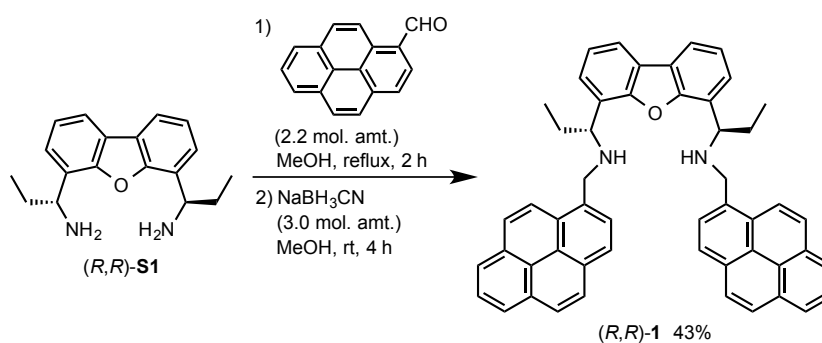


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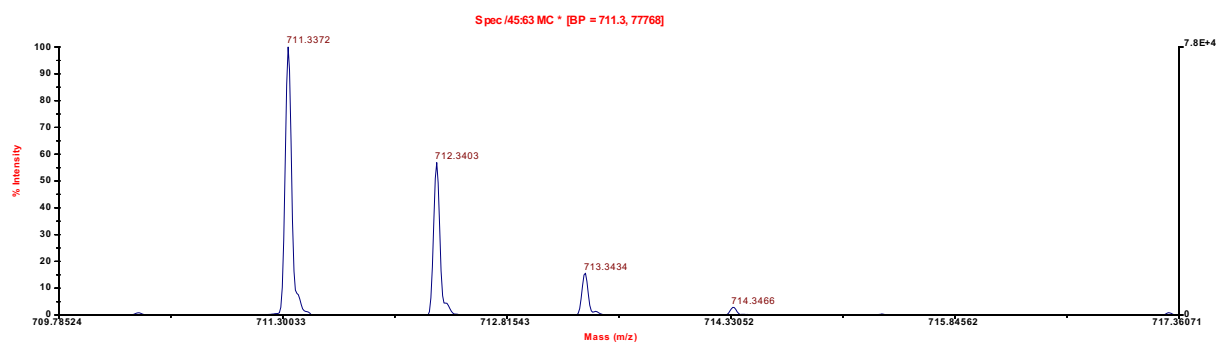
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1. Synthesis of (*R,R*)-1A



Scheme S1. Synthesis of chiral diamine (*R,R*)-1

HRMS data for (*R,R*)-1



Calcd., [M+H]⁺, 711.3375, Found, 711.3372

| Molecular Formula | Monoisotopic Mass | ppm | mda | unsaturation |
|---|-------------------|----------|----------|--------------|
| C ₅₂ H ₄₃ N ₂ O ₁ | 711.3375 | -0.47659 | -0.33901 | 32.5 |

2. Single-particle level observations and the measurement of fluorescence lifetimes

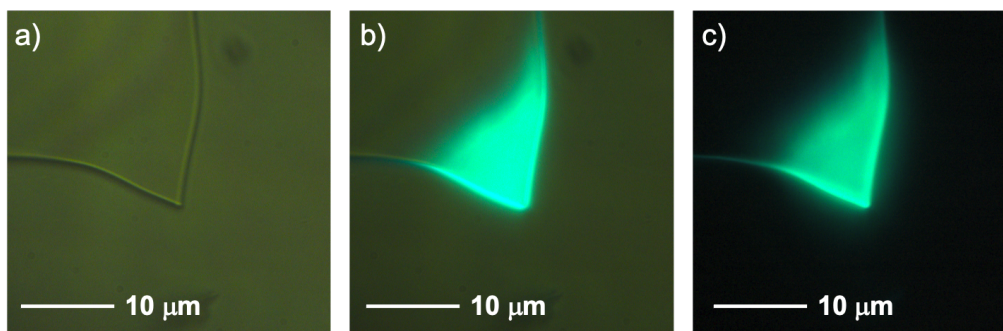


Figure S1 Photographs of as-prepared (*R,R*)-1A measured by fluorescence microscope. a) Under room light. b) Under room light excited with 405-nm continuous wave laser. c) In the dark excited with 405-nm continuous wave laser.

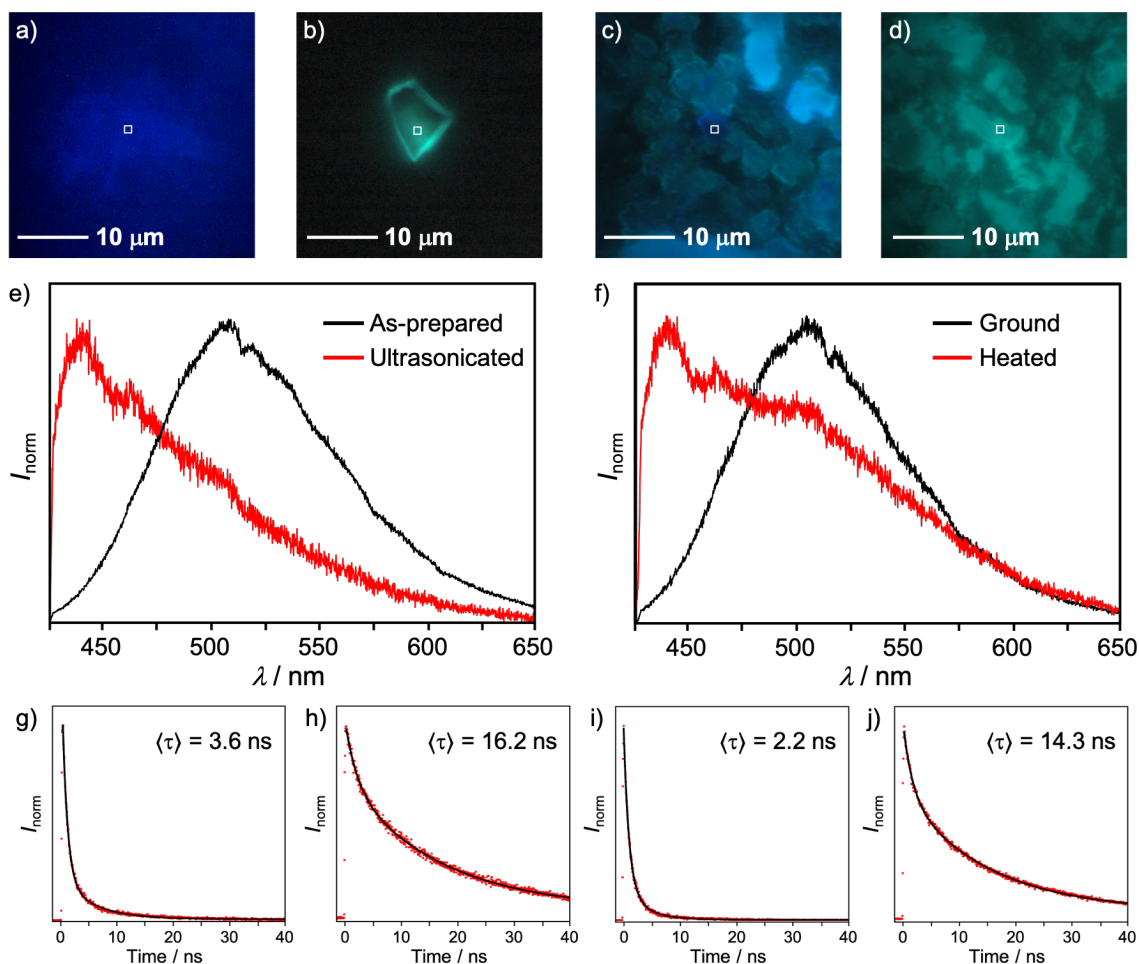


Figure S2 Photographs, fluorescence spectra, and fluorescence decay profiles for ultrasonicated (a, e, and g), as-prepared (b, e, and h), heated (c, f, and i), and ground (d, f, and j) samples of (*R,R*)-1 measured by fluorescence microscope. The square marks in the photographs indicate the measured locations. Excitation wavelength is 405 nm.

Table S1. Fluorescence lifetime (τ_n), intensity-weighted mean fluorescence lifetime ($\langle\tau\rangle$), and radiative (k_r) and non-radiative rate constants (k_{nr}) of ultrasonicated, as-prepared, ground, and heated samples of (R,R)-1.^a

| Sample | λ_{em} (nm) | τ_1 (ns) ^b | τ_2 (ns) ^b | $\langle\tau\rangle$ (ns) ^c | k_r (10^8 s ⁻¹) ^d | k_{nr} (10^8 s ⁻¹) ^d |
|----------------|---------------------|----------------------------|----------------------------|--|---|--|
| As-prepared | 508 | 2.06 (0.308) | 17.0 (0.653) | 16.2 | 0.3 | 0.3 |
| Ultrasonicated | 438 | 0.89 (0.941) | 5.60 (0.204) | 3.6 | 0.2 | 2.6 |
| Ground | 504 | 1.65 (0.340) | 15.1 (0.605) | 14.3 | 0.3 | 0.4 |
| Heated | 439 | 0.94 (0.866) | 3.76 (0.167) | 2.2 | 0.4 | 4.2 |

^a Excitation wavelength for the measurement of the fluorescence lifetime is 405 nm. ^b The coefficient a_n of the component is shown in parentheses. ^c Intensity-weighted mean fluorescence lifetime. $\langle\tau\rangle = (a_1\tau_1^2 + a_2\tau_2^2)/(a_1\tau_1 + a_2\tau_2)$. ^d k_r and k_{nr} were calculated from $\Phi_F = k_r/(k_r + k_{nr}) = \langle\tau\rangle k_r$.

3. Absorption and excitation spectra

The absorption bands of as-prepared, ultrasonicated, and ground samples of (*R,R*)-**1** were observed in almost the same region (Figure S3).

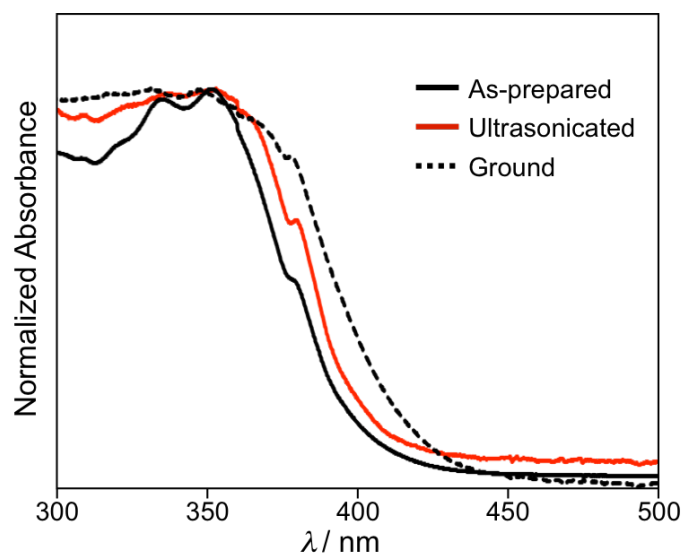


Figure S3 Solid-state absorption spectra of as-prepared, ultrasonicated, and ground samples of (*R,R*)-**1**.

The excitation maxima of as-prepared, ultrasonicated, and ground samples of (*R,R*)-**1** were also observed in almost the same region (Figure S4).

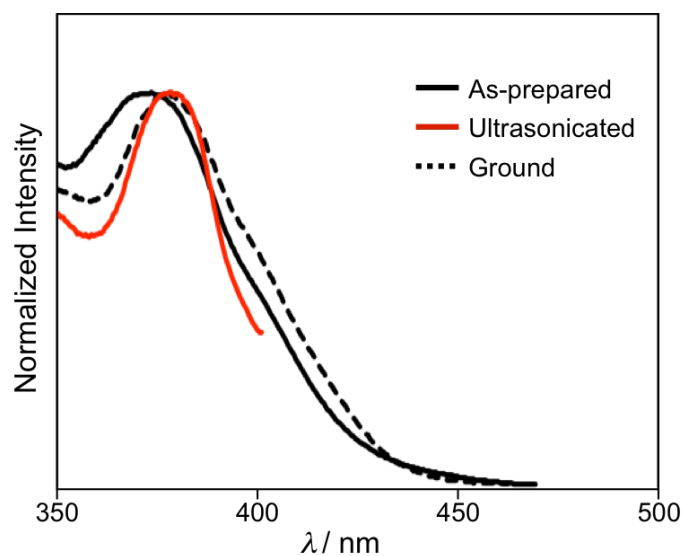


Figure S4 Solid-state excitation spectra of as-prepared (black line; $\lambda_{\text{em}} = 483$ nm), ultrasonicated (red line; $\lambda_{\text{em}} = 413$ nm), and ground (black dotted line; $\lambda_{\text{em}} = 483$ nm) samples of (*R,R*)-**1**.

4. Fluorescence spectra and PXRD patterns for ground and heated samples

Powder X-ray diffraction (PXRD) analysis showed the amorphization of crystal structures upon grinding ultrasonicated (*R,R*)-1. The intensity of diffraction patterns increased by heating the ground sample to 150 °C (Figure S5).

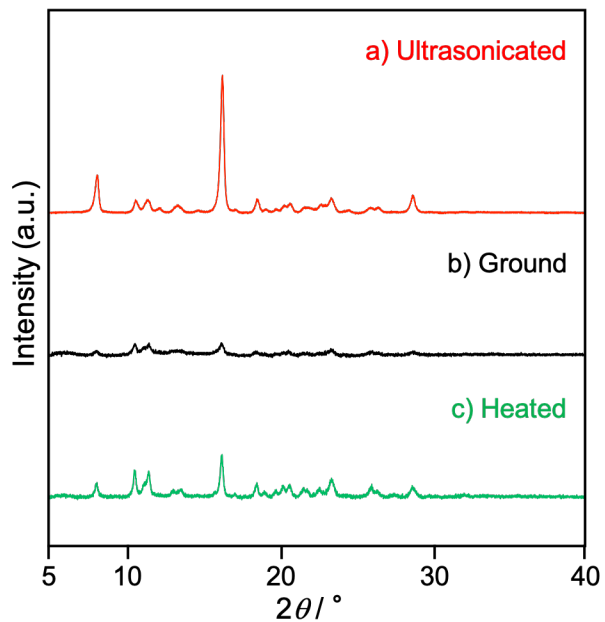
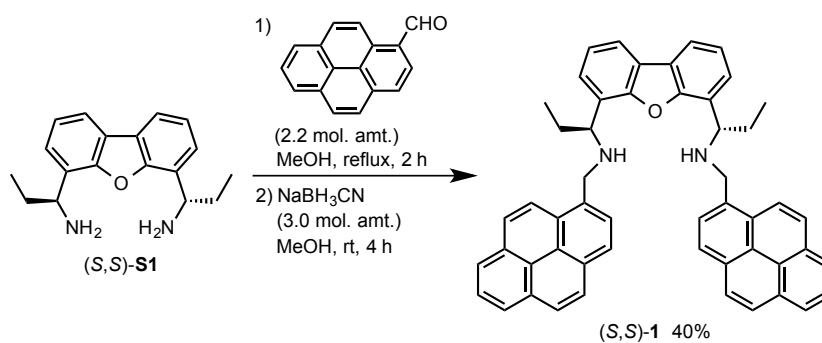


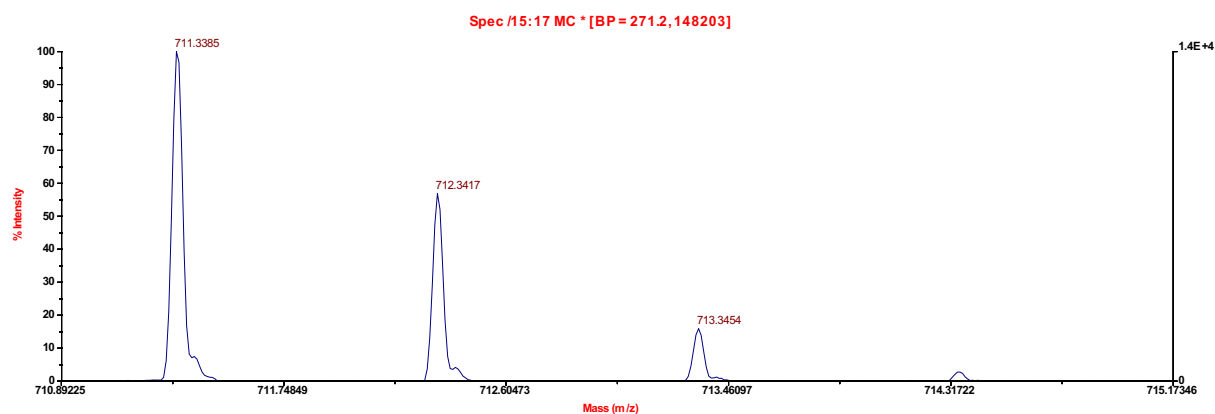
Figure S5 PXRD patterns for a) ultrasonicated, b) ground, and c) heated samples of (*R,R*)-1.

5. Synthesis of (*S,S*)-1



Scheme S2. Synthesis of chiral diamine (*S,S*)-1

HRMS data for (*S,S*)-1



Calcd., $[M+H]^+$, 711.3375, Found, 711.3385

| Molecular Formula | Monoisotopic Mass | ppm | mda | unsaturation |
|----------------------|-------------------|----------|----------|--------------|
| $C_{52}H_{43}N_2O_1$ | 711.3375 | 1.350956 | 0.960986 | 32.5 |

6. Fluorescence spectra for the MCL of (*S,S*)-1

Fluorescence spectra for the MCL of (*S,S*)-1 were in good agreement with those of (*R,R*)-1 (Figure S6).

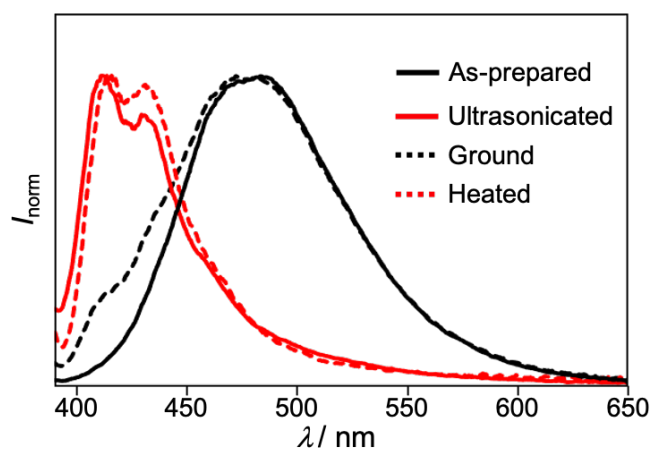
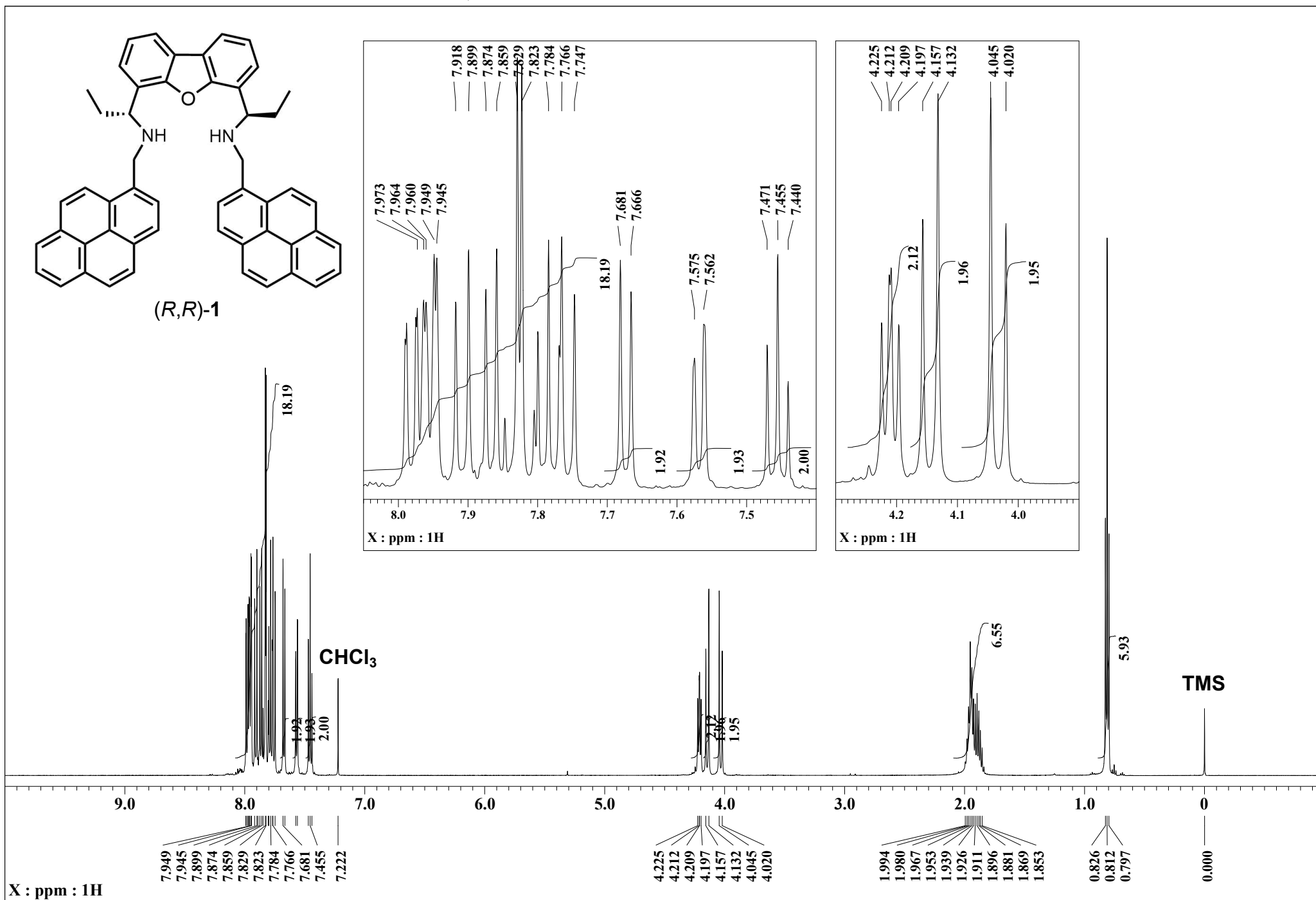


Figure S6 Fluorescence spectra for as-prepared, ultrasonicated, ground, and heated samples of (*S,S*)-1. Excitation wavelength is 365 nm.

¹H NMR spectrum of (*R,R*)-1 (500 MHz, in CDCl₃, rt)



Chemical Structure of (S,S)-1:

CC[C@H](NCC1=CC=CC=C2C=CC=CC=C12)C3=CC=C4C(=C3)C(=C5C=CC=CC=C45)O[C@H](NCC6=CC=CC=C7C=CC=CC=C67)C

¹H NMR Spectrum (CDCl₃):

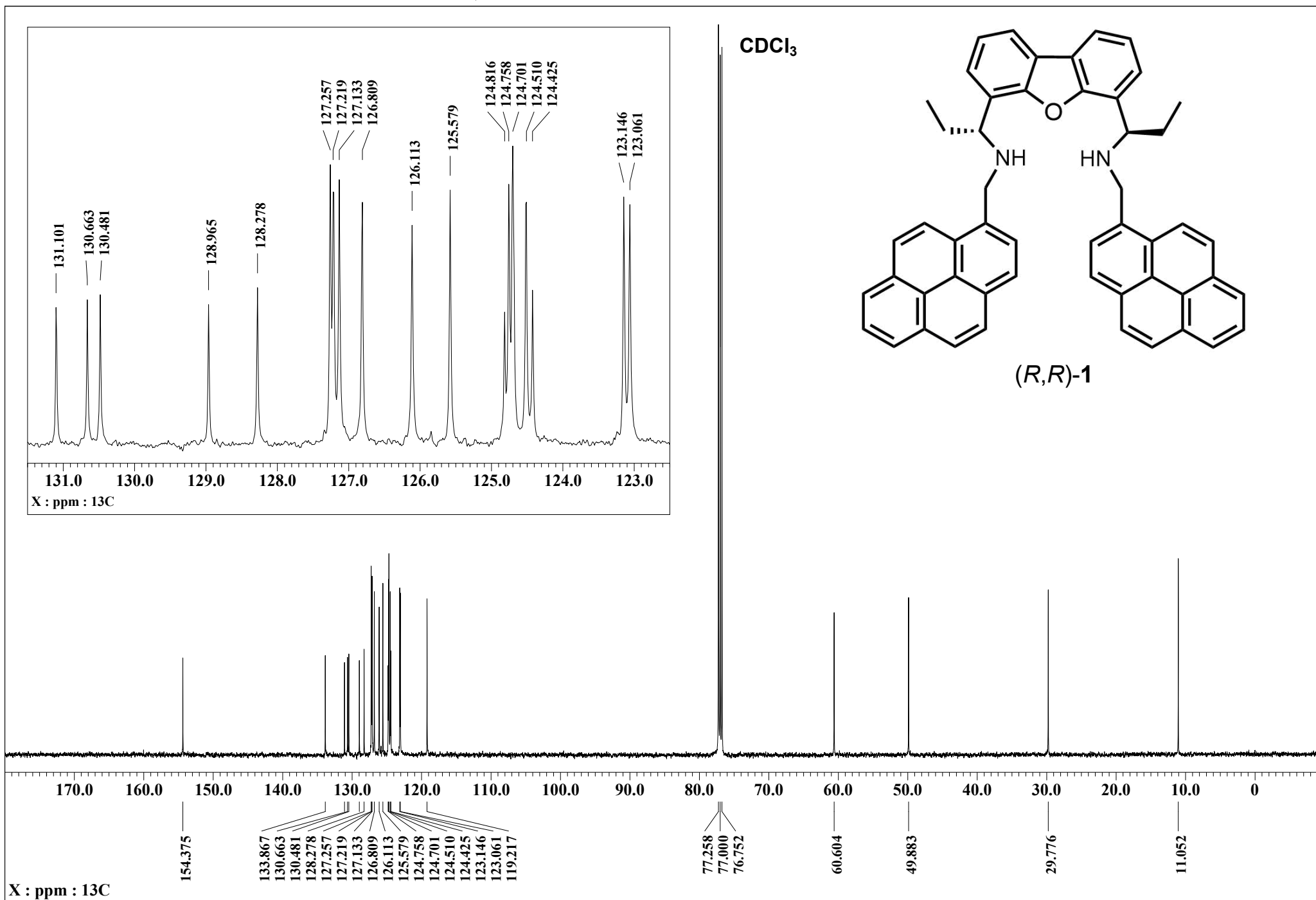
Chemical Shifts (ppm): 7.967, 7.962, 7.911, 7.891, 7.876, 7.848, 7.841, 7.801, 7.782, 7.694, 7.472, 7.251, 4.233, 4.221, 4.218, 4.205, 4.170, 4.144, 4.056, 4.032, 1.989, 1.977, 1.962, 1.947, 1.935, 1.919, 1.904, 1.889, 1.877, 1.862, 1.818, 0.833, 0.818, 0.803, 0.000.

Integration Values: 1.99, 1.94, 2.00, 1.99, 2.05, 2.02, 1.97, 6.44, 6.05.

Inset 1 (Aromatic Region): X : ppm : 1H. Peaks at 7.981, 7.978, 7.967, 7.962, 7.829, 7.911, 7.891, 7.876, 7.848, 7.841, 7.801, 7.782, 7.694, 7.678.

Inset 2 (Aliphatic Region): X : ppm : 1H. Peaks at 4.233, 4.221, 4.218, 4.205, 4.170, 4.144, 4.056, 4.032.

¹³C NMR spectrum of (*R,R*)-1 (126 MHz, in CDCl₃, rt)



¹³C NMR spectrum of (S,S)-1 (126 MHz, in CDCl₃, rt)

