Supplementary information

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

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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 $\Delta\beta = 10°$ 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 ϕ, graphite-monochromated Mo Kα radiation, crystal-to-detector distance = 127.40 mm) where the goniometers for χ and ϕ angles were removed. The rotation speed ω of the glass capillary was switched between ω_s = 0.93 rpm and ω_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 2θ value of 54.8°.   The angle α 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 ω_s and ω_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[1] and then expanded using Fourier techniques and refined. All calculations were performed using the CrystalStructure[2] crystallographic software package except for refinement, which was performed using SHELXL-2013.[3] The calculation was performed without prior knowledge of the L-alanine crystal. The diffraction spots located beyond $2\theta = 50.9°$ were not used because this area was shadowed by the magnetic unit.

References

S1. M. C. Burla, R. Caliandro, M. Camalli, B. Carrozzini, G. L. Cascarano, C. Giacovazzo, M. Mallamo, A. Mazzone, G. Polidori and R. Spagna, *J. Appl. Crystallogr.*, 2012, **45**, 357.

S2. CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation, Inc.: Tokyo, Japan, 2000.

S3. Sheldrick, G. M. *Acta Crystallogr. C.* 2015, **71**, 3.