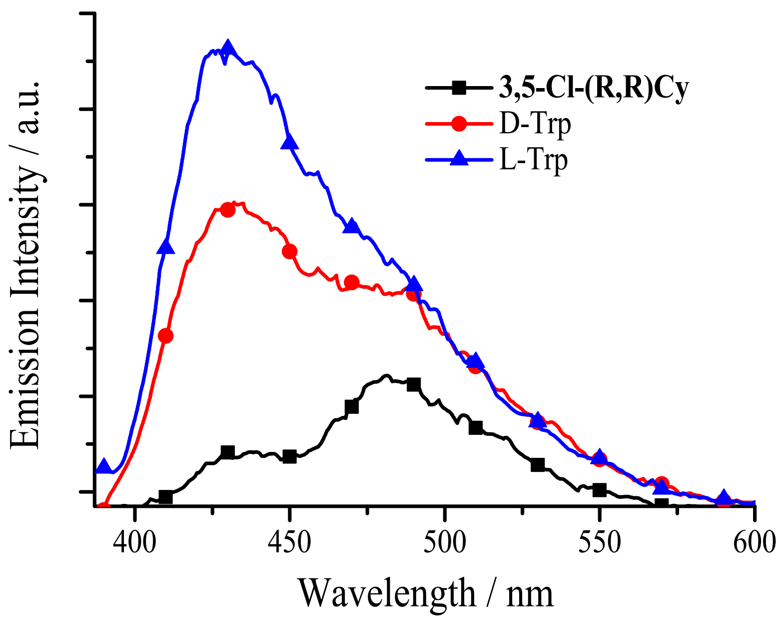

Fig. S46 Emission spectra of **3,5-Cl-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Trp.

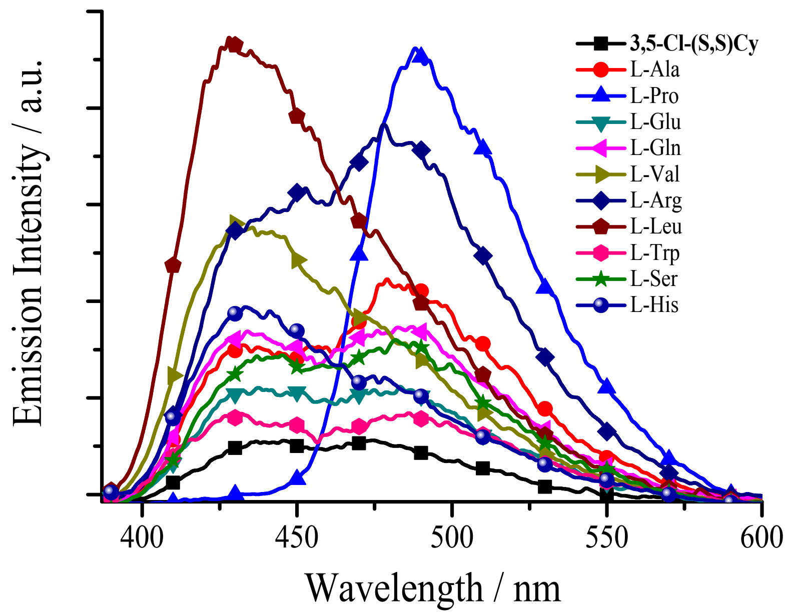

Fig. S47 Emission spectra of **3,5-Cl-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-amino acid.

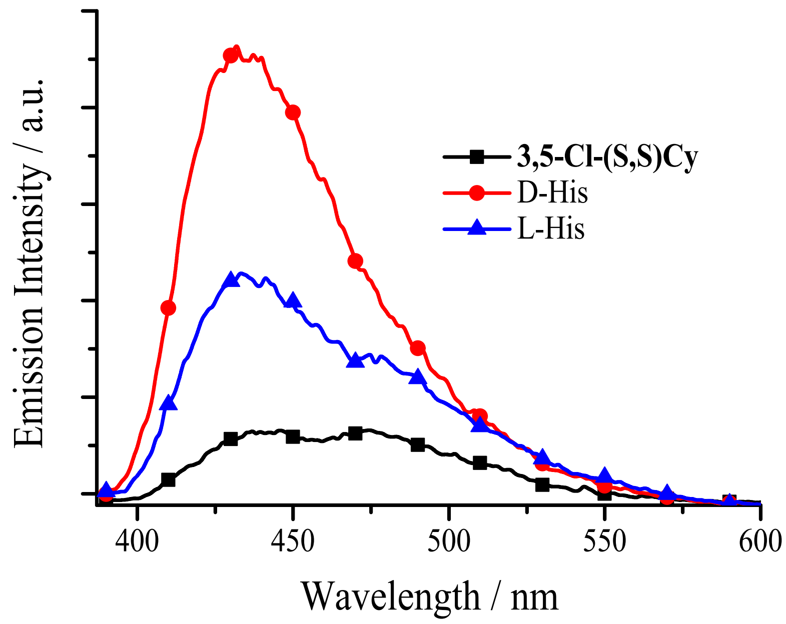

Fig. S48 Emission spectra of **3,5-Cl-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-His.

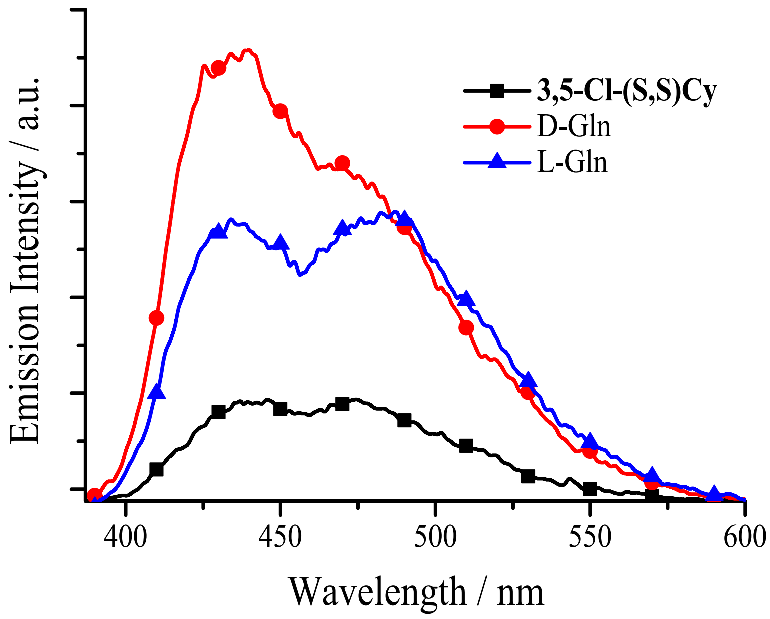

Fig. S49 Emission spectra of **3,5-Cl-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Gln.

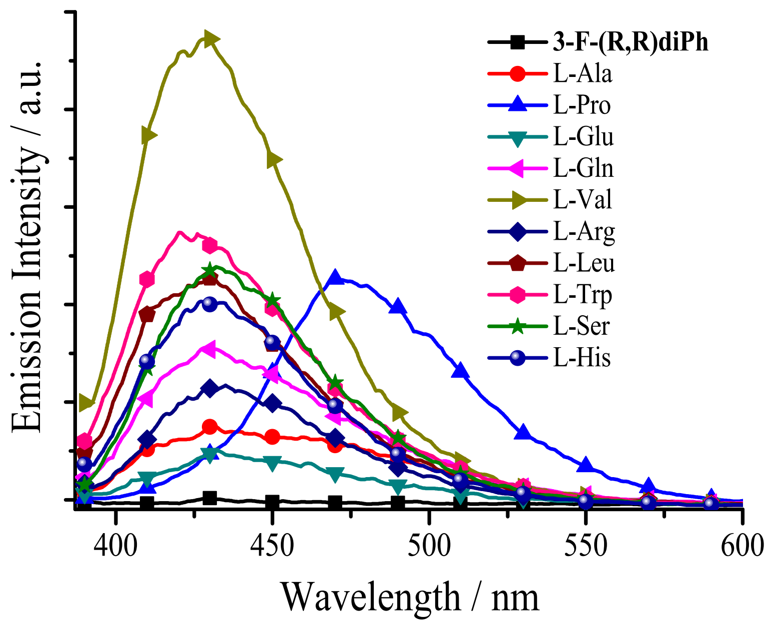

Fig. S50 Emission spectra of **3-F-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-amino acid.

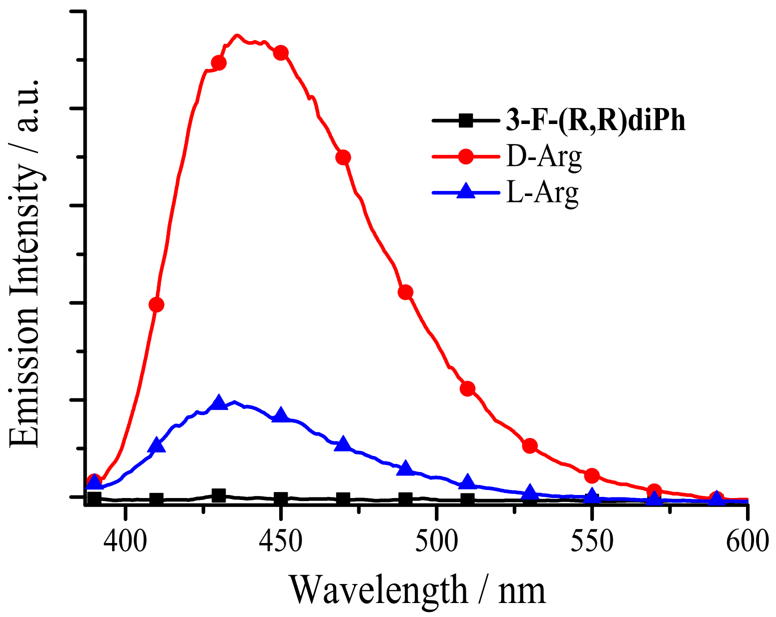

Fig. S51 Emission spectra of **3-F-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Arg.

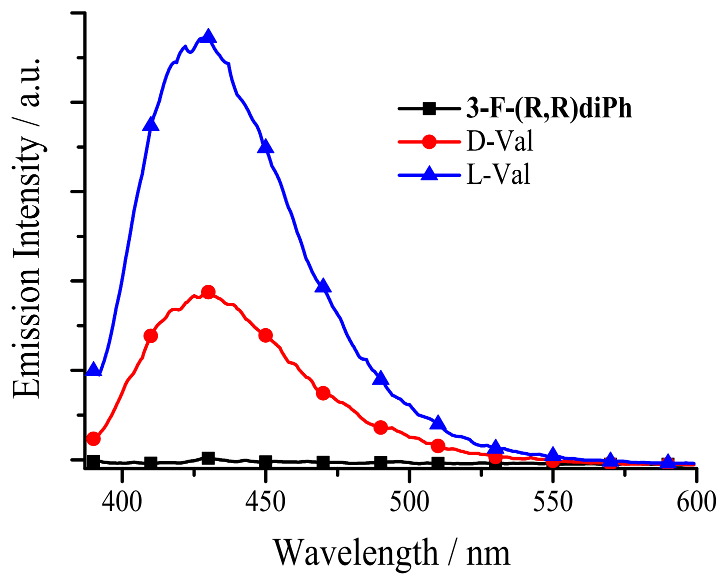

Fig. S52 Emission spectra of **3-F-(R,R)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Val.

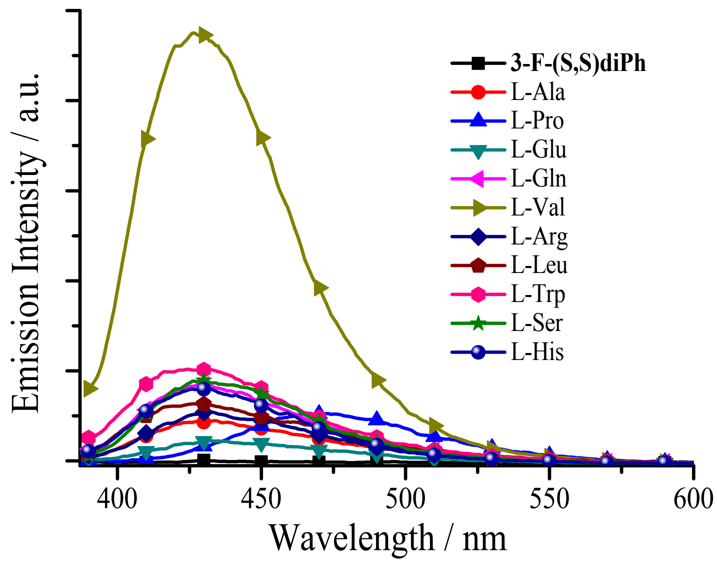

Fig. S53 Emission spectra of **3-F-(S,S)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L-amino acid.

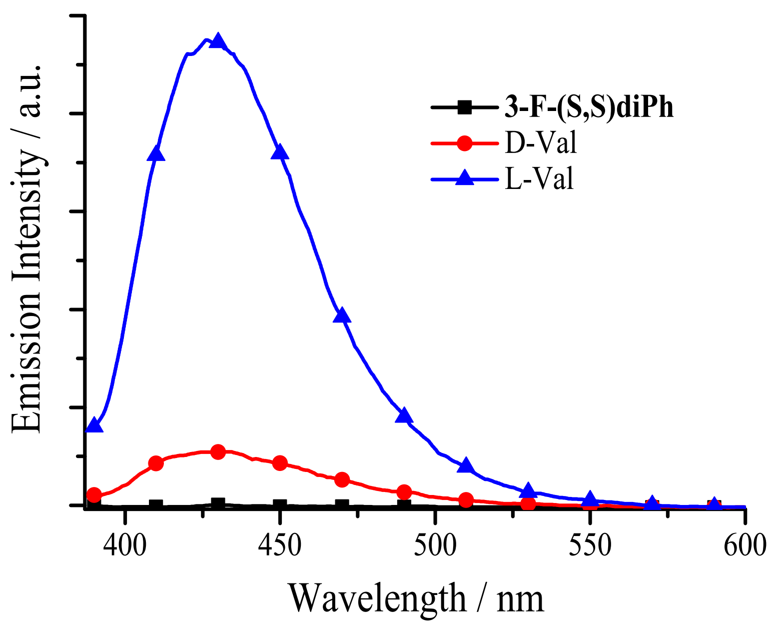

Fig. S54 Emission spectra of **3-F-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Val.

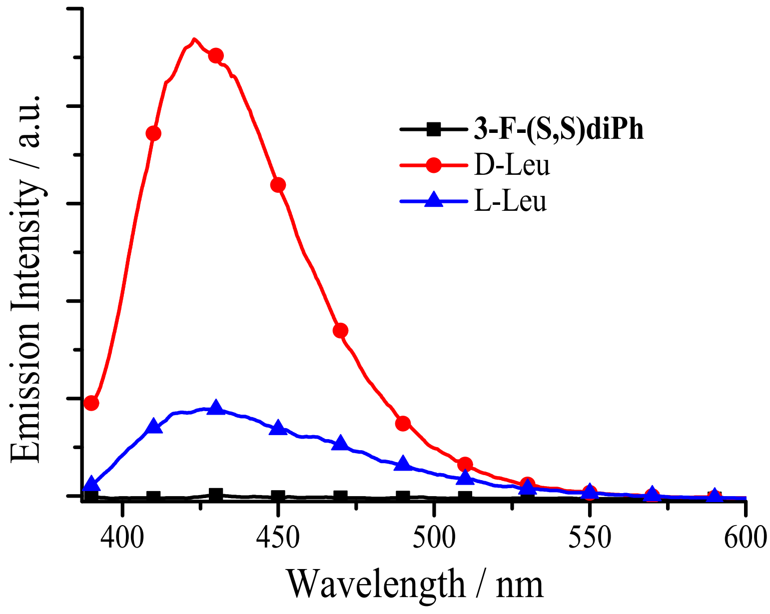

Fig. S55 Emission spectra of **3-F-(S,S)Cy** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Leu.

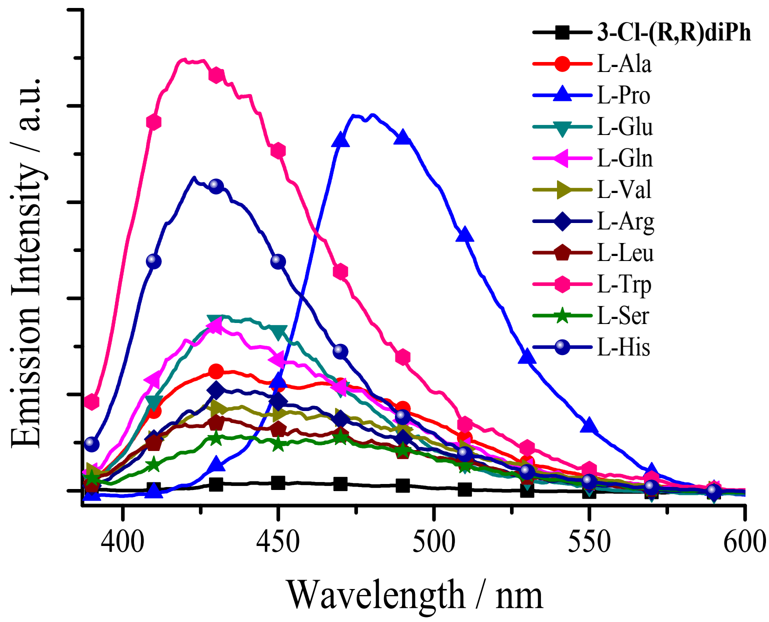

Fig. S56 Emission spectra of **3-Cl-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of different 100 equivalent of L-amino acid.

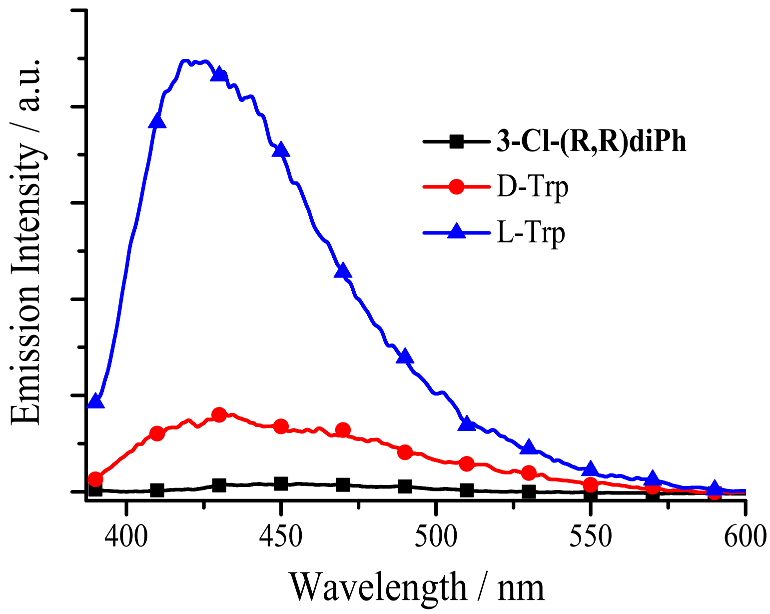

Fig. S57 Emission spectra of **3-Cl-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Trp.

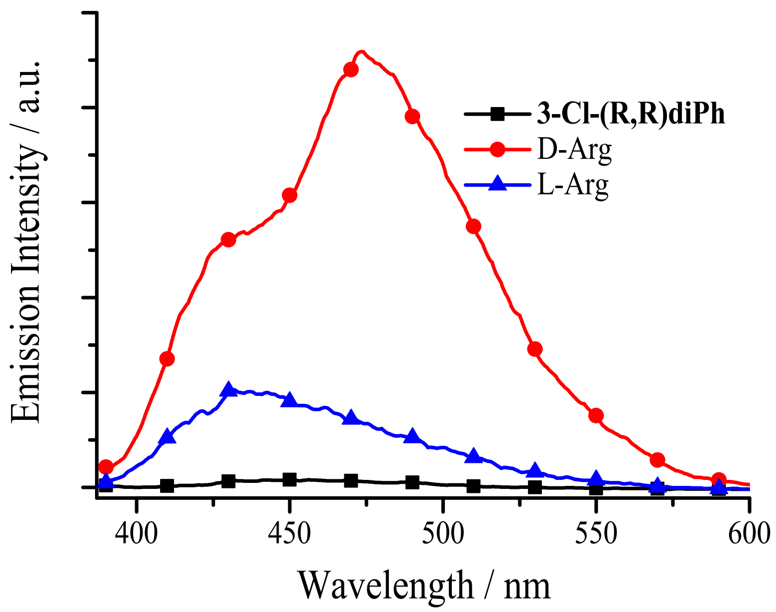

Fig. S58 Emission spectra of **3-Cl-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Arg.

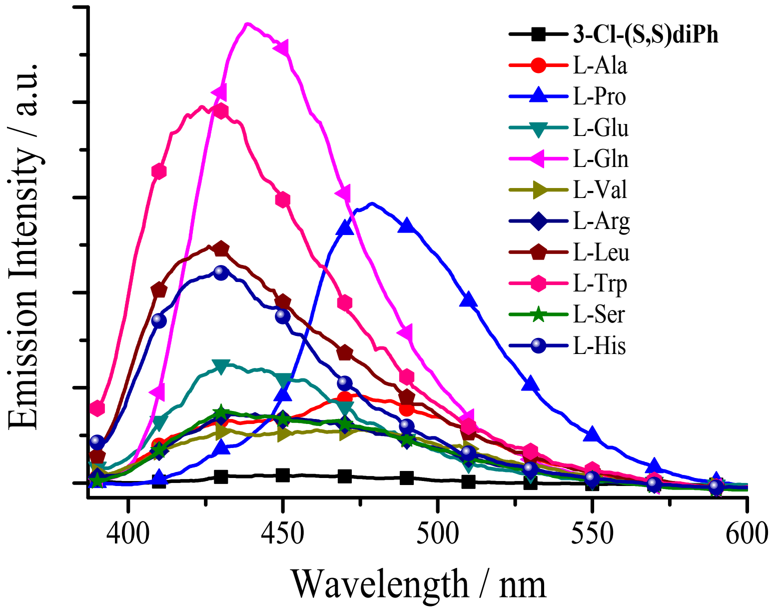

Fig. S59 Emission spectra of **3-Cl-(S,S)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of different 100 equivalent of L-amino acid.

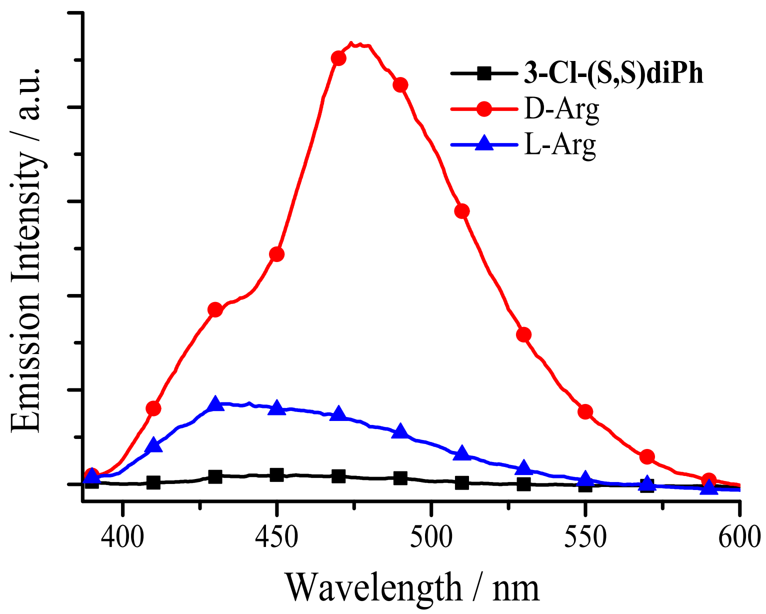

Fig. S60 Emission spectra of **3-Cl-(S,S)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Arg.

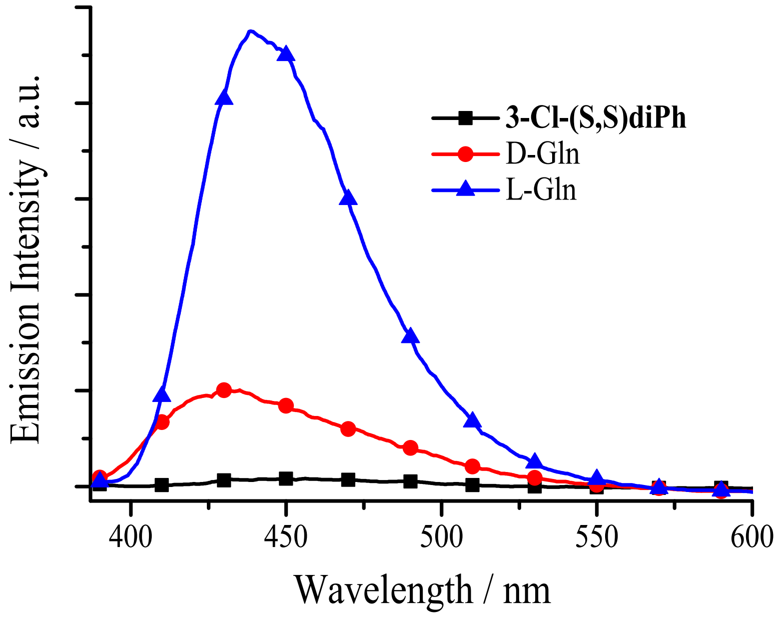

Fig. S61 Emission spectra of **3-Cl-(S,S)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of 100 equivalent of L- or D-Gln.

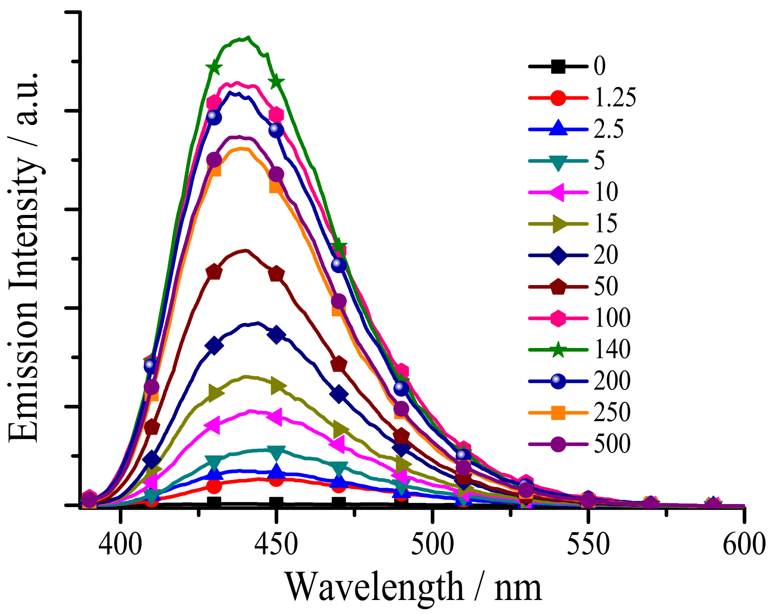

Fig.. S62 Emission spectra of **3,5-Cl-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO, excited at 380 nm) upon the addition of different equivalents of L-Ser.

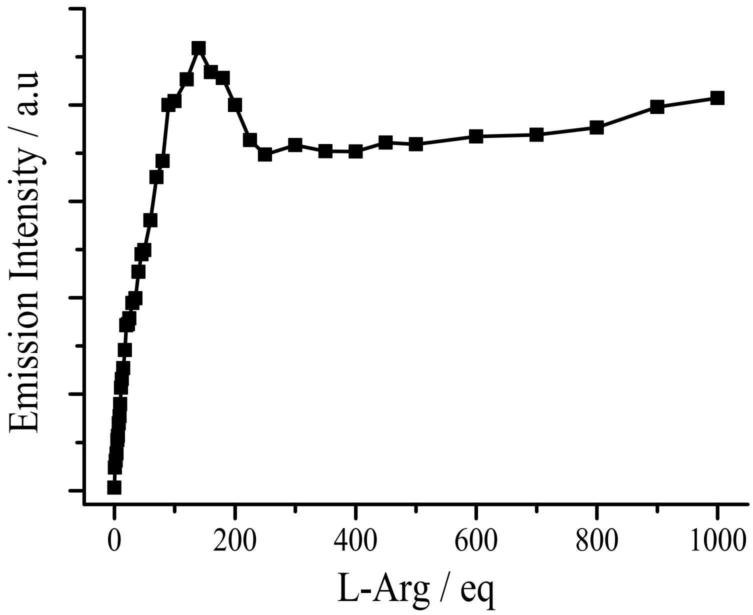

Fig. S63 Plot of emission intensity of **3,5-Cl-(R,R)diPh** (1.0×10^{-5} mol dm^{-3} in DMSO) at 440 nm (excited at 380 nm) as a function of L-Ser concentration.

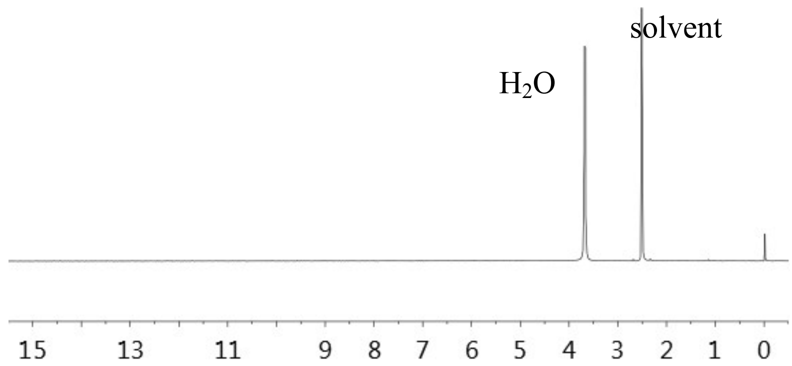

Fig. S64 ^{1}H NMR spectra of OH^- (top) and F^- (bottom) in DMSO-d_6.

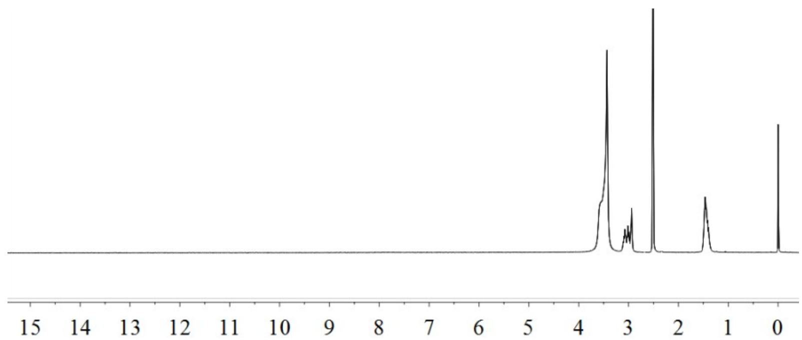

Fig. S65 ^{1}H NMR spectra of L-Arg in DMSO-d_6.

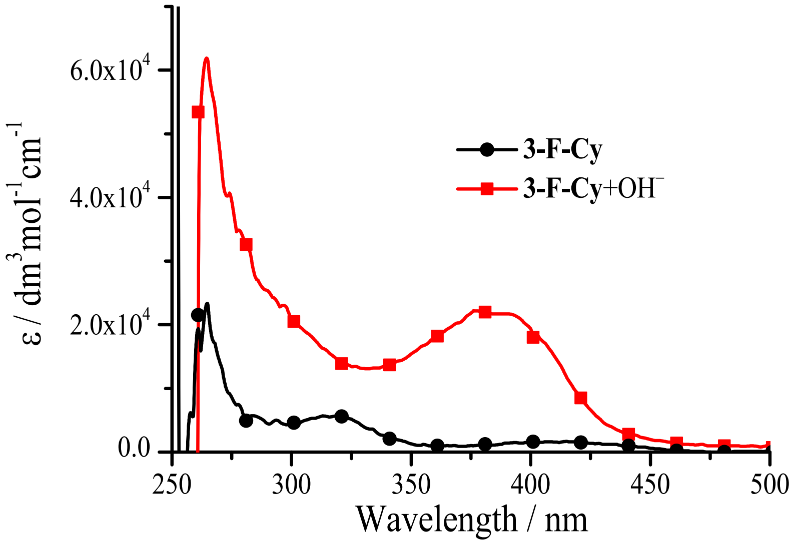

Fig. S66 Absorption spectra of **3-F-Cy** (1.0×10^{-5} mol dm^{-3} in DMSO) upon the addition of 100 equivalent of OH^-.