Supplementary information

Single-crystal structure determination from microcrystalline powders (~5 μm) by an orientation attachment mountable on an in-house X-ray diffractometer

Chiaki Tsuboi, Kazuaki Aburaya, Fumiko Kimura, Masataka Maeyama, and Tsunehisa Kimura*

Materials and methods

Preparation of an L-alanine microcrystal suspension

An as-received sample of L-alanine crystals (Wako Pure Chemical Industries, Ltd.) was pulverized using a mortar and pestle and passed consecutively through 125-, 75-, 45-, and 20-mesh sieves. Then, the powders remaining on the 20-mesh sieve (powder A) and the powders passed through (powder B) were collected. Powder A was dispersed in UV light-curable monomer (Kyoritsu Chemical XVL-14, viscosity 12±2 Pa s) to prepare 20 wt% microcrystal suspensions (suspension A). Powder B was also dispersed in the same monomer and allowed to stand for a few weeks to make larger microcrystals sink. Then, the upper part of the suspension was taken (suspension B). The sizes of microcrystals present in each of suspensions A and B determined by microscope were 20-45 μm and <5 μm, respectively. In this study, the UV light-curable monomer was used only for a suspending medium because it formed a stable well-dispersed suspension of microcrystals; photopolymerization was not performed. Each suspension was poured into a glass capillary (2.5 mmφ) and placed on a magnetic unit (a Kyoto University KU model χ10-3) comprising a rotating unit and a pair of neodymium magnets (~1 T in the center). The capillary was rotated by a stepping motor controlled by a computer. The shutter was a metal disk with a diameter of 44 mm made of aluminum and copper plates with a thickness of 2 and 1 mm,
respectively, rotated by a stepping motor in synchronization with the rotation of the capillary. The shutter contained two slits with a width of $\Delta\beta = 10^\circ$ separated by 180°.

**In situ X-ray measurements of a MOMS**

The orientation attachment (magnetic unit and shutter) was mounted on a Rigaku R-AXIS RAPID diffractometer (collimator size = 0.8 mm $\phi$, graphite-monochromated Mo K\textalpha radiation, crystal-to-detector distance = 127.40 mm) where the goniometers for $\chi$ and $\phi$ angles were removed. The rotation speed $\omega$ of the glass capillary was switched between $\omega_s = 0.93$ rpm and $\omega_q = 3.0$ rpm every 90°. The X-ray measurement began 3 h after the start of nonuniform sample rotation, when 3D alignment of the microcrystals was achieved.

Data were collected at room temperature to a maximum 2$\theta$ value of 54.8°. The angle $\alpha$ was altered from 0° to 200° and 340° to 360° for suspension A, and from 0° to 90° and 230° to 360° for suspension B in increments of 10° by changing the starting position of the slits in the shutter before each measurement, respectively. The measurement time for suspension A was 3000 s for the ranges between 0–90° and 180–200° and 930 s for 90–180° and 340° to 360° to ensure that the net X-ray irradiated time per unit degree was 8.3 s for every measurement. The measurement time for suspension B was 8000 s for the ranges between 0–90° and 230–270° and 2480 s for 270–360° to ensure that the net X-ray irradiated time per unit degree was 22.2 s for every measurement. Each measurement time was calculated on the basis of the rotation speeds $\omega_s$ and $\omega_q$ used. In both measurements, a total of 22 diffraction images were recorded corresponding to the number of different starting positions of the slits. A measurement without the shutter was also performed for suspension A.

**Single-crystal analyses**
Data were processed using RAPID AUTO software and the diffraction spots were indexed. The crystal structure was solved by direct methods\textsuperscript{[1]} and then expanded using Fourier techniques and refined. All calculations were performed using the CrystalStructure\textsuperscript{[2]} crystallographic software package except for refinement, which was performed using SHELXL-2013\textsuperscript{[3]}. The calculation was performed without prior knowledge of the L-alanine crystal. The diffraction spots located beyond $2\theta = 50.9^\circ$ were not used because this area was shadowed by the magnetic unit.

References

