

Dynamically Stable co-Assembled Supramolecular BOPPY Systems with Chiral Amplification

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1. General methods

Reagents and solvents were used as received from commercial suppliers unless noted otherwise. All reactions were performed in oven-dried or flame-dried glassware unless otherwise stated and monitored by TLC using 0.25 mm silica gel plates with UV indicator. ^1H and ^{13}C NMR were recorded on a 400 MHz or 500 MHz NMR spectrometer at room temperature. Chemical shifts (δ) are given in ppm relative to internal TMS in CDCl_3 . High-resolution mass spectra (HRMS) were obtained using quadrupole-time-of-flight mass spectrometers with APCI ion source in positive mode.

Crystals of (R)-**1a** (CCDC 2232686), (S)-**1a** (CCDC 2232685), and (S)-**1b** (CCDC 2232684) suitable for X-ray analysis were obtained by slow diffusion of hexane into their dichloromethane solutions. Diffraction was performed on a Bruker SMART APEXII CCD area detector diffractometer using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) at 293(2) K, with φ and ω scan techniques. An empirical absorption correction was applied with the SADABS program. The structures were solved using Olex2¹ by direct methods using the ShelXT structure solution program. Non-hydrogen atoms were anisotropically refined by full-matrix least-squares calculations based on F^2 using the ShelXL program. Hydrogen atom coordinates were calculated with SHELXTL by using an appropriate riding model with varied thermal parameters. The residual electron densities were not chemically significant. The corresponding data could be obtained from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

UV-visible absorption and fluorescence emission spectra were recorded on commercial spectrophotometers (UV-2450 and Edinburg FLS 1000) at room temperature (10-mm quartz cuvette). Absolute luminescence efficiency (Φ_F) of chiral BOPPY dyes **1a** and **1b** in different solvents are measured by comparing the areas under the corrected emission spectrum of the test sample using an integrating sphere according to the definition of fluorescence efficiency. The enantiomers were analysed by reverse-phase high-pressure liquid chromatography (HPLC) using a chiral Ig

column. The analysis was done by a HPLC system. Chromatography condition: hexane/isopropyl alcohol = 78/22 (v/v), 1 mL/min, 25 °C.

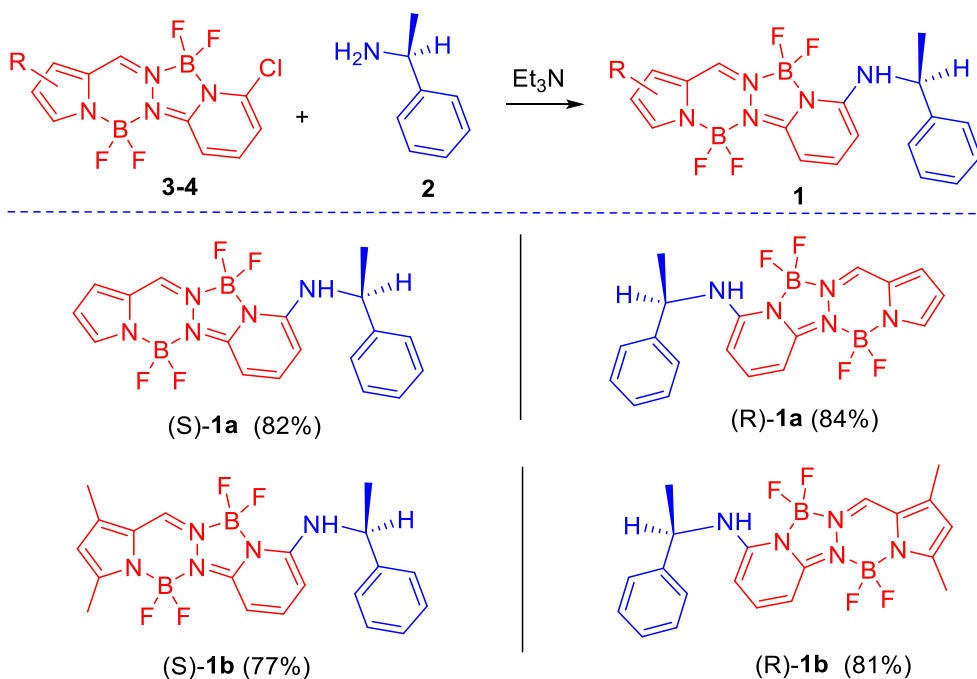
Circular dichroism (CD) spectra were obtained on a BioLogic MOS-500 circular dichroism spectrometer. Samples were prepared for testing in quartz cuvettes with light path of 0.1 mm. For the CD spectroscopy, the difference in absorbance between the left and right circularly polarized light, represented as $CD = \Delta A = A_{LCP} - A_{RCP}$, was calculated. The delta absorbance (ΔA) was determined using the Beer-Lambert law, which stated that $\Delta A = (\epsilon_L - \epsilon_R) \cdot C \cdot L = \Delta \epsilon \cdot C \cdot L$ (where A represents absorbance, $\Delta \epsilon$ represents molar circular dichroism, C represents molar concentration, and L represents cuvette pathlength). CD spectra were obtained using the Bio-Kine Software, with θ being the unit displayed in mdeg (one mdeg equals 0.001 deg) and is also known as standard ellipticity. To convert ellipticity to circular dichroic absorption (ΔA or ΔOD), the following equation was used: $\Delta A = \Delta OD = \theta/32980$. The parameter gabs is calculated as $\Delta A/A$, with values ranging from -1 to 1.

Circularly polarized luminescence (CPL) measurements were performed on an OLIS CPL SOLO spectrometer. The performance of circularly polarized luminescence (CPL) was evaluated by the luminescence dissymmetry factor (g_{lum}). $g_{lum} = 2(I_L - I_R)/(I_L + I_R)$ ($-2 \leq g_{lum} \leq 2$), where I_L and I_R mean the strength of the left and right circularly polarized emissions. To calibrate the CPL spectrometer, standard $Eu(facam)_3$ (Europium(III) tris[3-(trifluoromethylhydroxymethylene)-d-camphorate]) (1 mM in DMSO) was used. The emission wavelength range was set from 570 nm to 640 nm. The theoretical value of g_{lum} for $Eu(facam)_3$ has been reported to peak at approximately -0.78 at 595 nm. The measured value was about -0.77 at 595 nm, which was agreed to the theoretical value. For the CPL test of a co-assembled supramolecular system, the sample was shaken and placed in a quartz cell. For the solid CPL, KBr and our BOPPY (mass ratio = 50:1) were carefully grounded together and pressed onto two quartz slides. A number of spectra were accumulated and then averaged to reduce the influence of accidental errors. All samples were excited at 390 nm unless otherwise noted.

Scanning electron microscopy (SEM) images were recorded on a Hitachi S-4800

FE-SEM instrument with an accelerating voltage of 10 kV, the samples were prepared on single-crystal silicon wafer, dried under vacuum, and coated with a thin layer of Pt to increase the contrast. Transmission electron microscopy (TEM) images were obtained on a T20 electron microscope at an accelerating voltage of 110 kV. A small amount of the suspension was diluted with a mixed solvent of acetonitrile/di-water, dropped on the surface of the copper mesh of the carbon support film, dried in vacuum, and observed under an electron microscope. Laser scanning confocal microscopy (LSCM) images were recorded on a Leica instrument with an accelerating voltage of 10 kV, the samples were prepared on quartz plate, dried under vacuum.

2. Experimental procedures



Scheme S1. The synthesis and yields of (S/R)-1-phenylethan-1-amine modified BOPPYs **1a-b**.

The general synthesis procedures for BOPPYs **1a** and **1b**

To BOPPY dye **3** (0.2 mmol) in 1,2-dichloroethane (60 mL) were added (R/S)-1-phenylethan-1-amine **2a** or **2b** (1.2 mmol, 6 equiv.) and Et_3N (0.6 mmol, 3 equiv.). The reaction mixture was stirred at 80 °C for 12 h. After that, the reaction mixture was removed under vacuum. The crude product was purified from chromatograph (silica gel, hexane/ CH_2Cl_2 = 1/1, v/v), obtaining the chiral BOPPY as a yellow or orange powder.

Synthesis of (S)-1a: (S)-1a was prepared from BOPPY dye **3** (89 mg, 0.28 mmol), Et₃N (0.12 mL, 0.84 mmol, 3 equiv.) and (S)-1-phenylethan-1-amine **2a** (0.22 mL, 1.7 mmol, 6 equiv.) as a yellow solid in 82% yield (93 mg, 0.23 mmol). (S)-1a was measured by HPLC using a chiral stationary phase [*n*-hexane:*i*-PrOH = 82:18, 1 mL/min), t_R = 11.442 min]. ¹H NMR (500 MHz, CDCl₃) δ 7.73 (s, 1H), 7.59 (s, 1H), 7.49 (t, *J* = 8.3 Hz, 1H), 7.40 – 7.34 (m, 2H), 7.33 – 7.26 (m, 3H), 7.03 (d, *J* = 3.4 Hz, 1H), 6.67 (d, *J* = 8.2 Hz, 1H), 6.57 (dd, *J* = 3.6, 2.1 Hz, 1H), 5.78 (d, *J* = 8.3 Hz, 1H), 5.56 (d, *J* = 4.5 Hz, 1H), 4.66 – 4.57 (m, 1H), 1.64 (d, *J* = 6.8 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 150.8, 150.34, 145.3, 142.1, 132.4, 130.5, 129.3, 128.1, 125.5, 121.6, 115.7, 97.5, 96.7, 53.2, 24.7. ¹¹B NMR (128 MHz, CDCl₃) δ 3.14 (t, *J* = 29.0 Hz), 1.13 (t, *J* = 29.2 Hz). ¹⁹F NMR (377 MHz, CDCl₃) δ -142.31 – -143.63 (m, 2F), -148.10 (m, 2F). HRMS (APCI) calcd. for C₁₈H₁₈B₂F₄N₅[M + H]⁺: 402.1684, found: 402.1670.

Synthesis of (R)-1a: (R)-1a was prepared from BOPPY dye **3** (102 mg, 0.32 mmol), Et₃N (0.13 mL, 0.97 mmol, 3 equiv.) and (R)-1-phenylethan-1-amine **2b** (0.25 mL, 1.94 mmol, 6 equiv.) as a yellow solid in 84% yield (109 mg, 0.23 mmol). (R)-1a was measured by HPLC using a chiral stationary phase [*n*-hexane:*i*-PrOH = 82:18, 1 mL/min), t_R = 12.567 min]. ¹H NMR (400 MHz, CDCl₃) δ 7.72 (s, 1H), 7.59 (s, 1H), 7.48 (t, *J* = 8.3 Hz, 1H), 7.41 – 7.33 (m, 2H), 7.31 (d, *J* = 7.4 Hz, 3H), 7.02 (d, *J* = 3.7 Hz, 1H), 6.67 (d, *J* = 8.3 Hz, 1H), 6.56 (dd, *J* = 3.7, 2.2 Hz, 1H), 5.77 (d, *J* = 8.3 Hz, 1H), 5.55 (d, *J* = 5.3 Hz, 1H), 4.82 – 4.45 (m, 1H), 1.63 (d, *J* = 6.8 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 150.8, 150.4, 145.3, 142.1, 132.4, 130.5, 129.3, 125.5, 124.4, 121.6, 97.4, 96.6, 53.2, 24.6. ¹¹B NMR (128 MHz, CDCl₃) δ 3.15 (d, *J* = 57.7 Hz), 1.14 (t, *J* = 29.2 Hz). ¹⁹F NMR (376 MHz, CDCl₃) δ -142.51 – -143.92 (m, 2F), -148.08 (m, 2F). HRMS (APCI) calcd. for C₁₈H₁₈B₂F₄N₅ [M + H]⁺: 402.1684, found: 402.1694.

Synthesis of (S)-1b: (S)-1b was prepared from BOPPY dye **4** (82 mg, 0.24 mmol), Et₃N (0.1 mL, 0.71 mmol, 3 equiv.) and (S)-1-phenylethan-1-amine **2a** (0.18 mL, 1.4 mmol, 6 equiv.) as a yellow solid in 77% yield (79 mg, 0.18 mmol). (S)-1b was measured by HPLC using a chiral stationary phase [*n*-hexane:*i*-PrOH = 82:18, 1 mL/min), t_R = 9.975 min]. ¹H NMR (500 MHz, CDCl₃) δ 7.57 (s, 1H), 7.43 (t, *J* = 8.4 Hz, 1H), 7.40 – 7.34 (m, 2H), 7.34 – 7.27 (m, 3H), 6.63 (d, *J* = 8.4 Hz, 1H), 6.13 (s, 1H), 5.69 (d, *J* = 8.2 Hz, 1H), 5.53 (d, *J* = 5.8 Hz, 1H), 4.62 (m, 1H), 2.47 (s, 3H),

2.31 (s, 3H), 1.64 (d, $J = 6.8$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 150.5, 150.3, 146.5, 144.5, 142.3, 135.0, 129.1, 127.8, 127.3, 125.4, 122.8, 117.3, 96.4, 96.1, 53.0, 24.6, 13.9, 10.9. ^{11}B NMR (128 MHz, CDCl_3) δ 3.12 (t, $J = 29.4$ Hz), 1.06 (t, $J = 31.2$ Hz). ^{19}F NMR (471 MHz, CDCl_3) δ -140.66 – -142.23 (m, 2F), -147.37 (m, 2F). HRMS (APCI) calcd. for $\text{C}_{20}\text{H}_{21}\text{B}_2\text{F}_3\text{N}_5[\text{M} - \text{F}]^+$: 410.1935, found: 410.1942.

Synthesis of (R)-1b: (R)-1b was prepared from BOPPY dye **4** (103 mg, 0.3 mmol), Et_3N (0.12 mL, 0.90 mmol, 3 equiv.) and (R)-1-phenylethan-1-amine **2b** (0.23 mL, 1.8 mmol, 6 equiv.) as a yellow solid in 81% yield (104 mg, 0.24 mmol). (R)-1b was measured by HPLC using a chiral stationary phase [*n*-hexane:*i*-PrOH = 82:18, 1 mL/min), $t_{\text{R}} = 10.425$ min]. ^1H NMR (500 MHz, CDCl_3) δ 7.57 (s, 1H), 7.43 (t, $J = 8.4$ Hz, 1H), 7.40 – 7.34 (m, 2H), 7.34 – 7.27 (m, 3H), 6.63 (d, $J = 8.4$ Hz, 1H), 6.13 (s, 1H), 5.69 (d, $J = 8.2$ Hz, 1H), 5.53 (d, $J = 5.8$ Hz, 1H), 4.62 (m, 1H), 2.47 (s, 3H), 2.31 (s, 3H), 1.64 (d, $J = 6.8$ Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 150.5, 150.3, 146.5, 144.5, 142.3, 135.0, 129.1, 127.8, 127.2, 125.4, 122.8, 117.3, 96.4, 96.1, 53.0, 24.6, 13.9, 10.9. ^{11}B NMR (128 MHz, CDCl_3) δ 3.12 (t, $J = 29.3$ Hz), 1.06 (t, $J = 31.2$ Hz). ^{19}F NMR (471 MHz, CDCl_3) δ -140.71 – -141.99 (m, 2F), -146.65 – -148.05 (m, 2F). HRMS (APCI) calcd. for $\text{C}_{20}\text{H}_{21}\text{B}_2\text{F}_3\text{N}_5[\text{M} - \text{F}]^+$: 410.1935, found: 410.1931.

Taking the preparation of as an example.

Preparation of SDS aqueous solution: To 60 mL deionized aqueous solution was added sodium dodecyl sulfate (120 mg, 0.42 mmol), and ultrasonically assisted dissolution for 1 min to obtain SDS aqueous solution with a concentration of 2 mg/mL.

Preparation of acetonitrile stock of chiral BOPPY dyes: (S)-1a (2.0 mg, 5 μmol) was dissolved in pure acetonitrile (1.7 mL) and then ultrasonically assisted dissolution for 1 min to obtain the stock acetonitrile solution with a concentration of 3 mM.

Synthesis of (S/R)-1a micelles: The prepared SDS aqueous solution (3 mL, 2 mg/mL) was placed in a 4 mL centrifuge tube, and the acetonitrile stock solution (S/R)-1a (10 μL , 3 mM) was injected with a pipetting gun to form the concentration of (S/R)-1a in the mixed solution of 10 μM . The cover of the centrifuge tube was covered, and the solution was shaken up and down for 20 times, and then left to age at room temperature (25 $^\circ\text{C}$) for 96 h to obtain stable BOPPY micelles.

Synthesis of (S/R)-1a assembled microrods: The prepared SDS aqueous solution (3 mL, 2 mg/mL) was placed in a 4 mL centrifuge tube, and the acetonitrile stock solution (S/R)-1a (30 μ L, 3 mM) was injected with a pipetting gun to form the concentration of (S/R)-1a in the mixed solution of 30 μ M. The cover of the centrifuge tube was covered, and the solution was shaken up and down for 20 times, and then left to age at room temperature (25 $^{\circ}$ C) for 96 h to obtain stable BOPPY assembled microrods. The concentrations from 30 to 100 μ M and time of chiral BOPPY dyes in the growth solution were tuned to obtain chiral nanocrystals with different sizes and morphologies.

Synthesis of (S/R)-1b micelles and assembled microrods: According to the above procedures of (S/R)-1a assemblies, the assembled nanostructures of (S/R)-1b were obtained adopting the same concentrations and time.

3. Chiroptical properties in molecular and solid states

3.1 CD and CPL of chiral BOPPYs (R/S)-1a and 1b

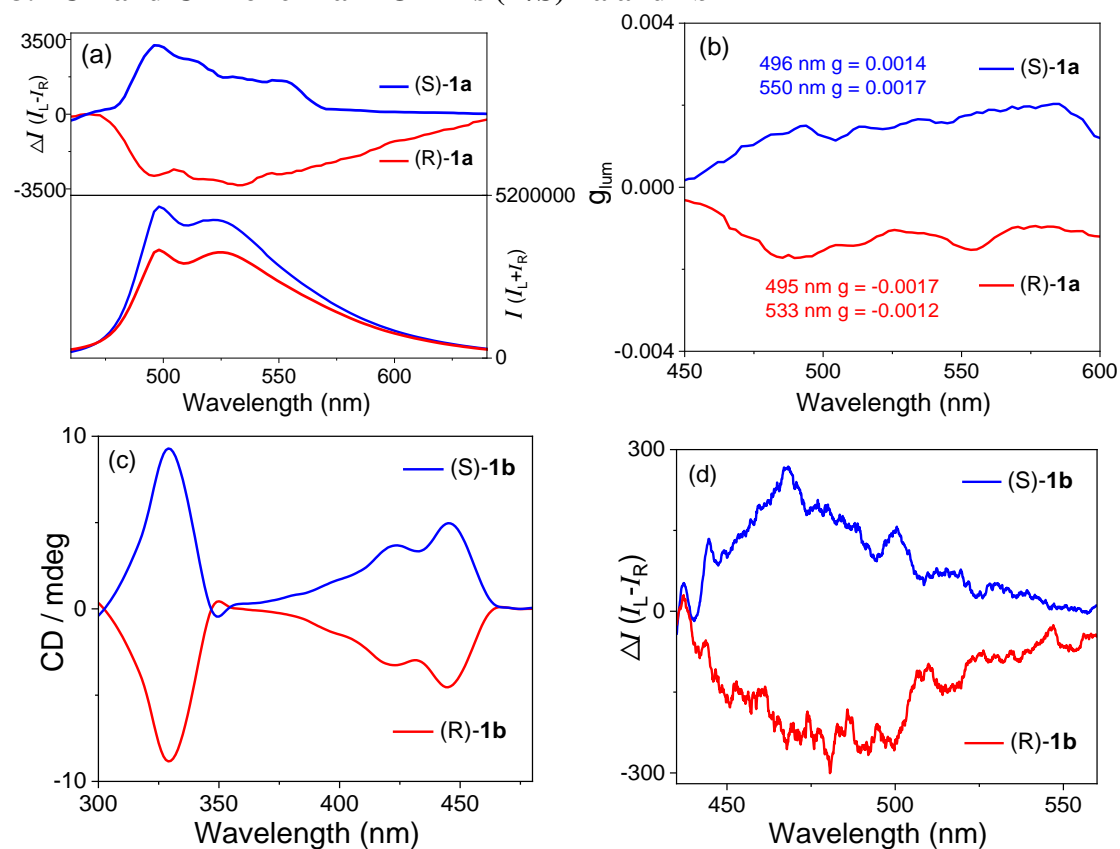


Figure S1. CPL and synchronous total luminescence spectra (a) and g_{lum} (b) of (S/R)-1a at grinding KBr tablets state. CD (c) and CPL (d) spectra of (S/R)-1b in hexane.

Table S1. The g_{abs} and g_{lum} of chiral BOPPYs (S/R)-**1a** and **1b** in hexane.

dyes	$\lambda_{\text{max}}/\text{nm}$ (g_{abs})	$\lambda_{\text{max}}/\text{nm}$ (g_{lum})
(S)- 1a	417 (1.397×10^{-4}), 439 (1.355×10^{-4})	449 (1.3×10^{-4}), 479 (1.6×10^{-4})
(R)- 1a	417 (-1.813×10^{-4}), 439 (-1.533×10^{-4})	452 (-2.3×10^{-4}), 475 (-2.4×10^{-4})
(S)- 1b	424 (1.045×10^{-4}), 449 (9.435×10^{-5})	468 (2.0×10^{-4})
(R)- 1b	424 (-1.223×10^{-4}), 449 (-1.165×10^{-4})	480 (-1.1×10^{-4})

3.2 Spectroscopic spectra in different solvents

Table S2. Photophysical properties of (S/R)-**1a** and **1b** in solvents.

dyes	solvents	$\lambda_{\text{abs}}^{\text{max}}/\text{nm}$ ($\log \epsilon_{\text{max}}$) ^a	$\lambda_{\text{em}}^{\text{max}}$ (nm)	Stokes	ϕ^{b}	τ^{c} (ns)
(S)- 1a	hexane	414 (4.62), 438 (4.74)	446, 473	3000	0.58	2.26
	toluene	416 (4.46), 438 (4.54)	454, 481	3300	0.49	2.02
	DCM	416 (4.47), 434 (4.52)	460, 480	3200	0.15	0.74
	THF	416 (4.47), 432 (4.52)	460, 480	3200	0.07	0.37
	CH ₃ CN	412 (4.44), 428 (4.46)	488	3800	0.02	0.15
(R)- 1a	hexane	414 (4.62), 438 (4.74)	447, 474	3100	0.65	1.94
	toluene	416 (4.49), 438 (4.57)	455, 480	3200	0.53	2.07
	DCM	416 (4.50), 434 (4.54)	461, 482	3300	0.17	0.73
	THF	416 (4.50), 432 (4.54)	460, 479	3200	0.07	0.38
	CH ₃ CN	412 (4.45), 428 (4.47)	484	3400	0.02	0.14
(S)- 1b	hexane	422 (4.41), 446 (4.58)	456, 483	3000	0.73	2.86
	toluene	424 (4.54), 448 (4.68)	461, 488	3100	0.74	2.60
	DCM	422 (4.54), 444 (4.65)	460, 486	3100	0.60	2.34
	THF	422 (4.56), 444 (4.66)	459, 484	3100	0.45	1.89
	CH ₃ CN	418 (4.53), 438 (4.61)	459, 483	3200	0.08	0.47
(R)- 1b	hexane	422 (4.46), 446 (4.67)	456, 482	3000	0.70	2.87
	toluene	424 (4.51), 448 (4.65)	461, 487	3100	0.64	2.61
	DCM	422 (4.46), 444 (4.57)	460, 486	3100	0.51	2.36
	THF	422 (4.48), 444 (4.59)	460, 485	3100	0.38	1.91
	CH ₃ CN	418 (4.48), 438 (4.56)	459, 483	3200	0.08	0.48

^aMolar absorption coefficients are in the maximum of the highest peak. ^bAbsolute Φ_{FS} for chiral BOPPYs **1a** and **1b** were measured using integrating sphere in different solvents (excited at 410 nm for **1a** and **1b**).

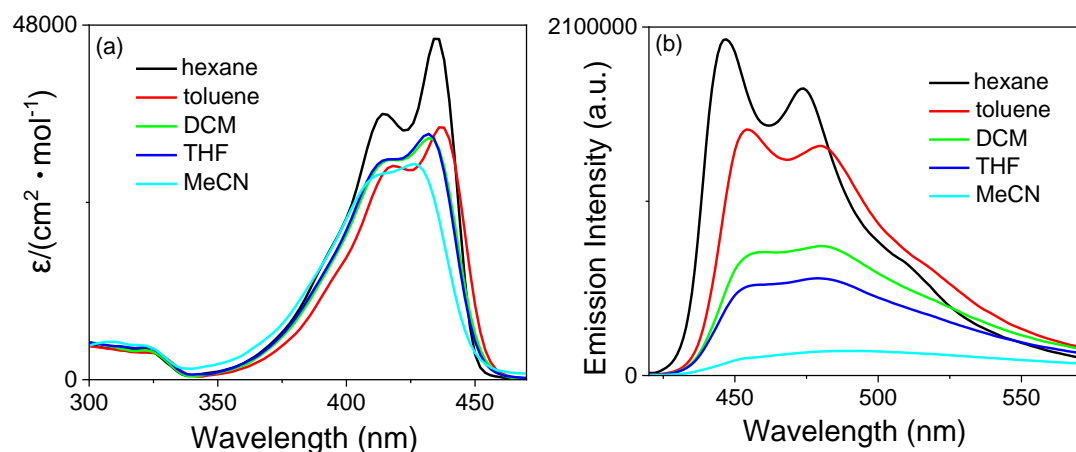


Figure S2. Absorption (a) and fluorescence (b) spectra of (S)-1a (10 μM) in hexane, toluene, dichloromethane, tetrahydrofuran and acetonitrile, excited at 410 nm.

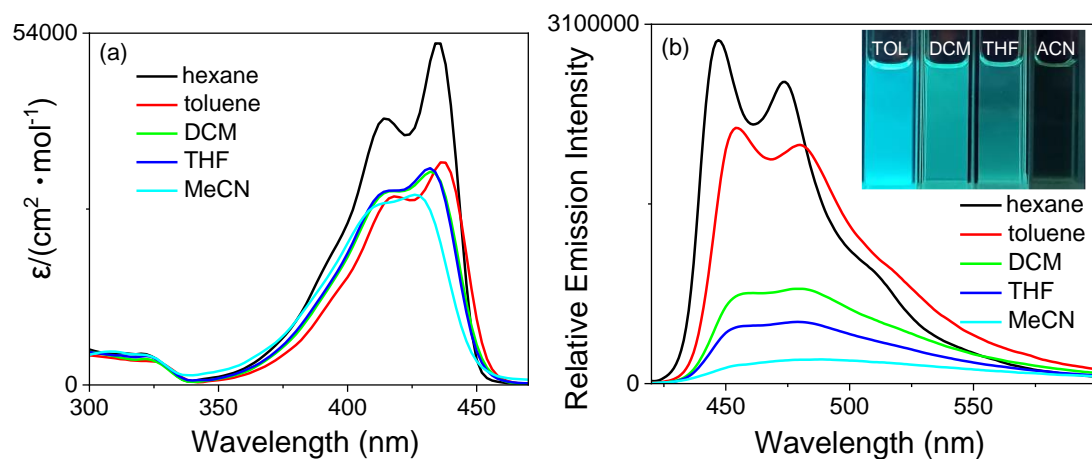


Figure S3. Absorption (a) and fluorescence (b) spectra of (R)-1a (10 μM) in hexane, toluene, dichloromethane, tetrahydrofuran and acetonitrile, excited at 410 nm.

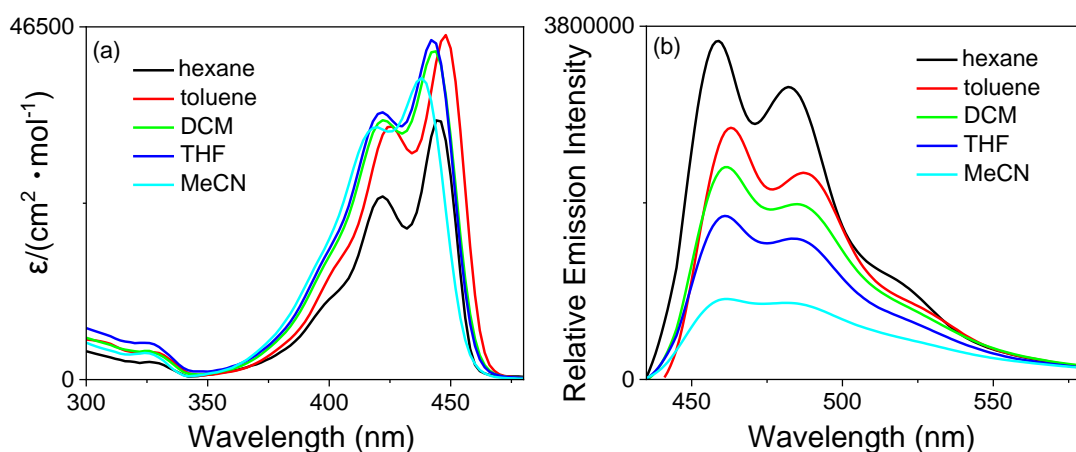


Figure S4. Absorption (a) and fluorescence (b) spectra of (S)-1b (10 μM) in hexane, toluene, dichloromethane, tetrahydrofuran and acetonitrile, excited at 415 nm.

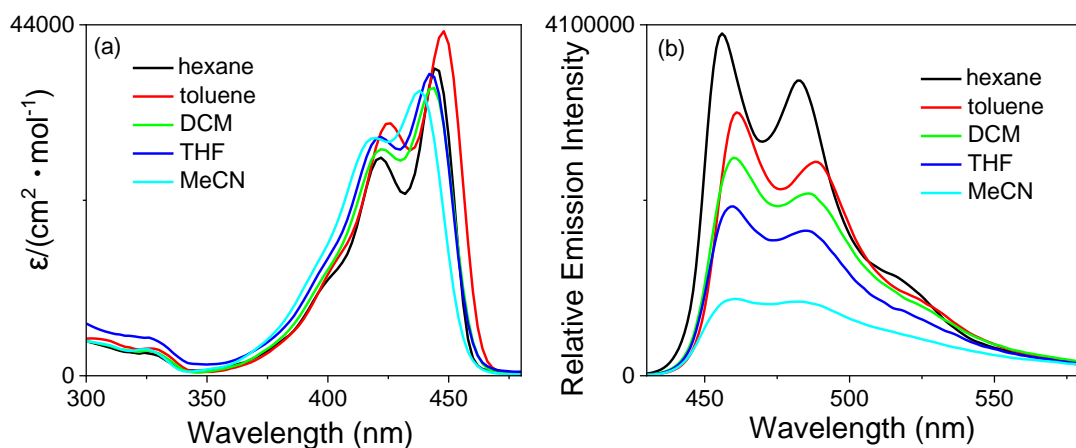


Figure S5. Absorption (a) and fluorescence (b) spectra of (R)-1b (10 μM) in hexane, toluene, dichloromethane, tetrahydrofuran and acetonitrile, excited at 415 nm.

3.2 Thermostability of chiral BOPPY 1a

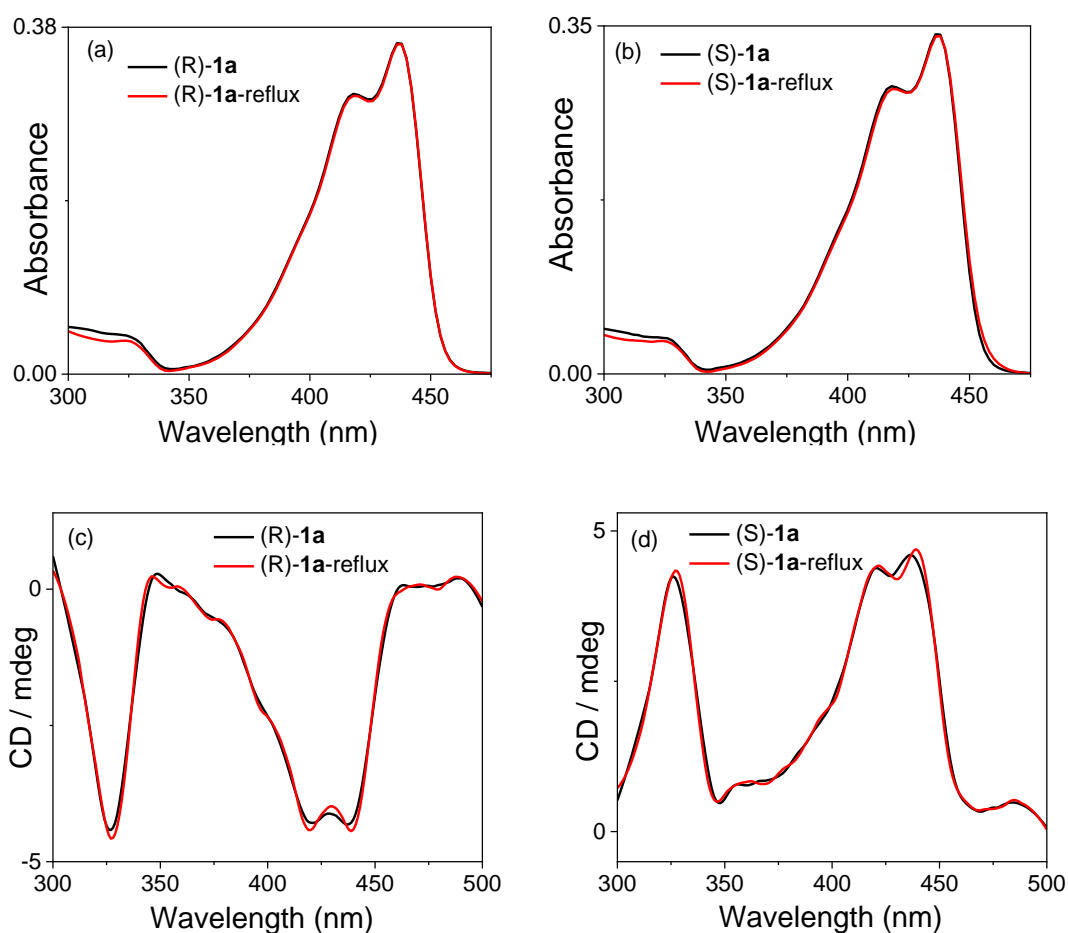


Figure S6. Absorption spectra (a, b) and the corresponding CD spectra (c, d) of (R/S)-1a (10 μM) before and after refluxing in toluene solution for 12 h.

3.3 Spectroscopic spectra of chiral BOPPYs (R/S)-1a and 1b in solid states

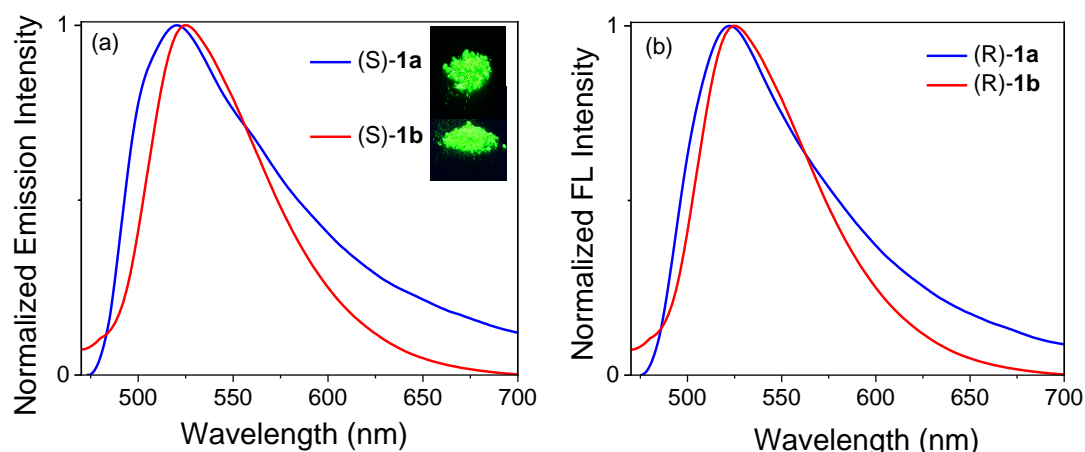


Figure S7. Emission spectra of (S/R)-**1a** and **1b** in the solid state, excited at 450 nm.

Table S3. Photophysical properties of (R/S)-**1a** and **1b** in solid states.

dyes	λ_{em}^{max} (nm) ^a	ϕ^b	τ^c (ns)
(R)- 1a	522	0.29	1.56
(S)- 1a	520	0.15	0.88
(R)- 1b	526	0.27	2.35
(S)- 1b	526	0.18	1.28

^aMolar absorption coefficients are in the maximum of the highest peak. ^bAbsolute fluorescence quantum yields for chiral BOPPYs **1a** and **1b** were measured using integrating sphere in different solvents (excited at 450 nm for **1a** and **1b**). All ϕ_f values are corrected for changes in refractive indexes of different solvents.

3.4 Viscosity-induced emission enhancement

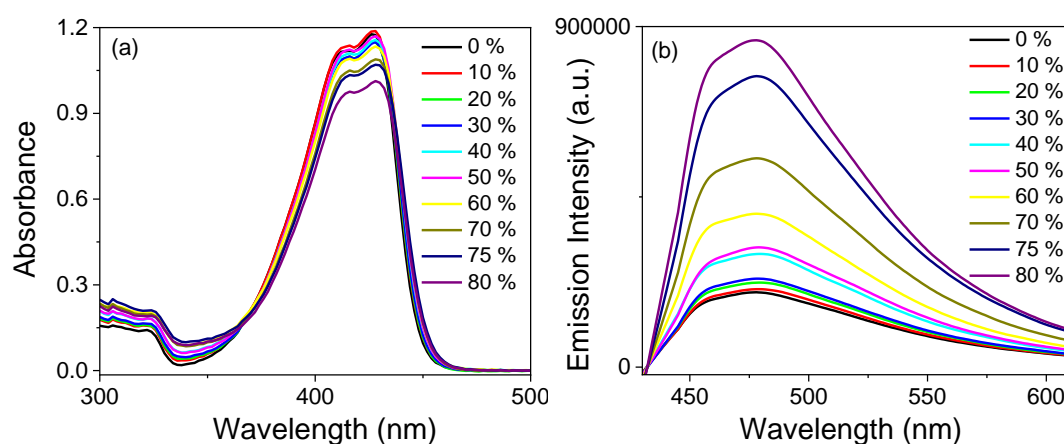


Figure S8. The absorbance (a) and emission spectra (b) of (S)-**1a** (30 μ M) in methanol-glycerol mixture with different viscosity, excited at 410 nm.

Table S4. Photophysical properties of (S)-**1a** in methanol-glycerol mixture.

solvents	λ_{abs}^{max}/nm ($\log \epsilon_{max}$)	λ_{em}^{max} (nm)	ϕ	τ (ns)
methanol	416 (4.57), 426 (4.59)	477	0.02	0.08
80% glycerol	415 (4.51), 428 (4.53)	479	0.09	0.38

4. AIE properties

Table S5. AIE properties of (R/S)-**1a** and **1b** in di-water system.

dyes	$\lambda_{\text{abs}}^{\text{max}}/\text{nm}(\log \epsilon_{\text{max}})^{\text{a}}$	$\lambda_{\text{em}}^{\text{max}}(\text{nm})$	ϕ^{b}	$\tau^{\text{c}}(\text{ns})$
(S)- 1a	417 (4.41), 443 (4.47)	473, 515	0.12	2.12
(R)- 1a	415 (4.44), 441 (4.47)	473, 495	0.09	0.94
(S)- 1b	424 (4.47), 452 (4.51)	476, 501	0.07	1.01
(R)- 1b	424 (4.57), 451 (4.60)	477, 520	0.10	1.99

^aMolar absorption coefficients are in the maximum of the highest peak. ^bAbsolute Φ_{FS} for **1a** and **1b** were measured using integrating sphere in different solvents (excited at 410 nm for **1a** and **1b**).

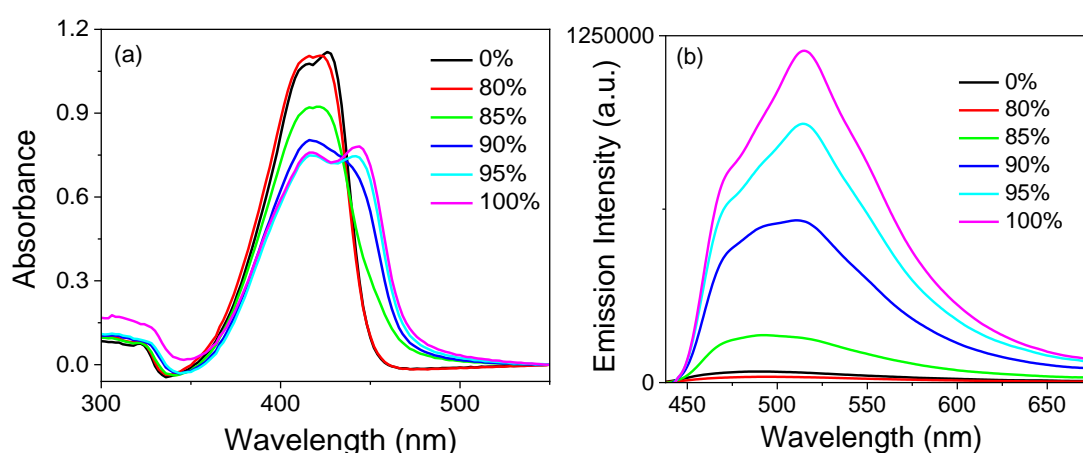


Figure S9. Absorption spectra (a) and relative FL intensity (b) of (S)-**1a** (30 μM) in acetonitrile/ di-water system with different di-water content ratios, excited at 410 nm.

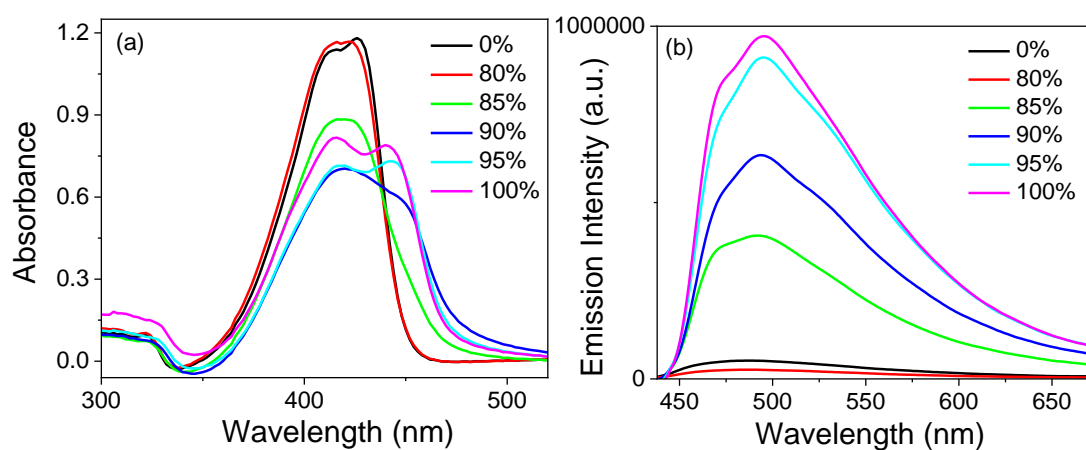


Figure S10. Absorption spectra (a) and relative FL intensity (b) of (R)-**1a** (30 μM) in acetonitrile/di-water system with different di-water content ratios, excited at 410 nm.

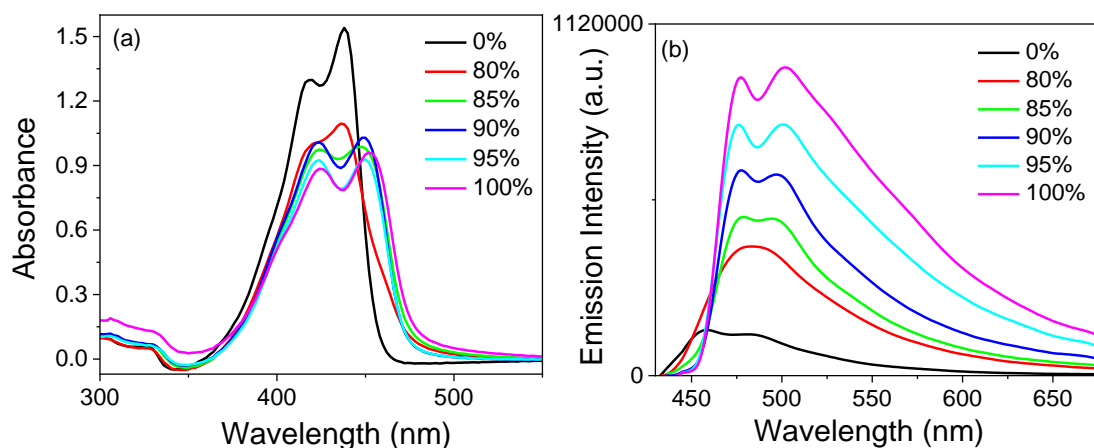


Figure S11. Absorption spectra (a) and relative FL intensity (b) of (S)-**1b** (30 μ M) in acetonitrile/di-water system with different di-water content ratios, excited at 410 nm.

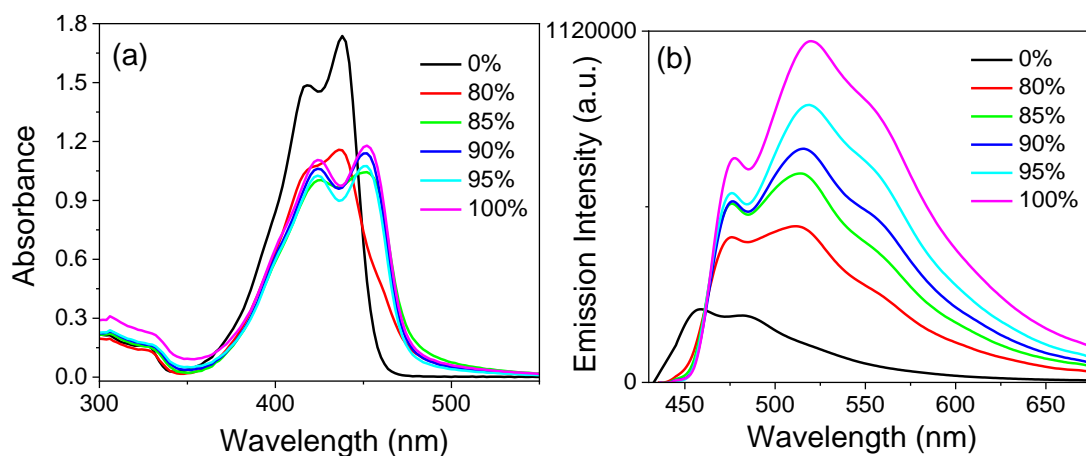


Figure S12. Absorption spectra (a) and relative FL Intensity (b) of (R)-**1b** (30 μ M) in acetonitrile and di-water system with different di-water content ratios, excited at 410 nm.

5. Chiroptical properties in micelles and aggregation state

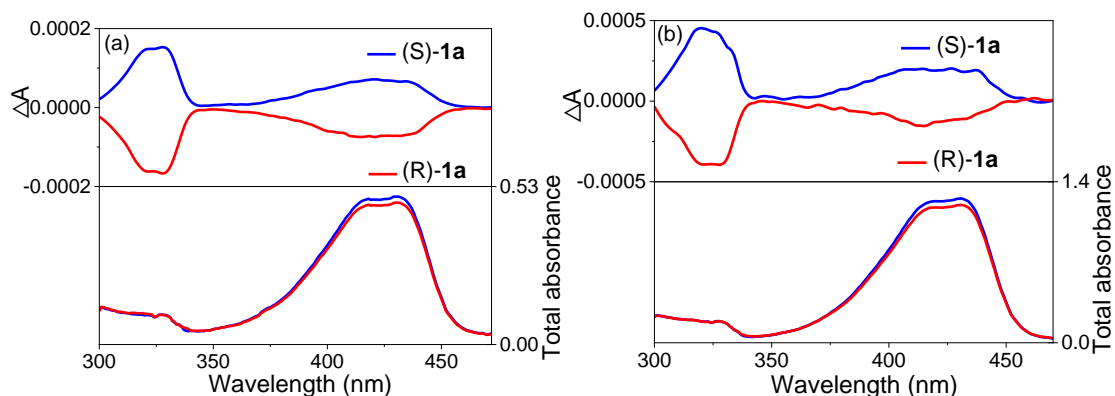


Figure S13. The CD and synchronous absorption spectra of BOPPY (S/R)-**1a** at 10 μ M (a) and 30 μ M (b).

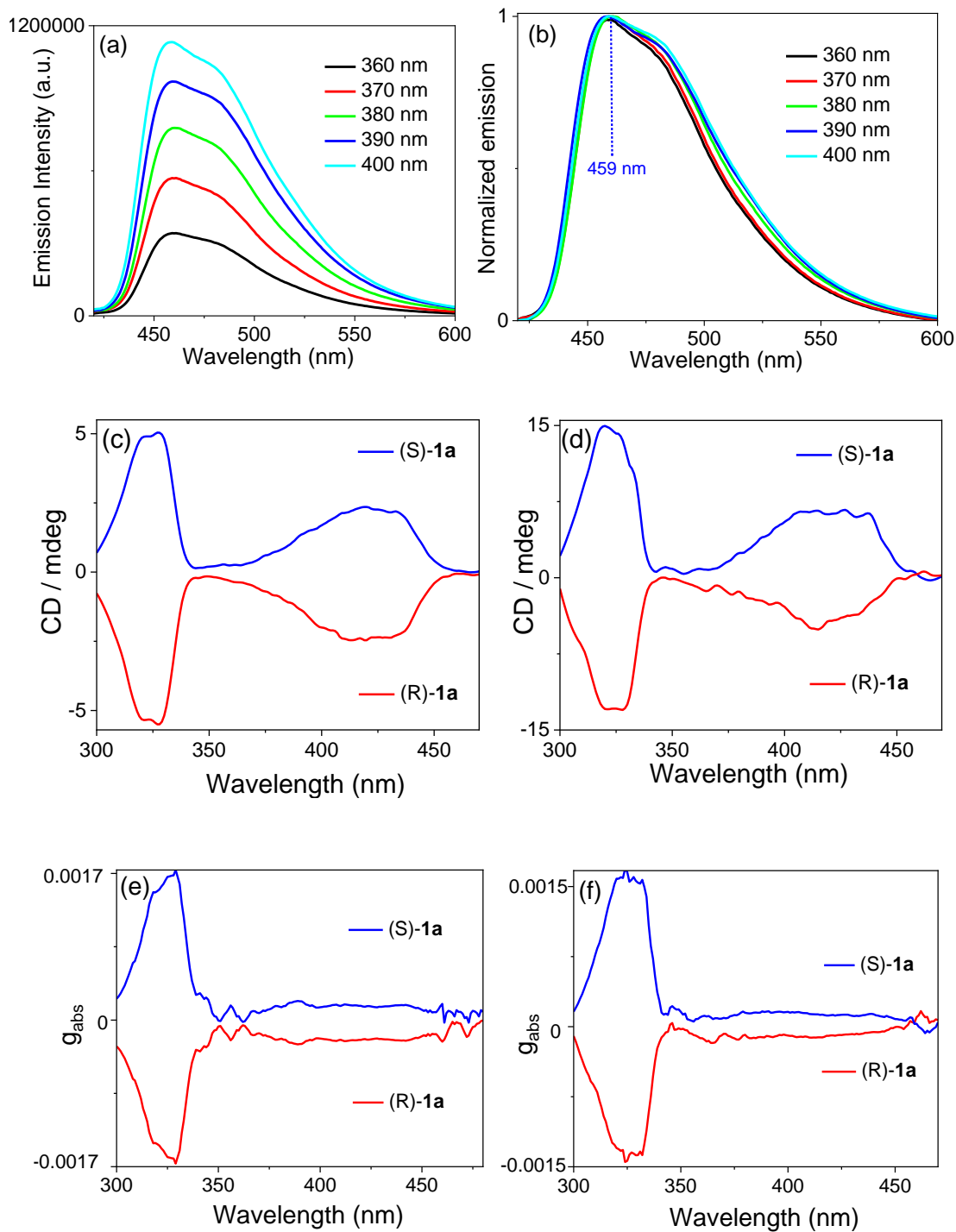


Figure S14. (a, b) The emission and normalized emission spectra of (S)-**1a** in the micelle states excited at 360, 370, 380, 390 and 400 nm. CD (c), synchronous g_{abs} (e) spectra of BOPPY (S/R)-**1a** (10 μ M) in SDS aqueous solution at the stable state. The CD (d), synchronous g_{abs} (f) spectra of BOPPY (S/R)-**1a** (30 μ M) in SDS aqueous solution at the initial state.

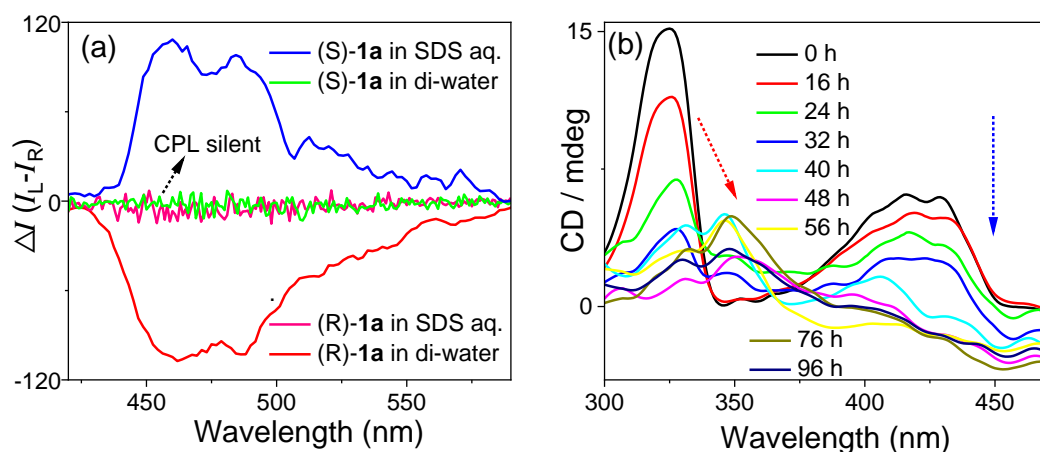


Figure S15. (a) The CPL spectra of BOPPY (S/R)-**1a** (30 μM) in di-water and SDS aqueous solution at the initial stage. (b) The CD spectra of (S)-**1a** (30 μM) in different times ranging from 0 to 96 h in SDS aqueous solution.

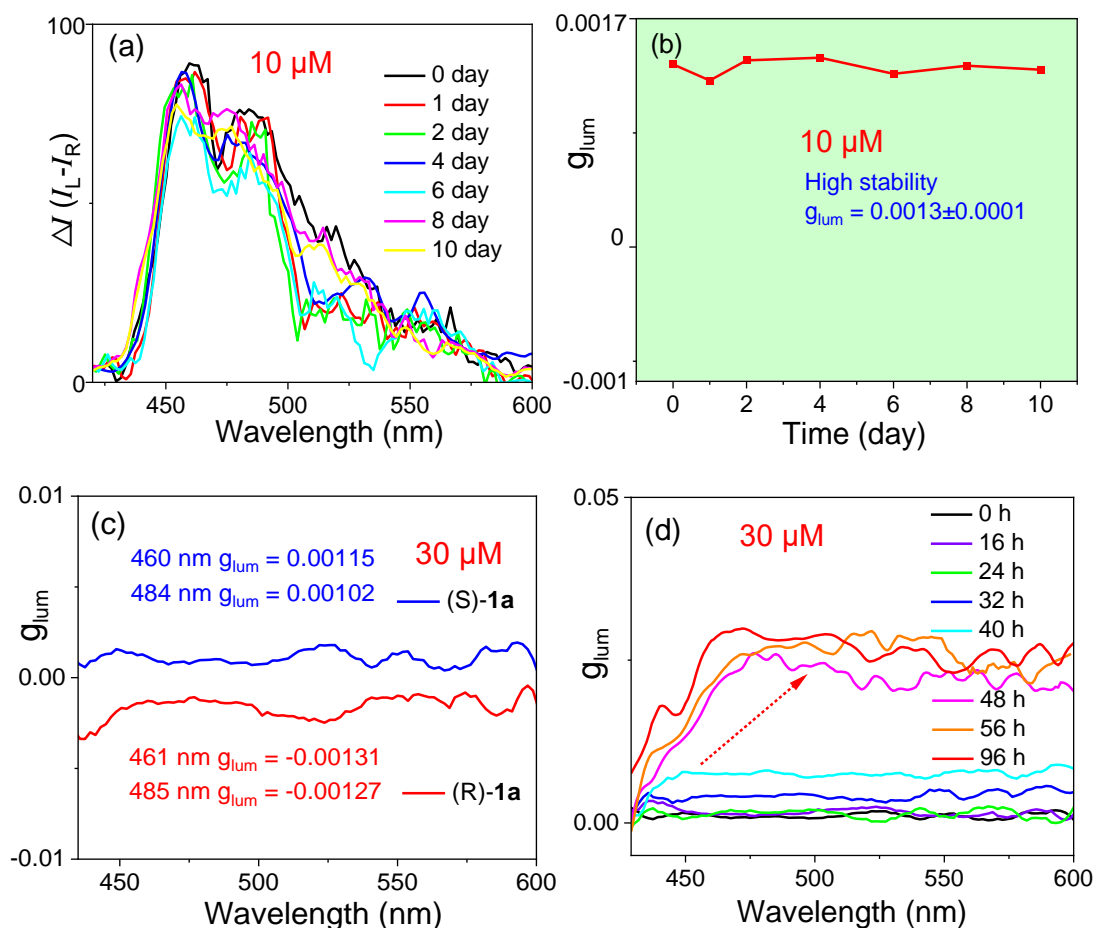


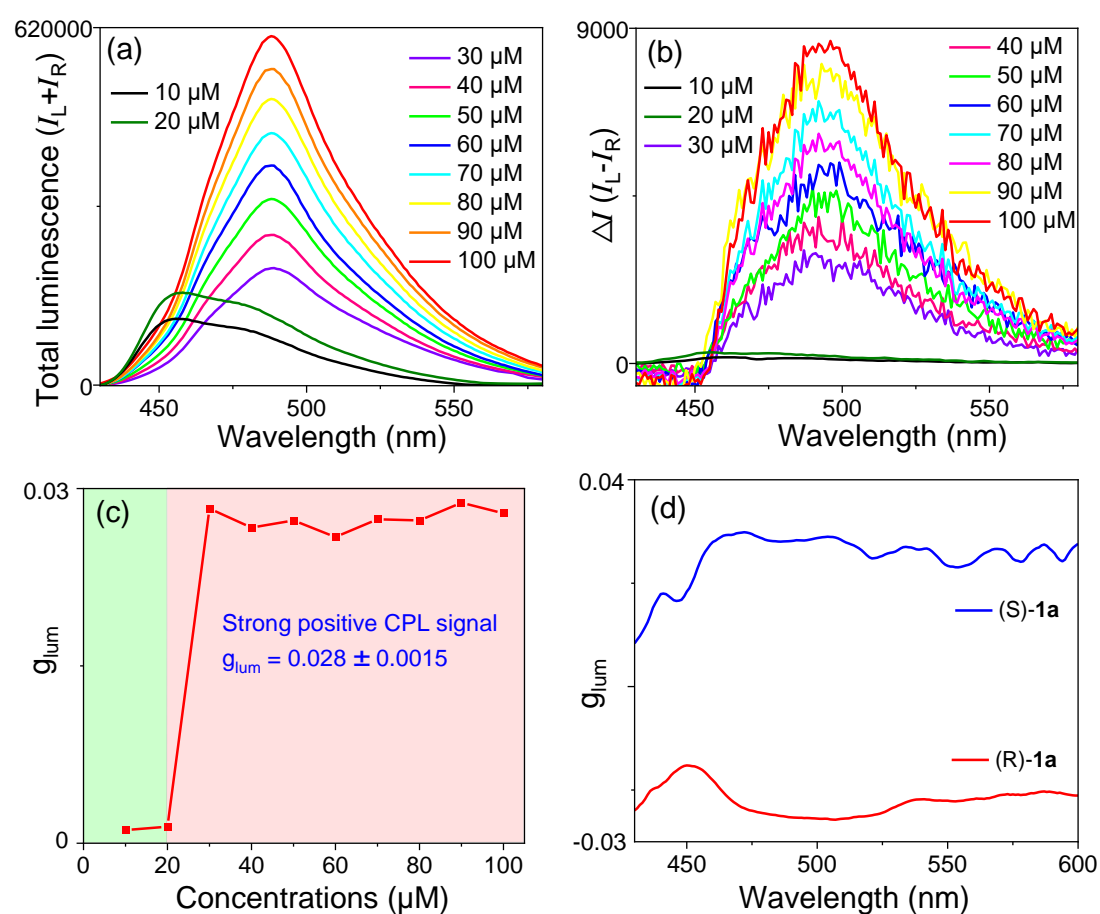
Figure S16. The stability study of CPL spectra of (S)-**1a** (10 μM) in SDS aqueous solution at different days and (b) the corresponding g_{lum} change plot. (c) The g_{lum} spectra of (S/R)-**1a** (30 μM) in SDS aqueous solution at the initial state. (d) The g_{lum} spectra of (S)-**1a** (30 μM) in different times ranging from 0 to 96 h in SDS aqueous solution.

Table S6. The g_{abs} of chiral BOPPY **1a** in SDS aqueous solution.

dyes	λ_{max}/nm (g_{abs} , 10 μM , stable state)	λ_{max}/nm (g_{abs} , 30 μM , initial state)
(S)- 1a	418 (1.70×10^{-4}), 433 (1.59×10^{-4})	414 (1.32×10^{-4}), 436 (1.25×10^{-4})
(R)- 1a	414 (-2.39×10^{-4}), 431 (-2.22×10^{-4})	415 (-1.70×10^{-4}), 431 (-1.59×10^{-4})

Table S7. The g_{lum} of chiral BOPPY **1a** in SDS aqueous solution in the steady state.

dyes	λ_{max}/nm (g_{lum} , 10 μM)	λ_{max}/nm (g_{lum} , 30 μM)
(S)- 1a	459 (0.00136), 480 (0.00114)	495 (0.0283)
(R)- 1a	459 (-0.00137), 480 (-0.00122)	495 (-0.0251)

**Figure S17.** The CPL spectra (a), emission spectra (b) and g_{lum} line chart (c) of BOPPY (S)-**1a** in different concentrations ranging from 10 to 100 μM in SDS aqueous solution test after 5 days of stability. The g_{lum} (d) spectra of final (S/R)-**1a** assembly under the same conditions in SDS aqueous solution.

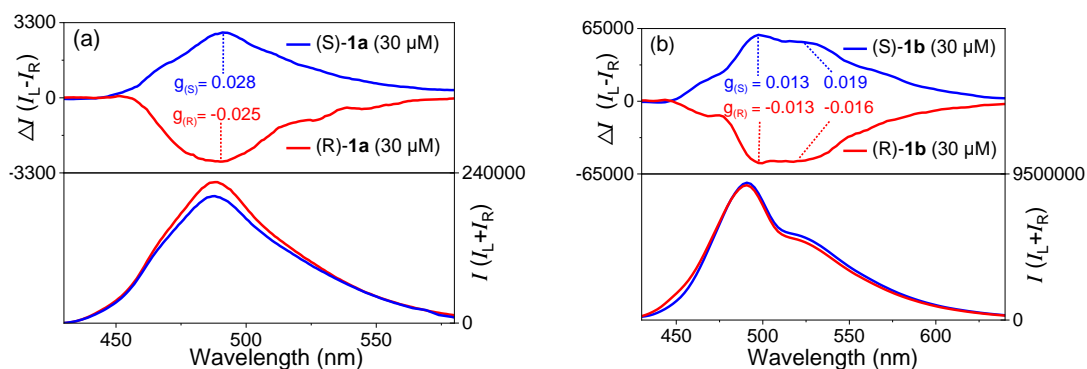


Figure S18 The CPL and synchronous total luminescence spectra of BOPPY (S/R)-1a (a) and 1b (b) at 30 μM at the steady state in SDS aqueous solution.

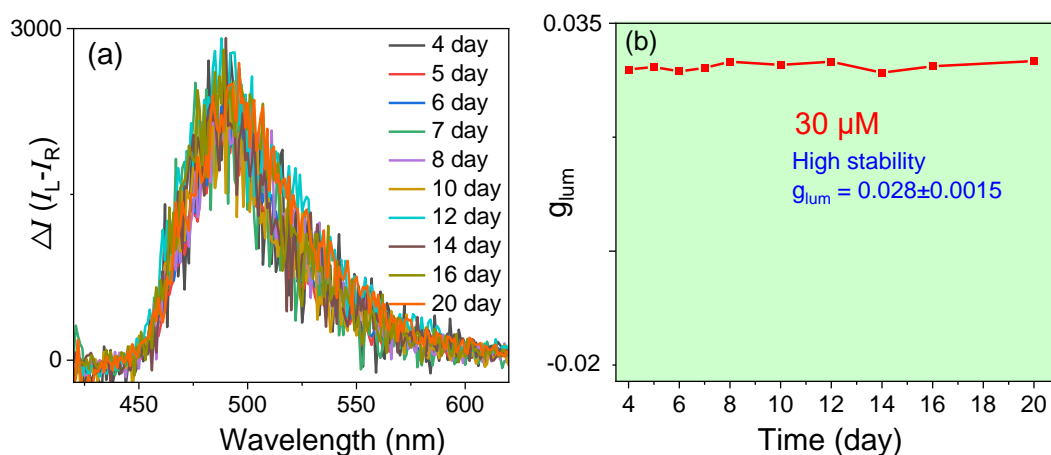


Figure S19. The stability study of CPL spectra (a) and g_{lum} change plot (b) of (S)-1a (30 μM) in SDS aqueous solution at different days.

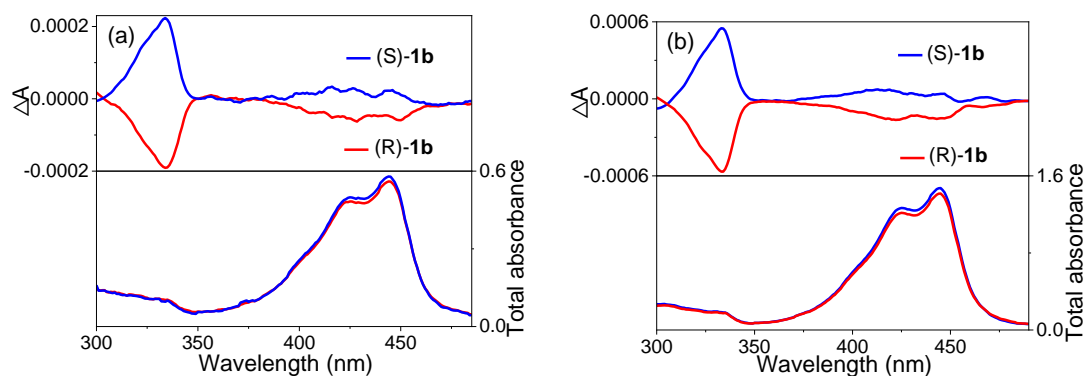


Figure S20. The CD and synchronous absorption spectra of BOPPY (S/R)-1b at 10 μM (a) and 30 μM (b).

Table S8. Stability study on every other day on (S)-1a (30 μM) in SDS aqueous solution.

day	4	5	6	7	8
g_{lum}	0.0279	0.0283	0.0276	0.0281	0.0291
day	10	12	14	16	20
g_{lum}	0.0286	0.0291	0.0274	0.0284	0.0292

Table S9. The g_{abs} of chiral BOPPYs **1b** in SDS aqueous solution.

dyes	λ_{max}/nm (g_{abs} , 10 μM , stable state)	λ_{max}/nm (g_{abs} , 30 μM , initial state)
(S)- 1b	416 (1.02×10^{-4}), 446 (8.2×10^{-5})	425 (1.08×10^{-4}), 445 (1.01×10^{-4})
(R)- 1b	421 (-1.33×10^{-4}), 446 (-1.04×10^{-4})	423 (-1.18×10^{-4}), 441 (-8.8×10^{-5})

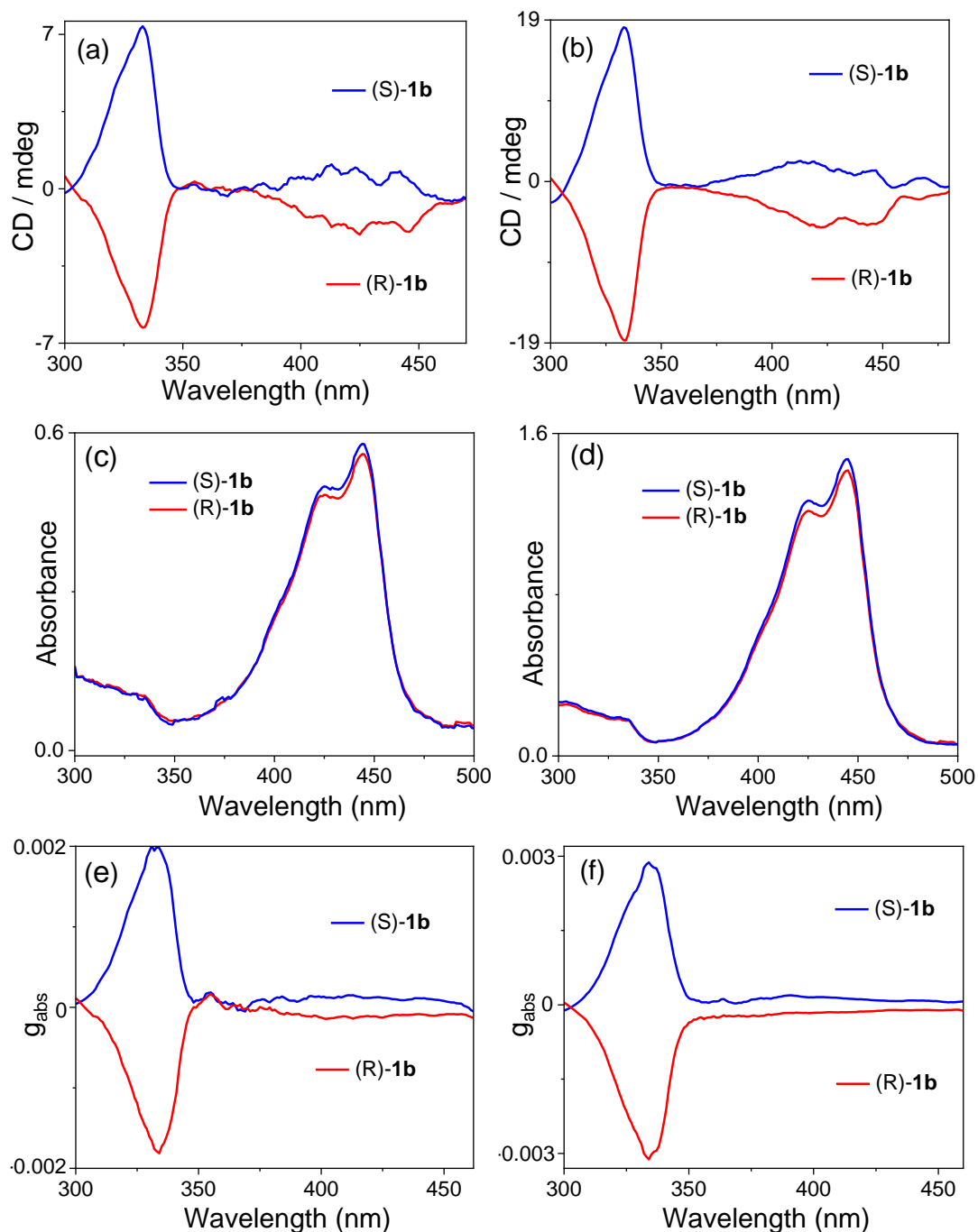
**Figure S21.** The CD (a), synchronous absorption (c) and g_{abs} (e) spectra of BOPPY (S/R)-**1b** (10 μM) in SDS aqueous solution at the stable state. The CD (b), synchronous absorption (d) and g_{abs} (f) spectra of BOPPY (S/R)-**1b** (30 μM) in SDS aqueous solution at the initial state.

Table S10. The g_{lum} of chiral BOPPYs **1b** in SDS aqueous solution at the stable state.

dyes	λ_{max}/nm (g_{lum} , 10 μM)	λ_{max}/nm (g_{lum} , 30 μM)
(S)- 1b	457 (0.00142), 480 (0.00140)	498 (0.01322), 528 (0.01862)
(R)- 1b	460 (-0.00169), 480 (-0.00151)	498 (-0.01274), 526 (-0.0157)

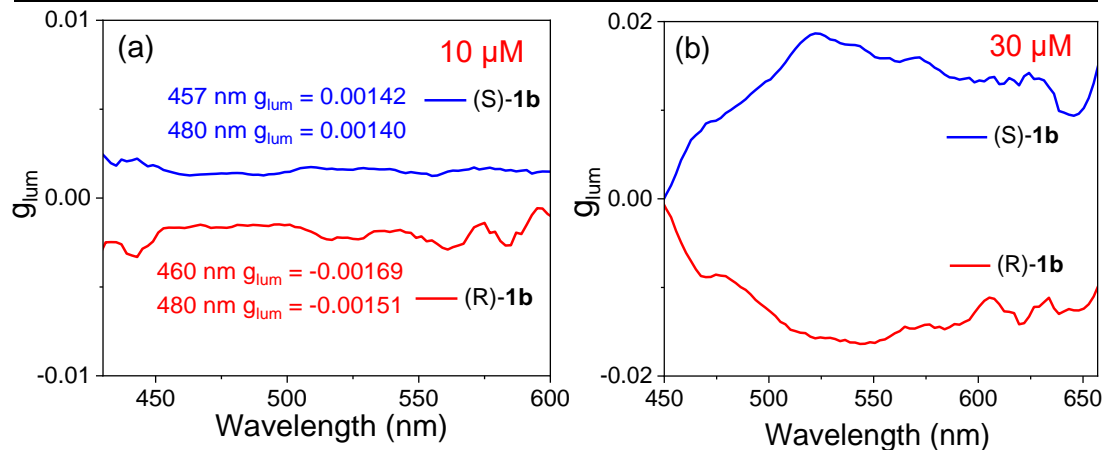


Figure S22. The g_{lum} spectra of (S/R)-**1b** in 10 μM (a) and 30 μM (b) at the steady state in SDS aqueous solution.

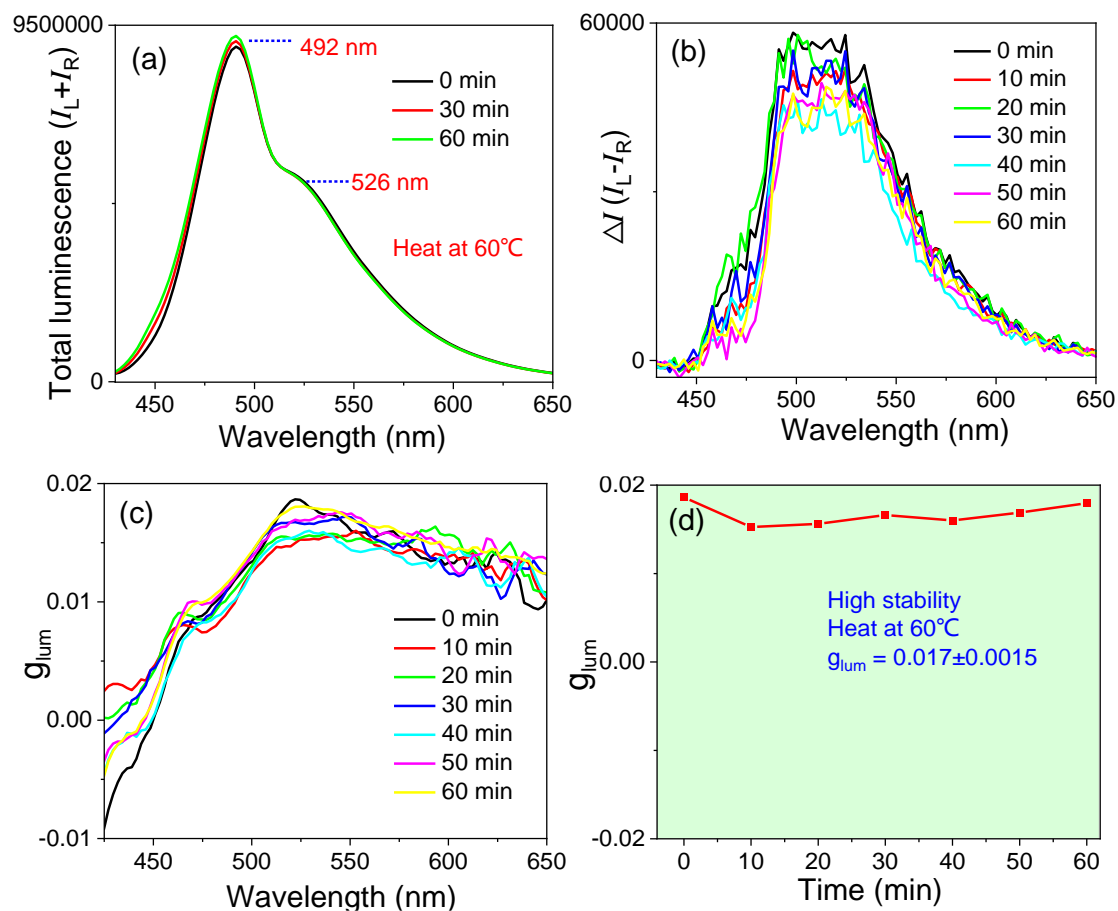


Figure S23. The total luminescence (a), CPL (b), g_{lum} (c) spectra and g_{lum} (monitoring at 528 nm) change plot (d) of (S)-**1b** (30 μM) in SDS aqueous solution in the final steady state heating at 60 °C ten minutes ultrasonic 30 s to study stability.

6. Helical single crystal packing diagrams

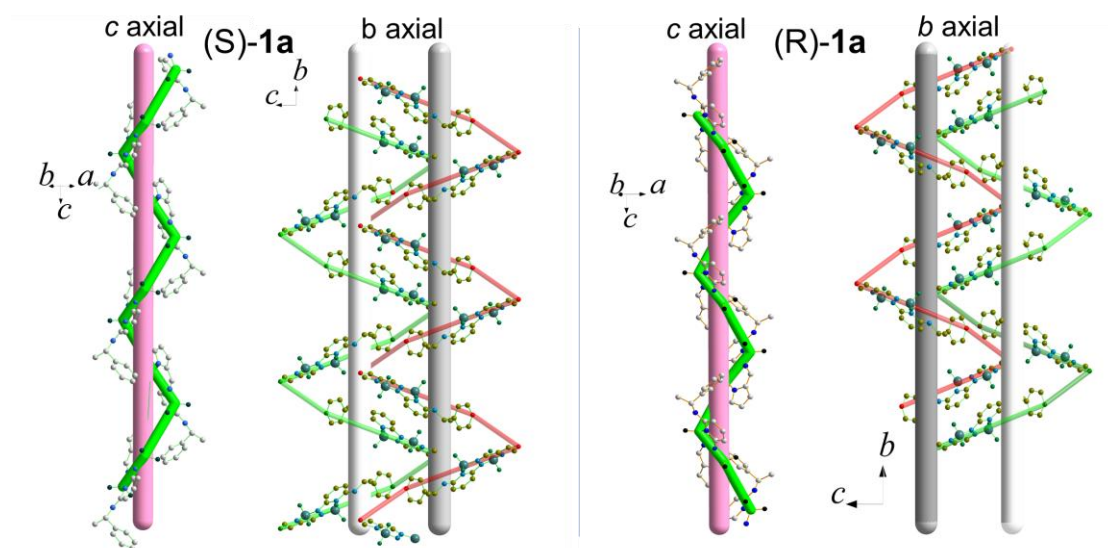


Figure S24. Single-crystal packing plots at the “c” axial direction revealing the formation of helical aggregates P-helix from (S)-**1a** and M-helix from (R)-**1a**. Single-crystal packing plots at the “b” axial direction revealing the formation of double helical aggregates from (S)-**1a** and (R)-**1a**.

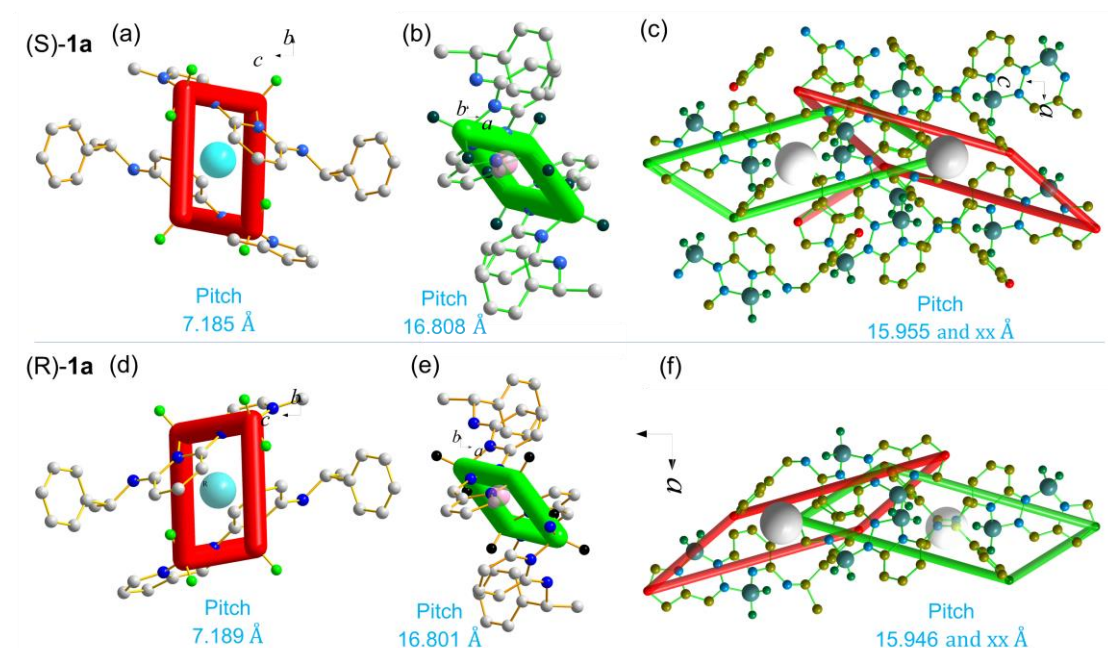


Figure S25. (a-c) Single-crystal packing helical distance at the “a”, “c” and “b” axial directions from the corresponding helix from (S)-**1a**, respectively. (d-f) Single-crystal packing helical distance at the “a”, “c” and “b” axial directions from the corresponding helix from (R)-**1a**, respectively.

7. SEM and TEM

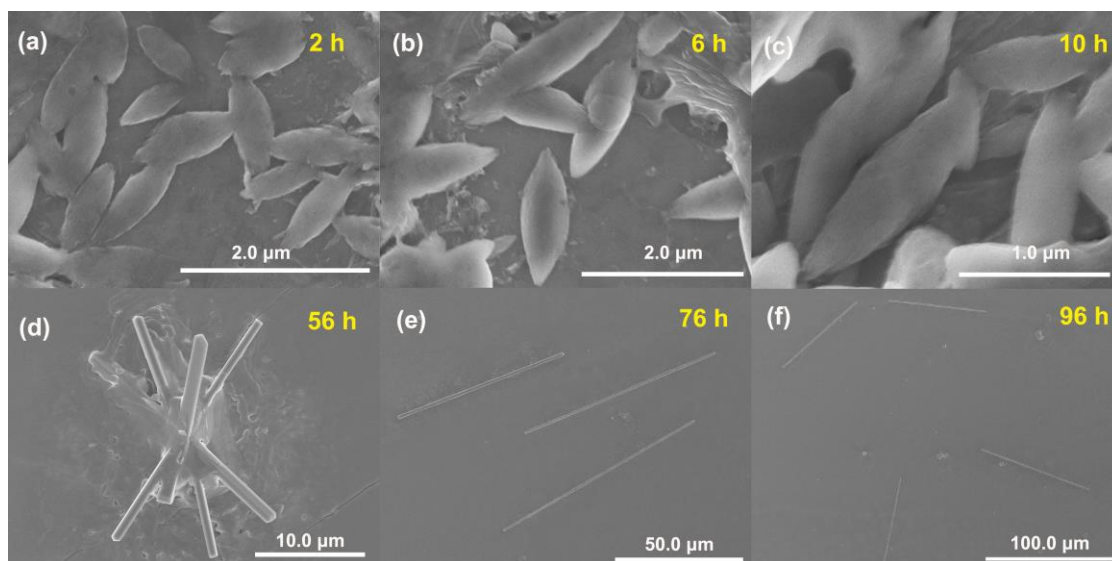


Figure S26. Time-dependent SEM images visualizing the self-assembly processes of (S)-1a (30 μM) in SDS aqueous solution under different time.

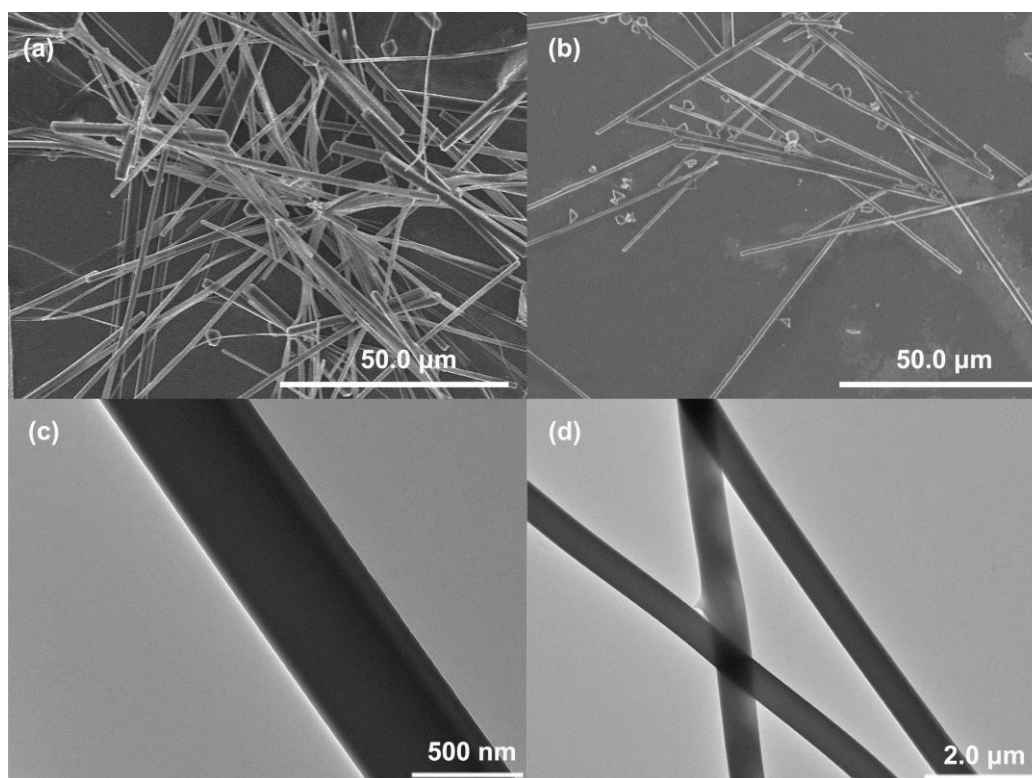
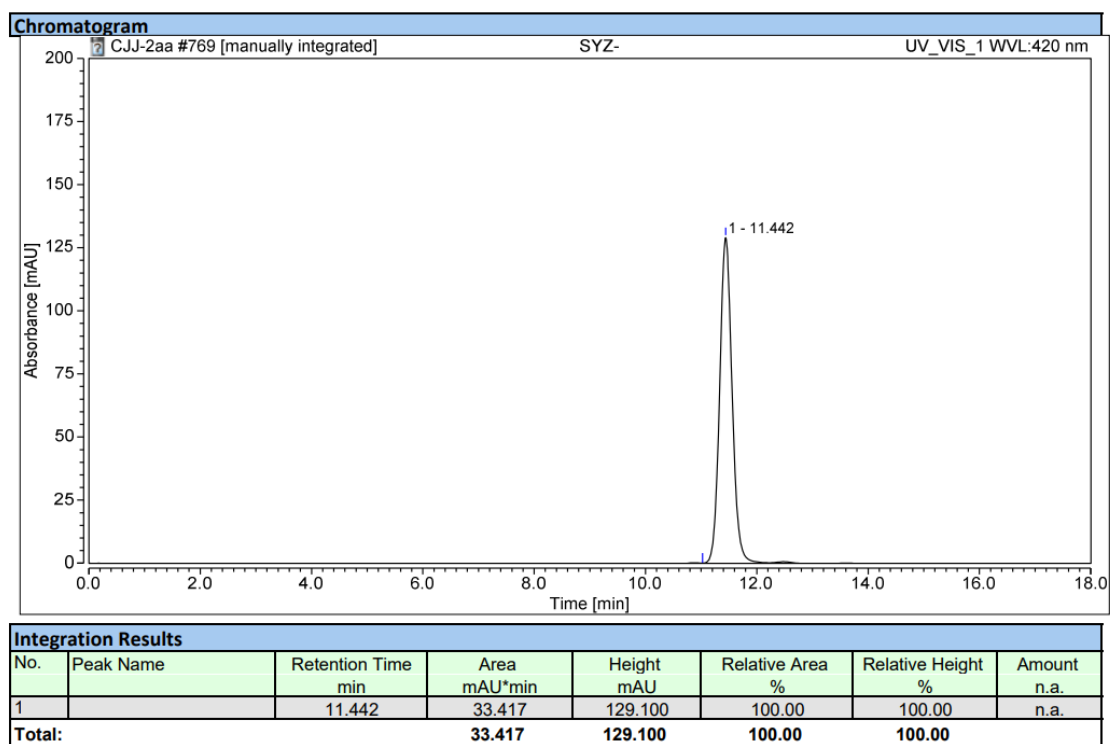


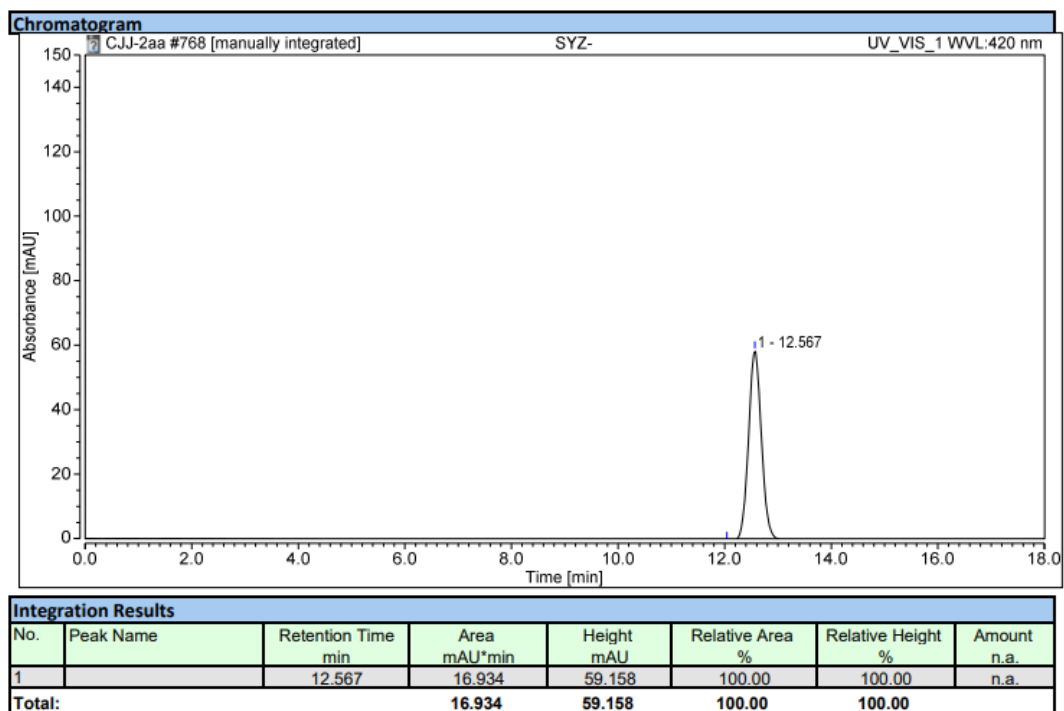
Figure S27. The SEM images of (S)-1a (a) and (R)-1a (b) in SDS aqueous solution after 96 h. The TEM images of (S)-1a (c) and (R)-1a (d) in SDS water after 96 h.

8. HPLC measurements for new Chiral BOPPY Dyes

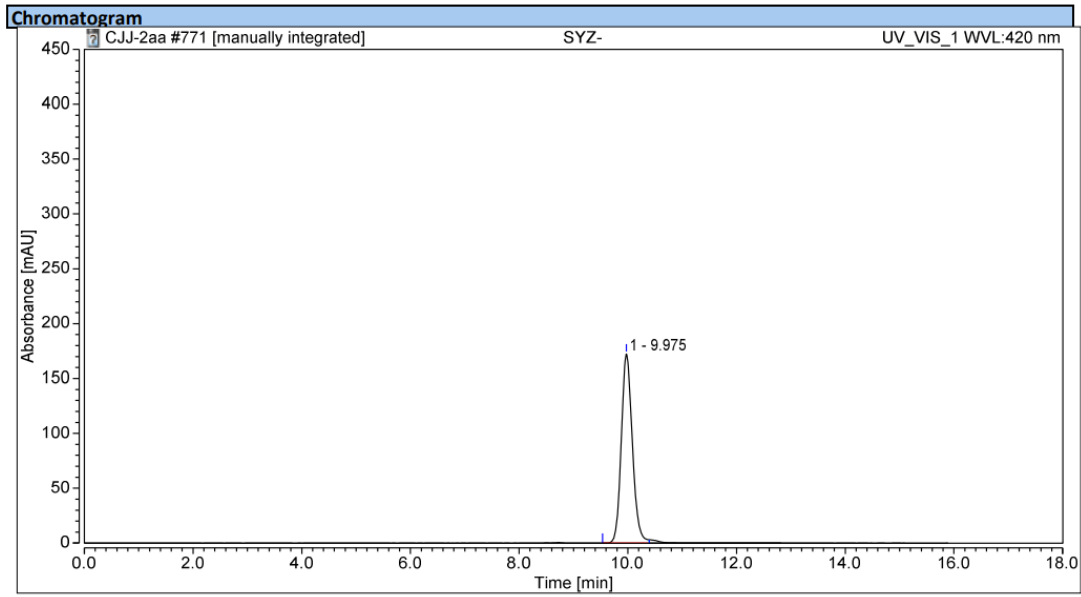
HPLC analysis: (S)-1a



HPLC analysis: (R)-1a

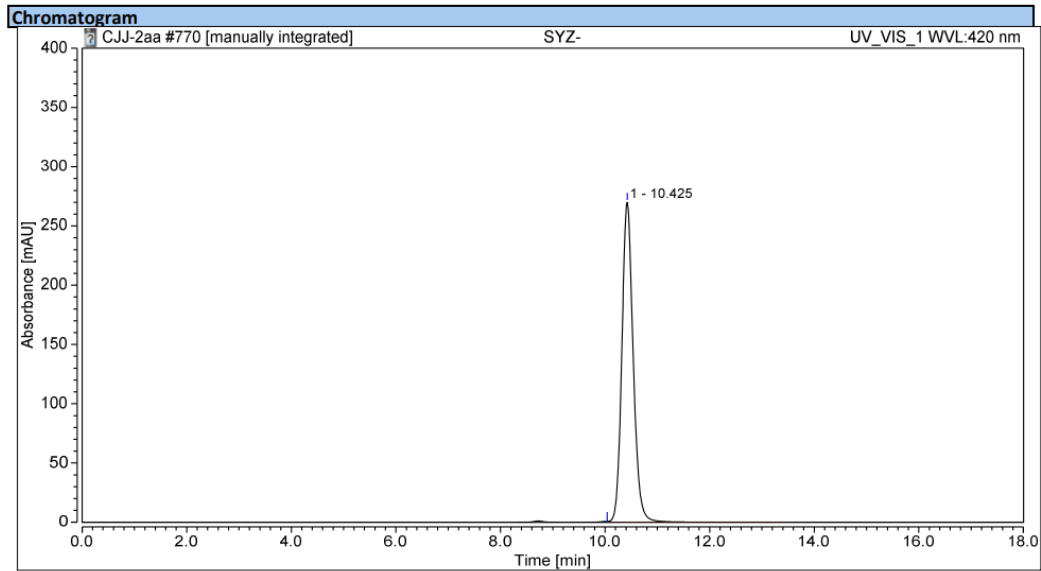


HPLC analysis: (S)-1b



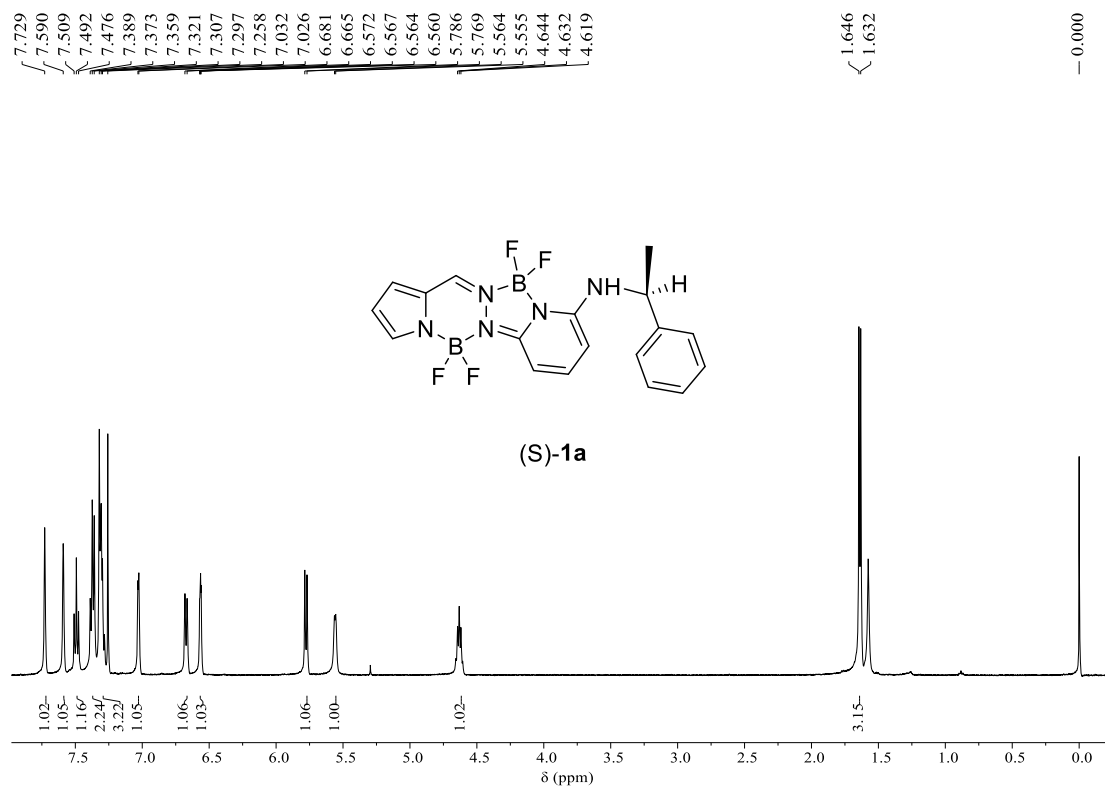
Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		9.975	40.590	172.351	100.00	100.00	n.a.
Total:			40.590	172.351	100.00	100.00	

HPLC analysis: (R)-1b

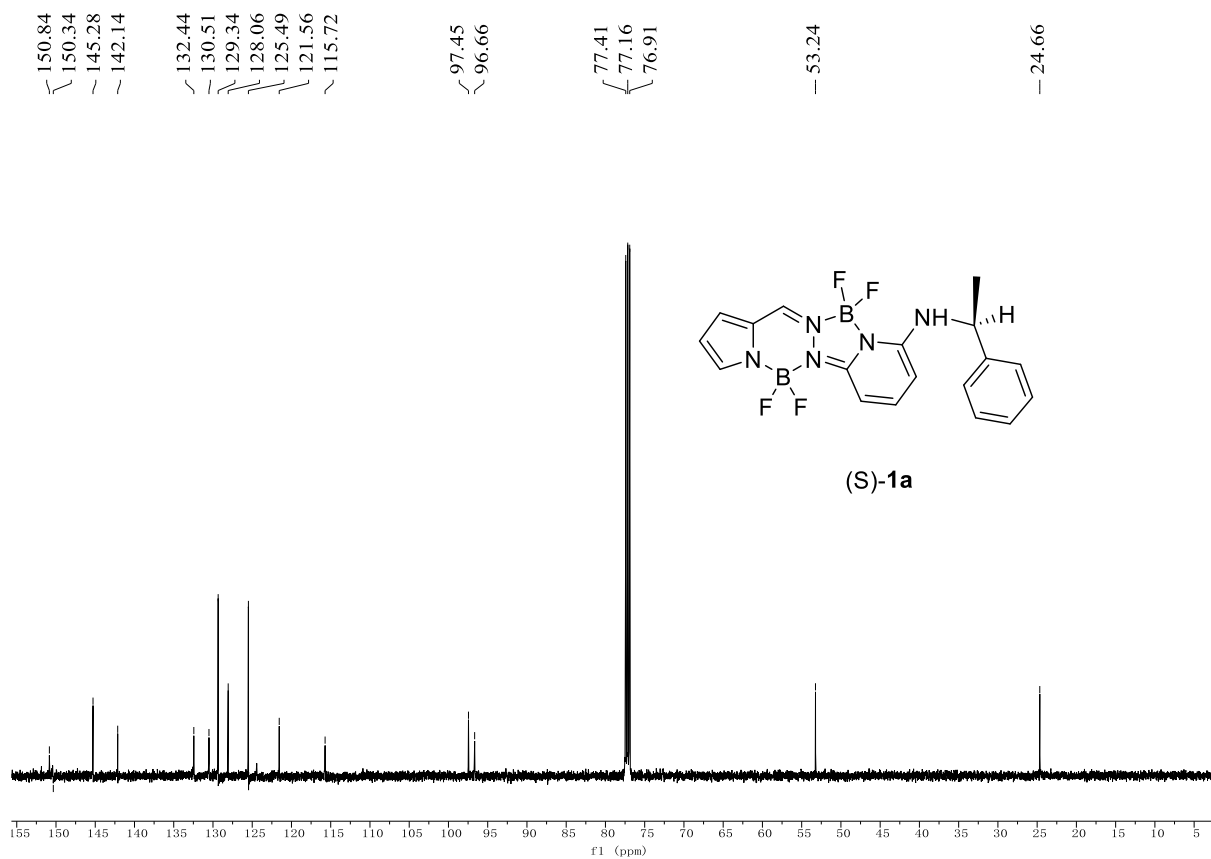


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		10.425	68.017	269.948	100.00	100.00	n.a.
Total:			68.017	269.948	100.00	100.00	

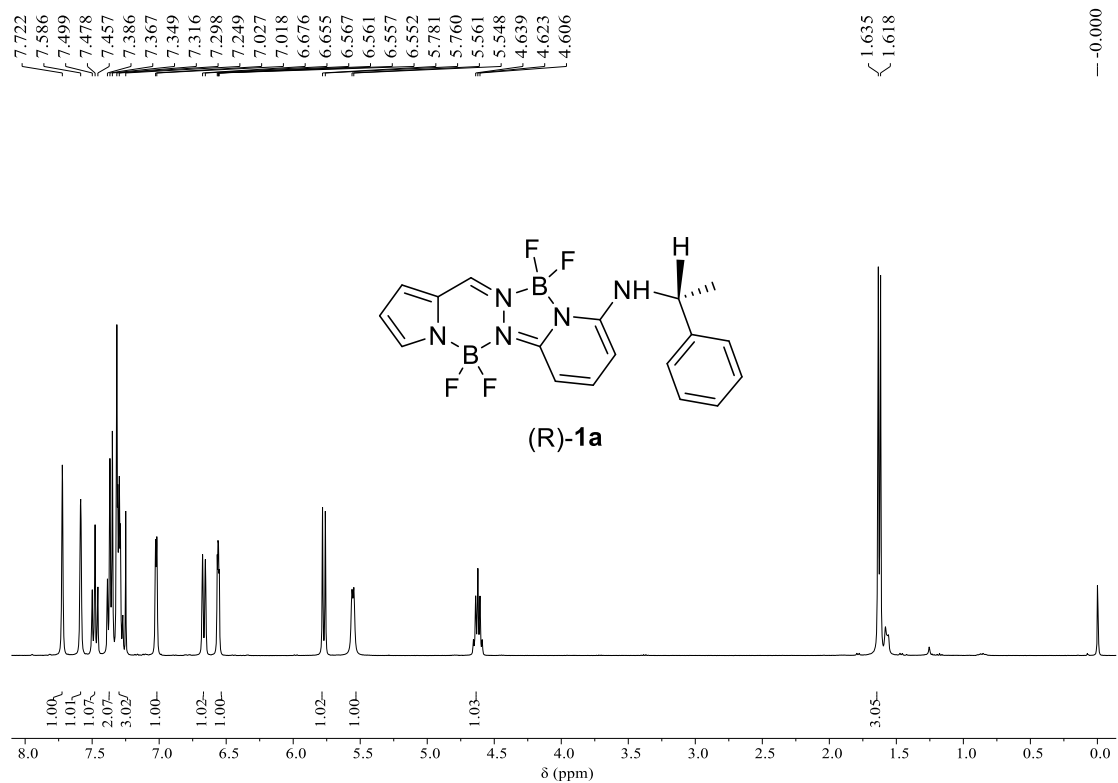
9. ¹H, ¹³C, ¹¹B and ¹⁹F NMR spectra for the new chiral BOPPYs



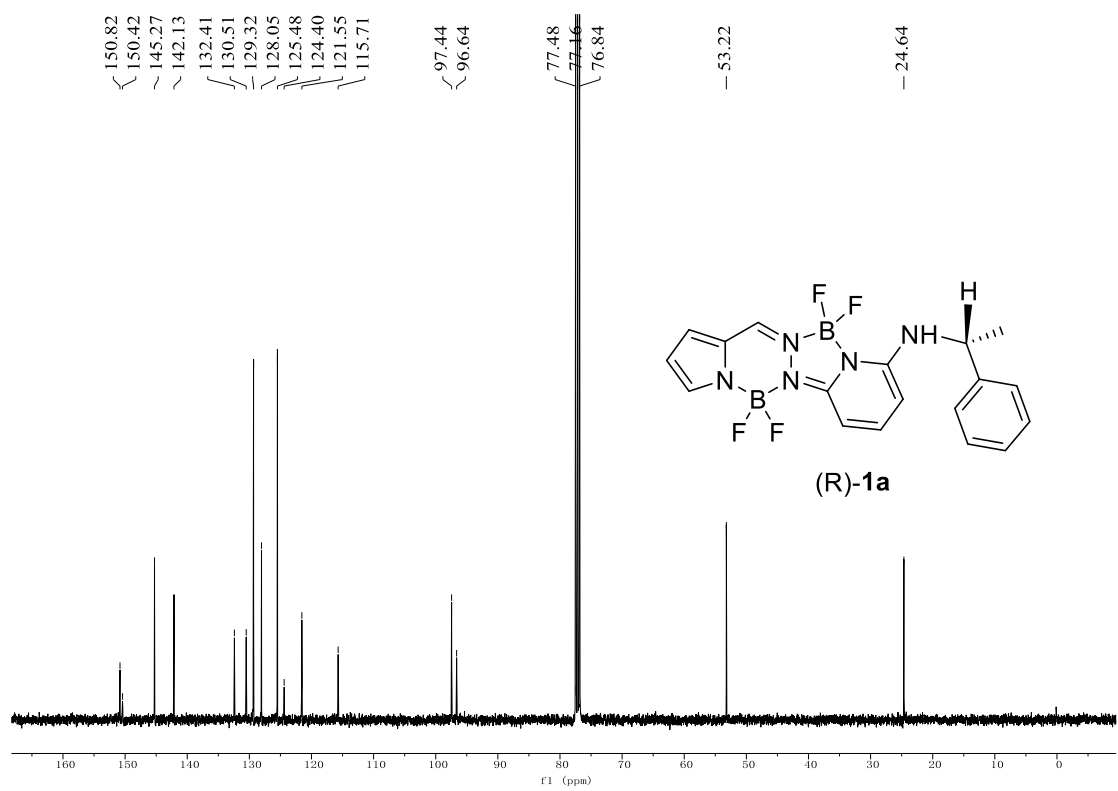
¹H NMR spectrum of (S)-1a in CDCl₃



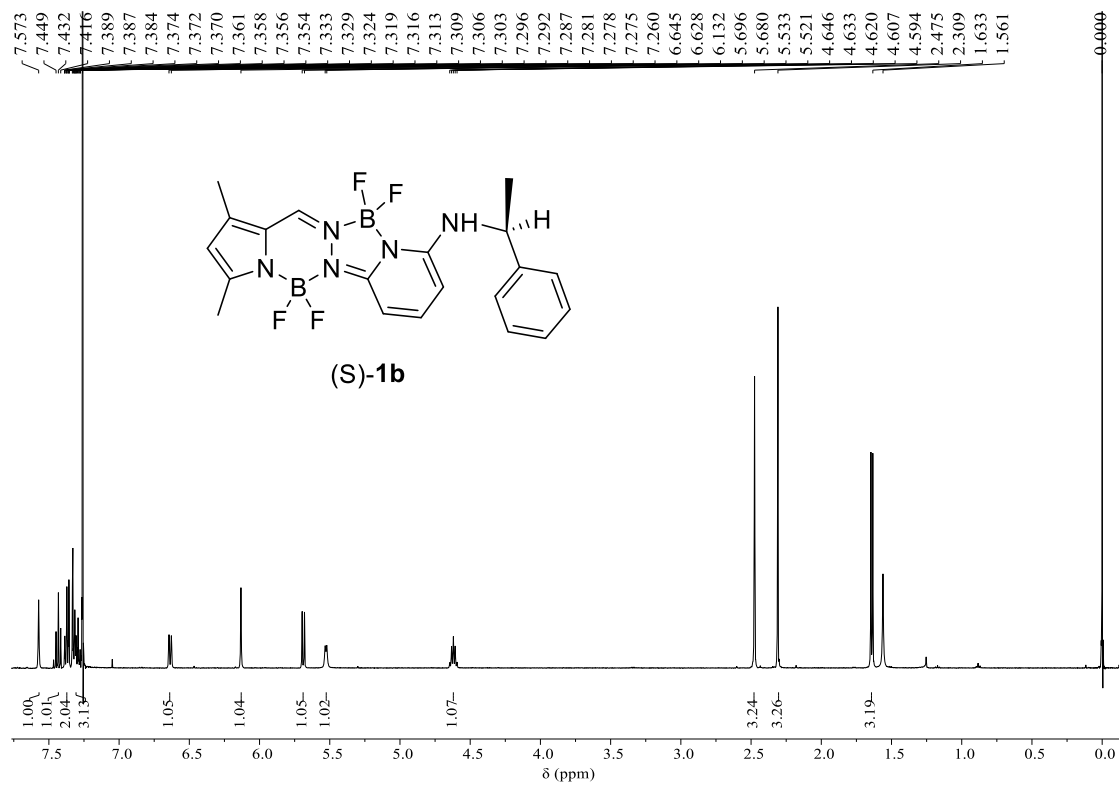
¹³C NMR spectrum of (S)-1a in CDCl₃



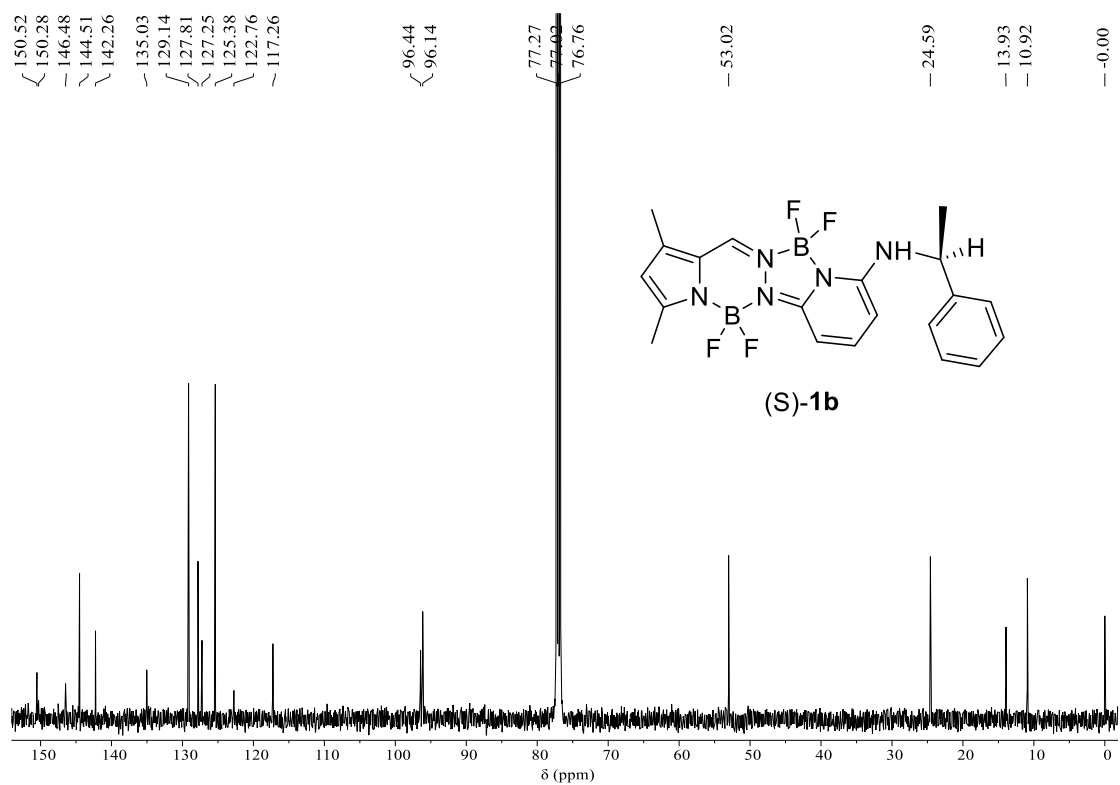
¹H NMR spectrum of (R)-1a in CDCl₃



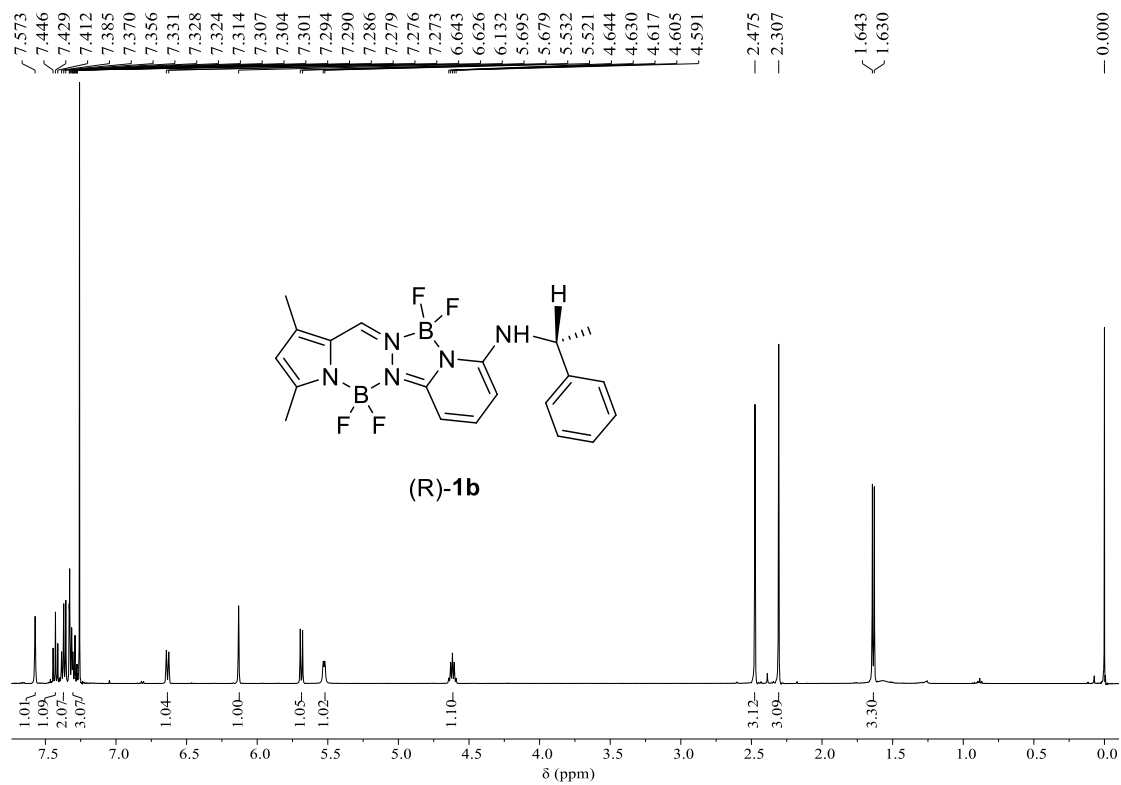
¹³C NMR spectrum of (R)-1a in CDCl₃



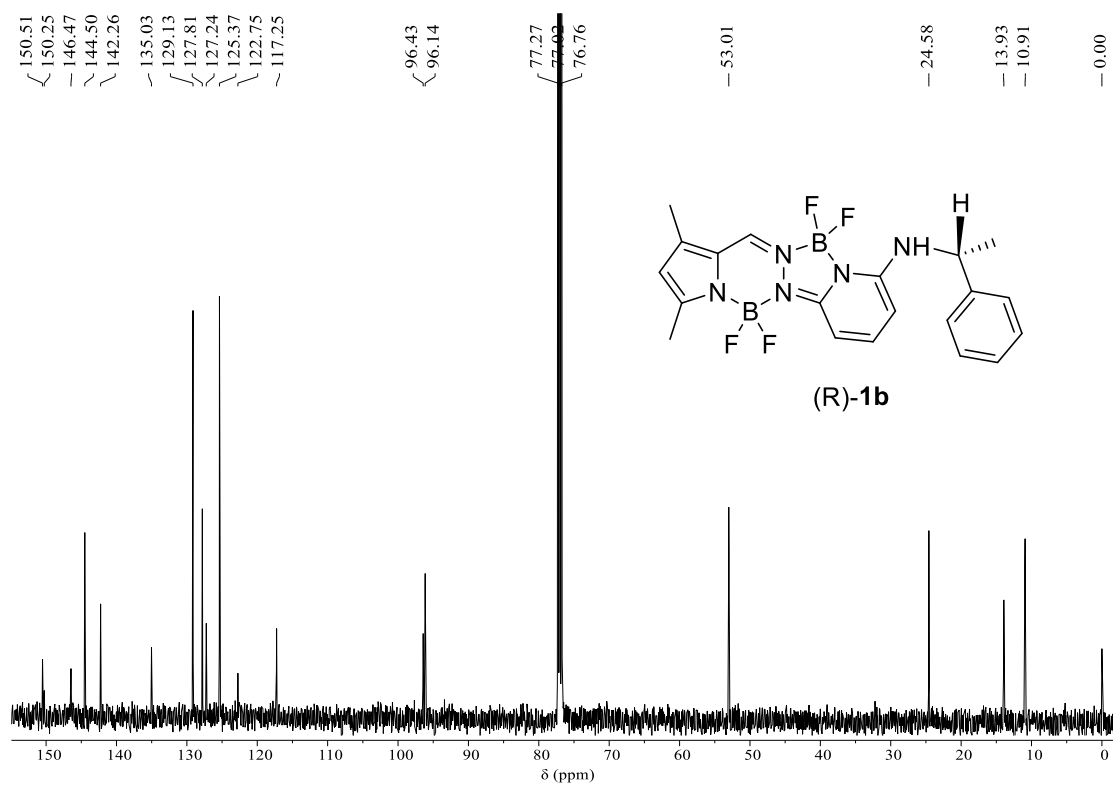
^1H NMR spectrum of **(S)-1b** in CDCl_3



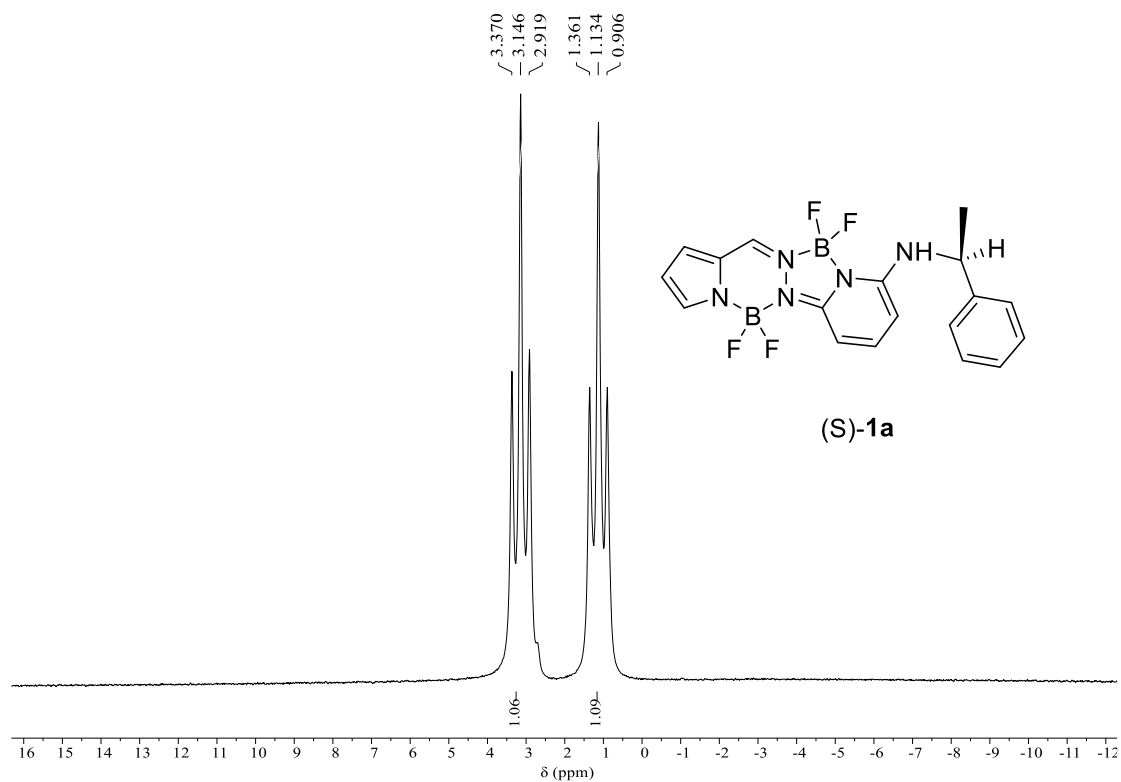
^{13}C NMR spectrum of **(S)-1b** in CDCl_3



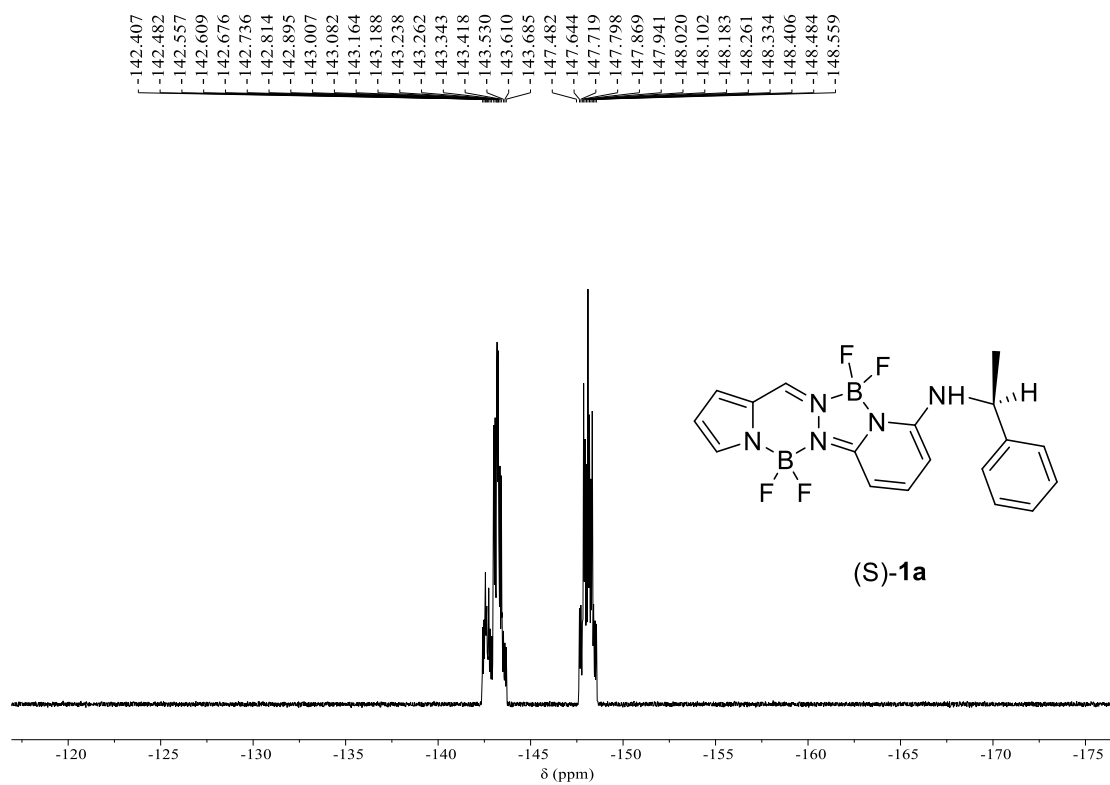
^1H NMR spectrum of (R)-1b in CDCl_3



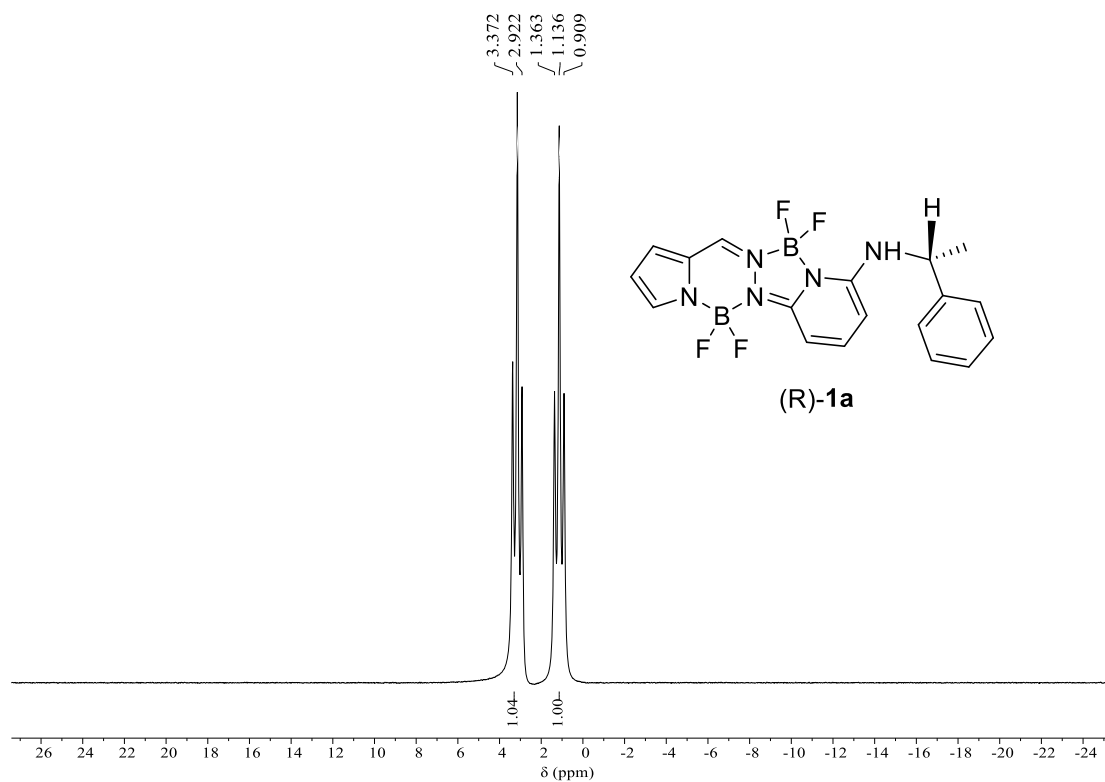
^{13}C NMR spectrum of (R)-1b in CDCl_3



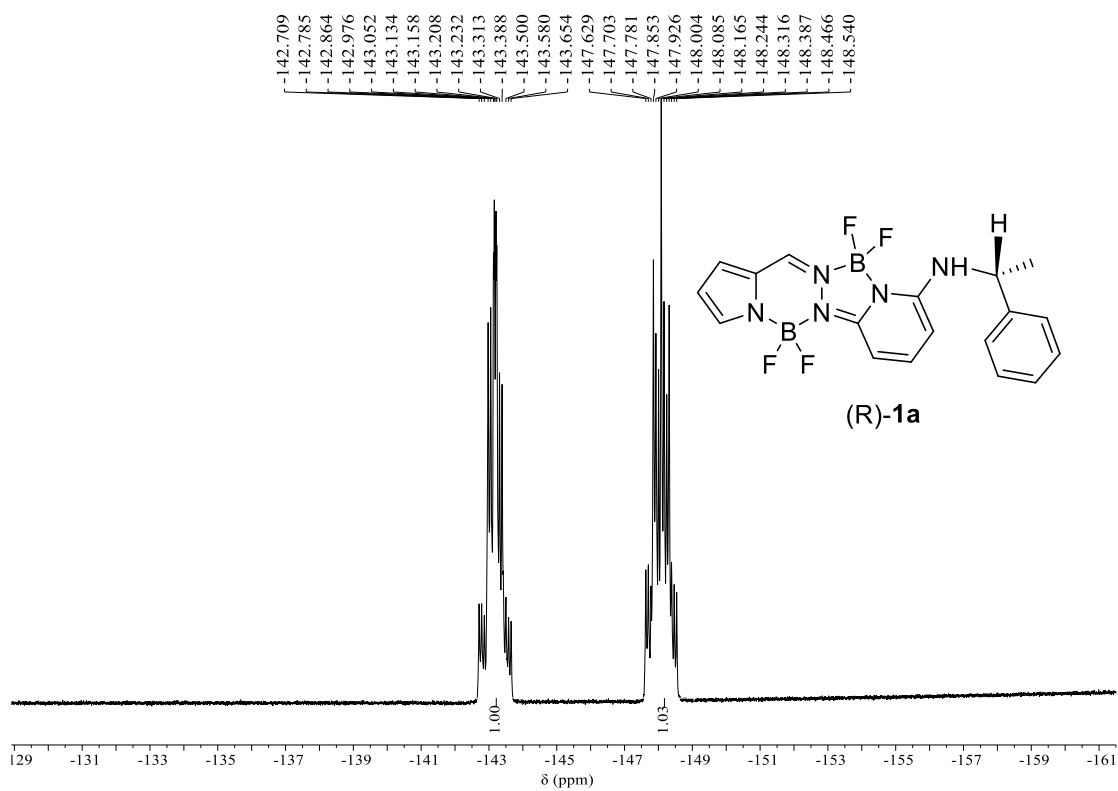
^{11}B NMR spectrum of **(S)-1a** in CDCl_3



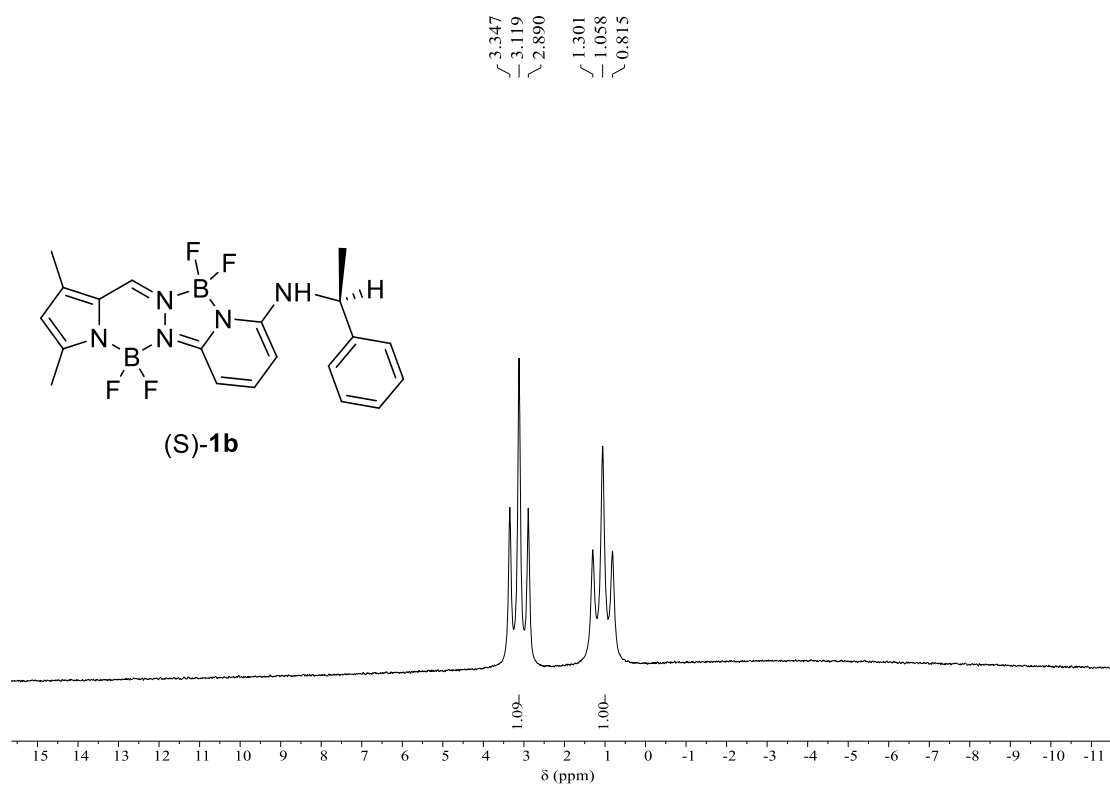
^{19}F NMR spectrum of **(S)-1a** in CDCl_3



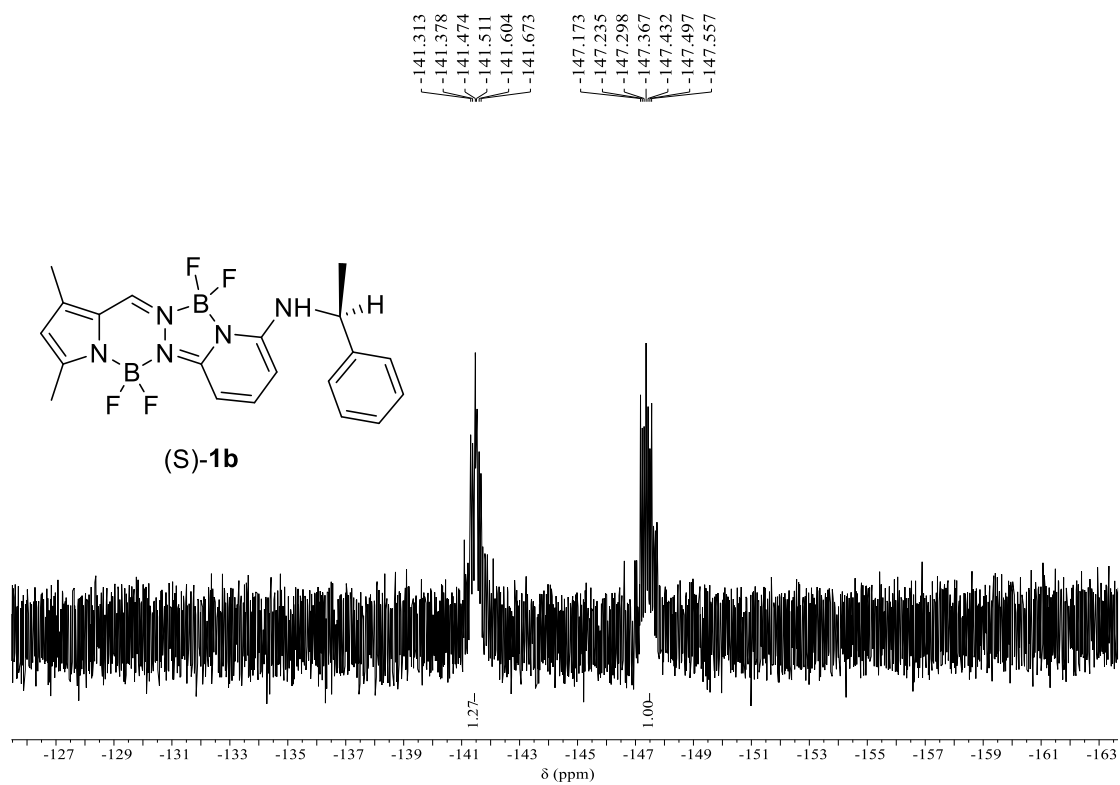
¹¹B NMR spectrum of (R)-**1a** in CDCl₃



¹⁹F NMR spectrum of (R)-**1a** in CDCl₃

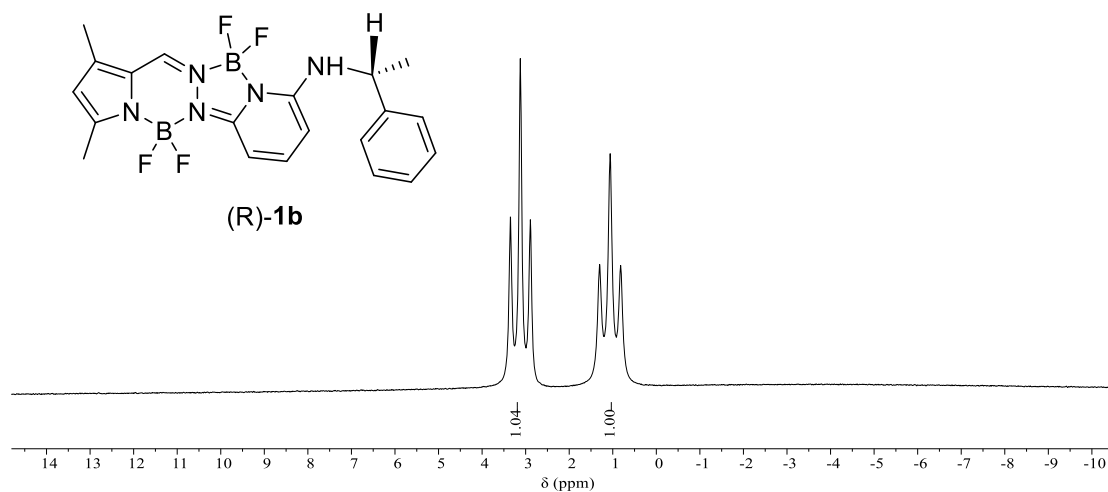


^{11}B NMR spectrum of (S)-**1b** in CDCl_3

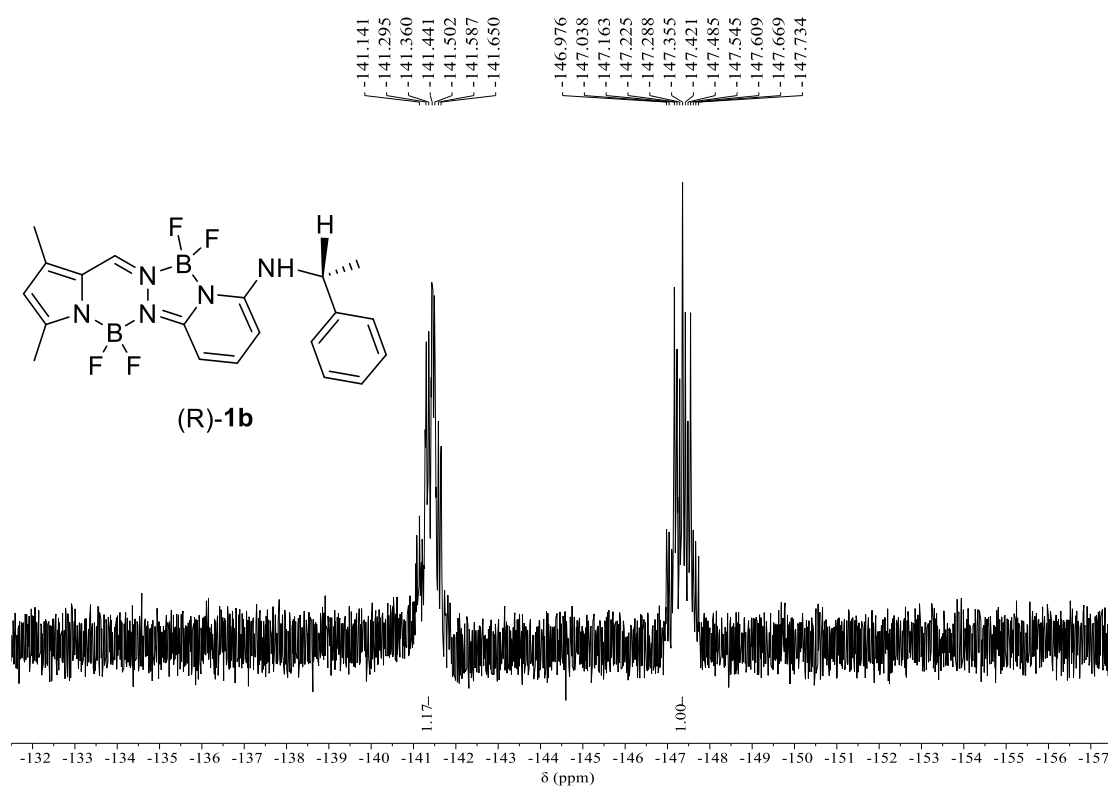


^{19}F NMR spectrum of (S)-**1b** in CDCl_3

/ 3.350
 \ 3.122
 \ 2.892
 / 1.304
 \ 1.060
 \ 0.818



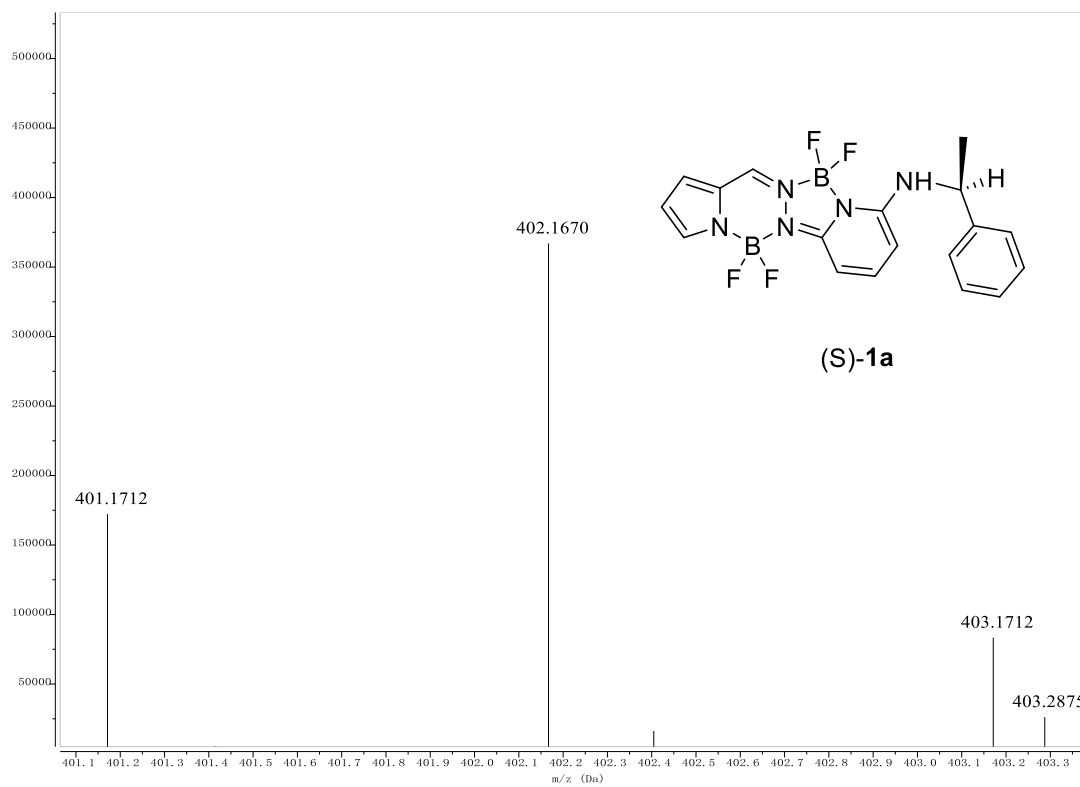
^{11}B NMR spectrum of (R)-**1b** in CDCl_3



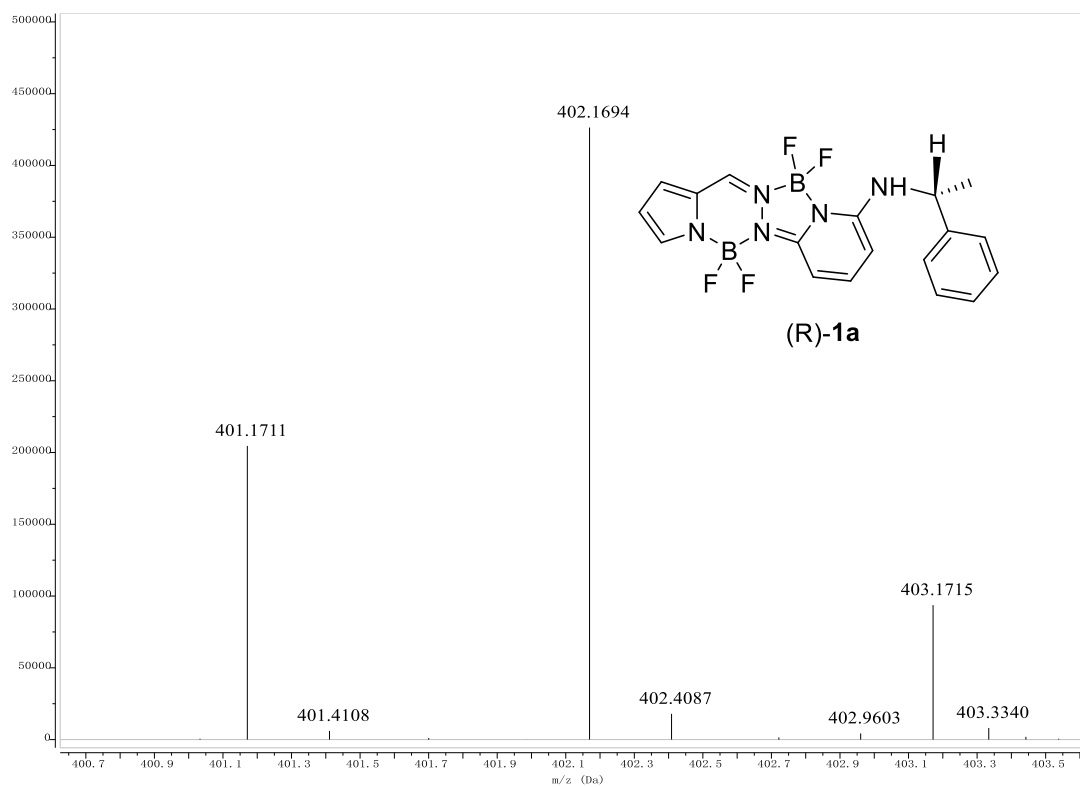
^{19}F NMR spectrum of (R)-**1b** in CDCl_3

10. HRMS for the new chiral BOPPYs

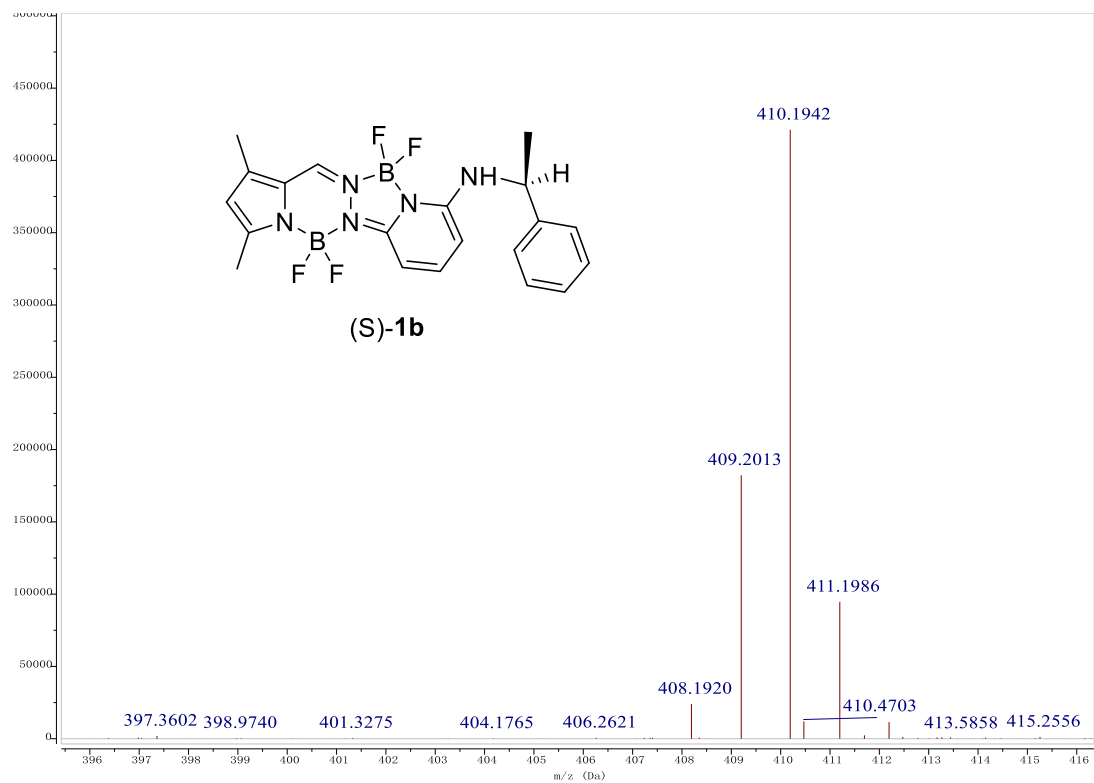
HRMS for (S)-1a



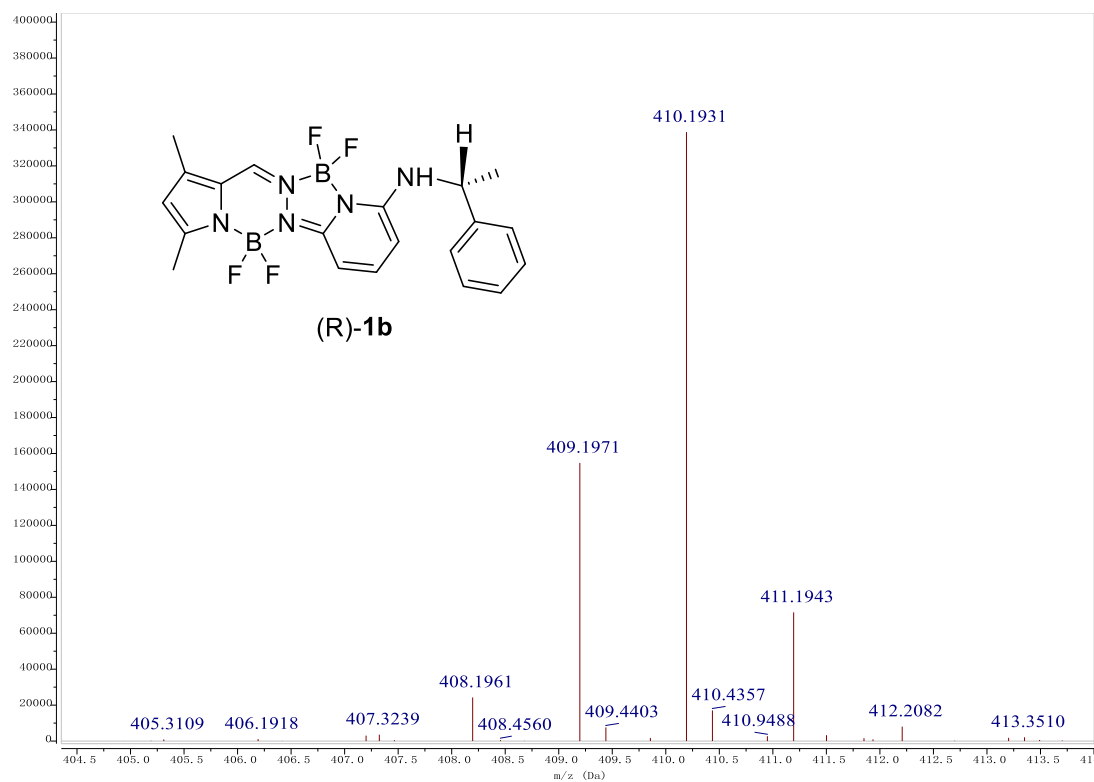
HRMS for (R)-1a



HRMS for (S)-1b



HRMS for (R)-1b



11. Author contribution statement

Yingzhu Sun: Investigation, data testing, validation, editing.

Changjiang Yu: Writing, supervision, reviewing, funding acquisition.

Wanping Qian: Data testing.

Hongtao Zhang: Writing about crystals.

Lijuan Jiao: Reviewing, supervision.

Jiazhu Li: Supervision, reviewing.

Minghua Liu: Supervision, reviewing.

Erhong Hao: Funding acquisition, validation, reviewing, supervision.