Supramolecular chirality measured by diffuse reflectance circular dichroism spectroscopy

Natsuyo Asano,[a] Takunori Harada,[b] Tomohiro Sato, [b] Nobuo Tajima [c] and Reiko Kuroda*[ab]

ELECTRONIC SUPPLEMENTARY INFORMATION

1. Experimental.

Preparation of a KBr disc. Crystal **1** (PYR/Q) (62 μg) was ground with 130 mg of KBr and PYR (56 μg) with 133 mg of KBr. The water in the mixture was evaporated under vacuum for 10 min using a vacuum pump (ALVAC G-20DA) and the mixture was pressed into a pellet by a hand-operated hydraulic pump (RIKEN P-16B) at 70 MPa.

Measurements. Transmittance circular dichroism (CD) and electronic absorption spectra were measured by a Universal Chiroptical Spectrophotometer (UCS-1: Jasco J-800KCM). Diffuse reflectance circular dichroism (DRCD) and electronic absorption spectra were measured by UCS-3 (Jasco J-800KCMFII). X-ray data collection of single crystals was carried out on a Bruker APEX (CCD detector) at 100 K with Mo Kα radiation. The crystal structure was solved by direct methods and refined by full-matrix least-squares using SHELXS-97. X-ray powder patterns were recorded on a Rigaku X-ray diffractometer Multi Flex. The absolute configurations of complexes were determined by the Flack parameters.

2. DRCD and electronic absorption spectra of (*d*)- and (*l*)- camphorsulfonate.

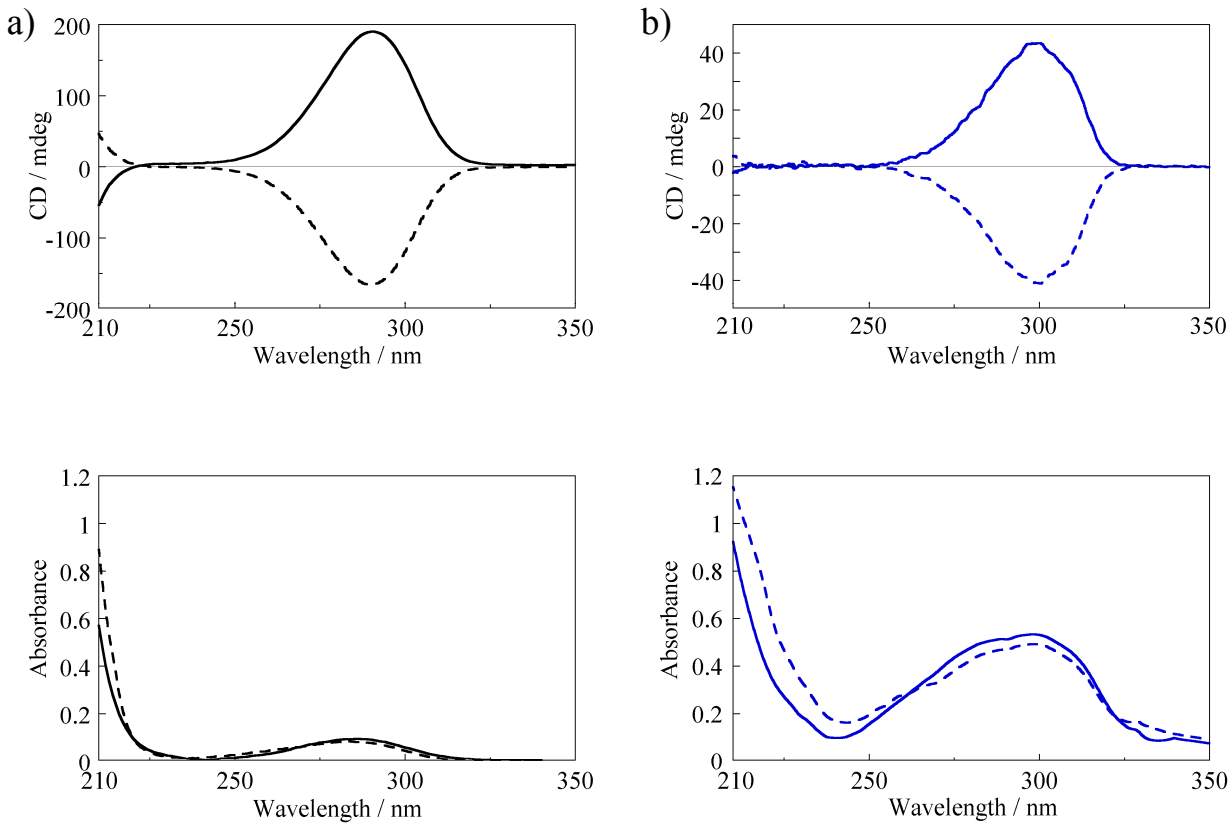

Fig. S1. CD (top) and electronic absorption (bottom) spectra of (*d*)- (solid line) and (*l*)- (dotted line) camphorsulfonate, in solution (a), and as microcrystallines measured by diffuse reflectance (DR) mode on UCS-3(b).

3. Additional data and structure of crystals 1 and 2.

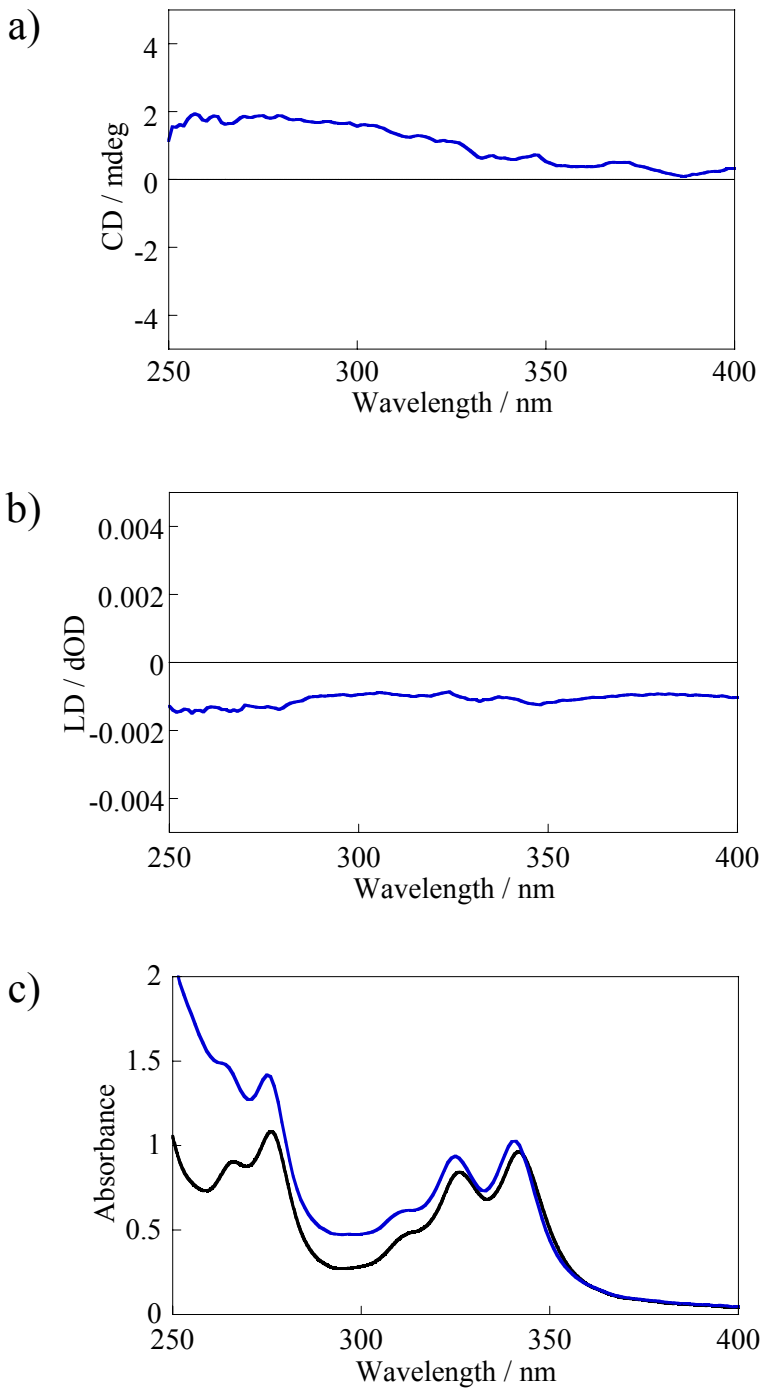

Fig. S2. (a) Transmittance CD, (b) LD and (c) electronic absorption spectra of **1** (PYR/Q) in a KBr pellet. A total amount was 130 mg containing 62 μg of crystal **1**. (c) PYR spectrum (black) is shown for comparison. A total amount was 133 mg containing 56 μg of PYR. During the disc formation, crystal **1** was changed to PYR crystal by sublimation of Q.

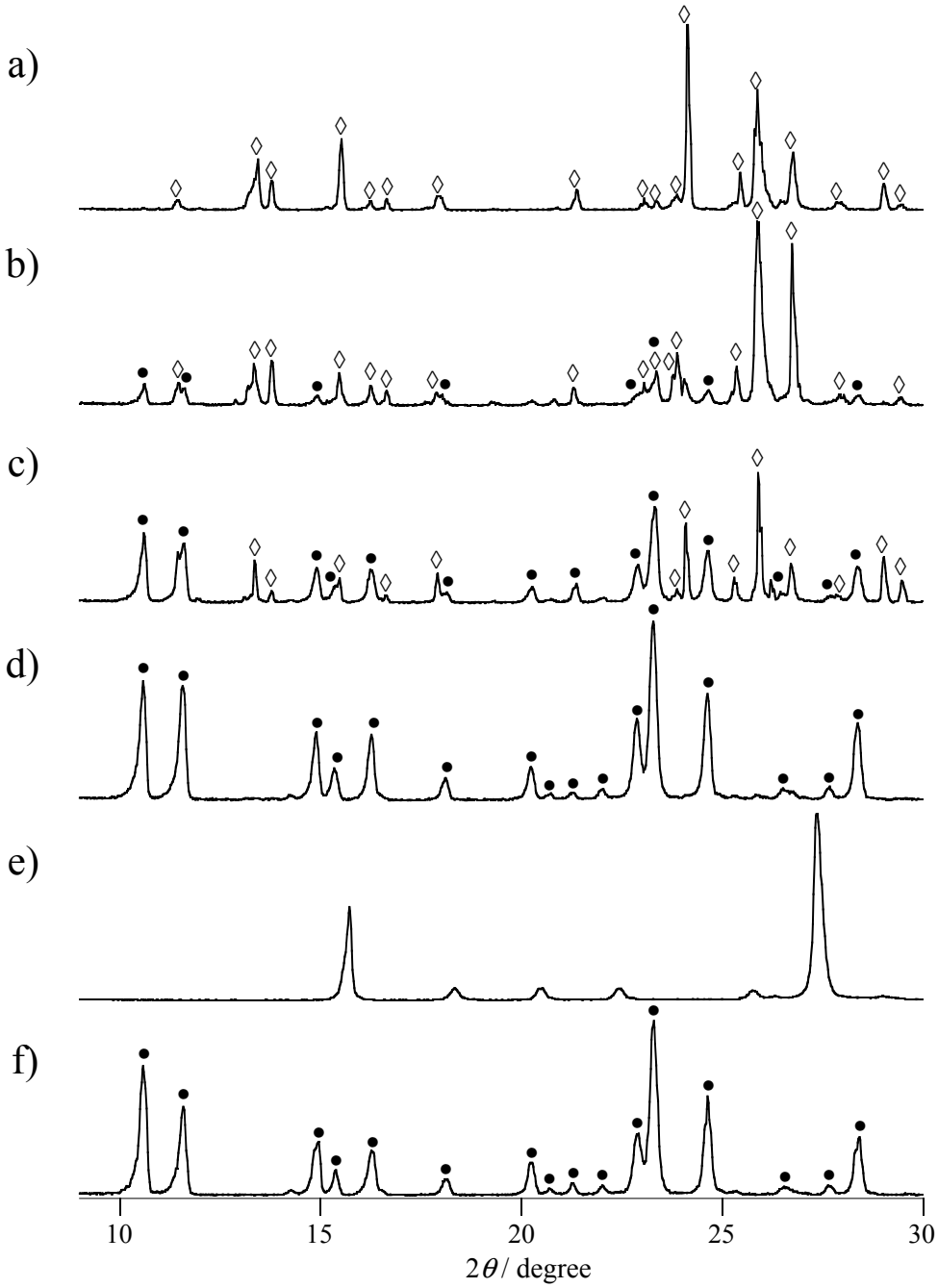

Fig. S3. X-ray diffraction patterns of **1** (PYR/Q) on exposure to air. (a) **1** at the beginning, (b) after 2 days of exposure, (c) after 9 days of exposure, and (d) after 18 days of exposure. (e) Q and (f) PYR are shown for comparison. Solid circle (•) and rhombus (◊) indicate the peaks belonging to PYR and **1**, respectively.

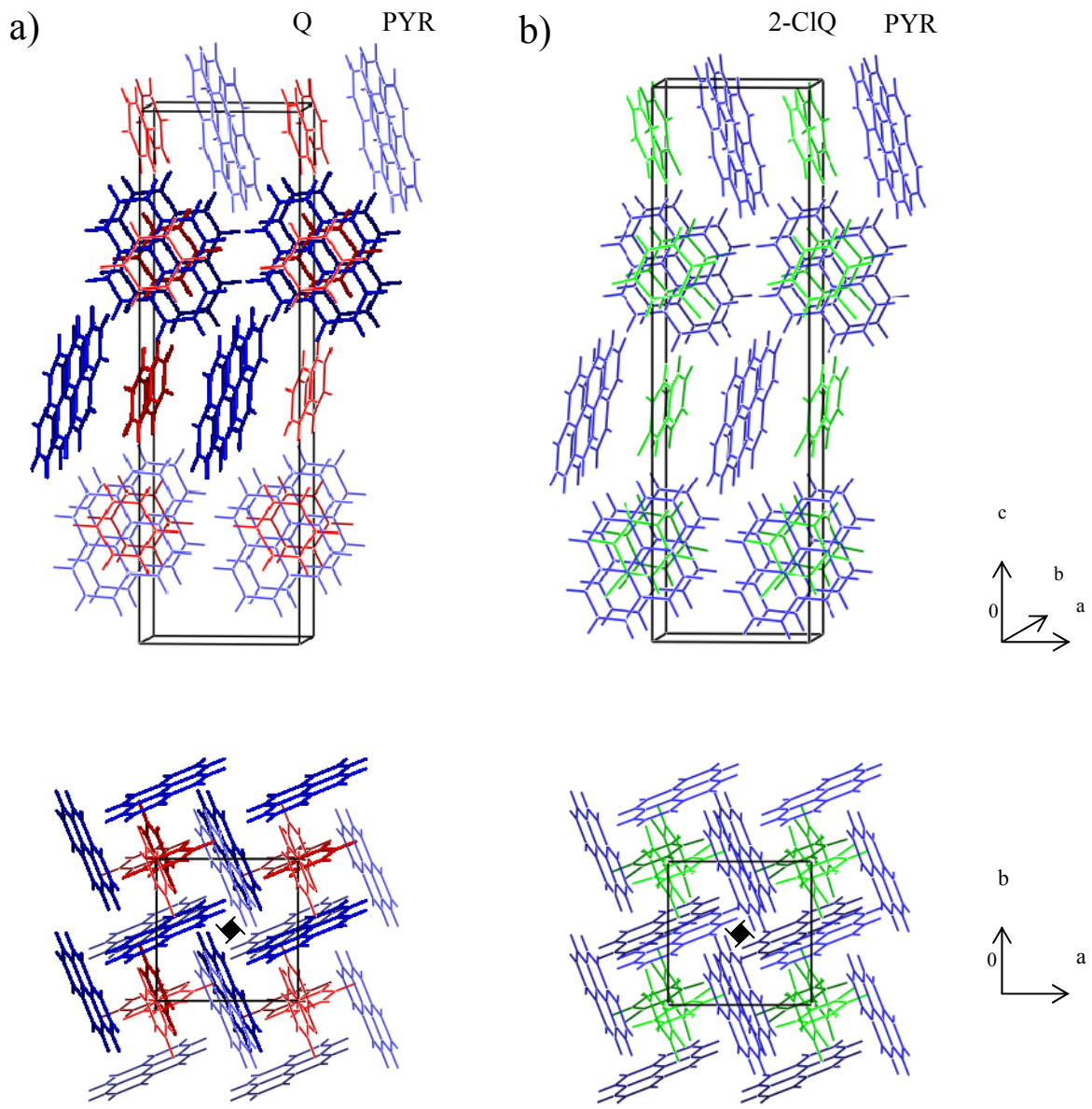

Fig. S4. Crystal structures of (a) **1** (PYR/Q) and (b) **2** (PYR/2-ClQ) in the space group of $P4_3$, along the *b* (top) and *c* axes (bottom). The molecular cluster $[(Q)_4(PYR)_8]$ is shown in darker colour.

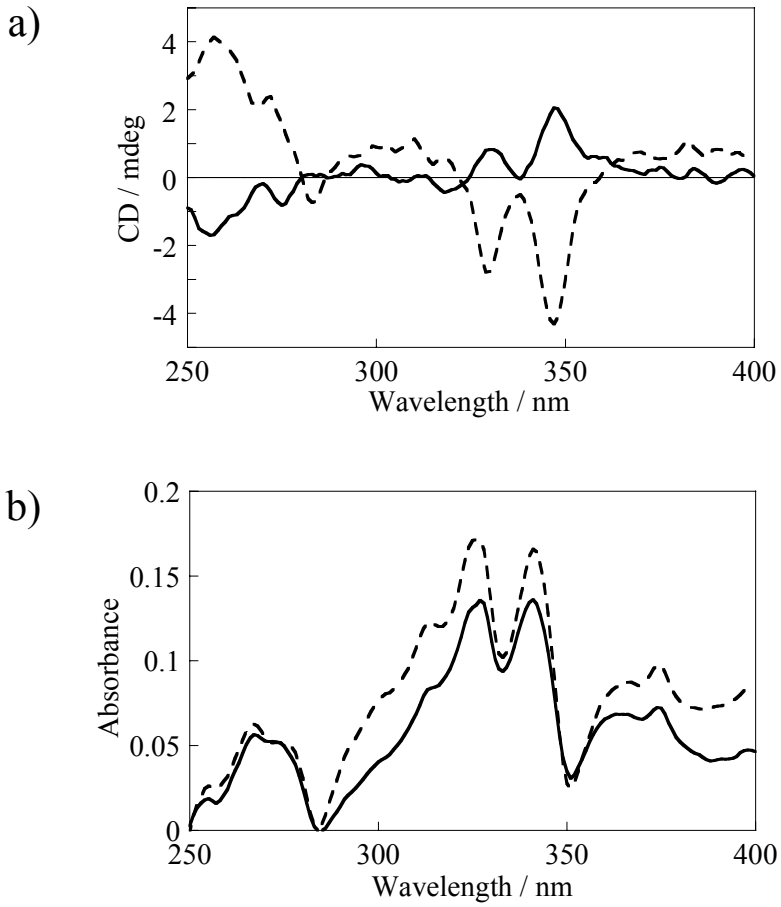

Fig. S5. (a) DRCD and (b) electronic absorption spectra of **2** measured on UCS-3 for the enantiomeric crystals (diluted with KBr matrix: 41 wt% (solid line) and 49 wt% (broken line)).