X-ray Structure Analysis of Host-Guest Organic MicroCrystals

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Although the very useful synthetic route of *trans*-dihydrofuran derivatives (Product:**P**) by photoreaction of the Guest(**G**) molecule in MeOH has been established, the reaction gives *rac*-**P** together with some other by-products. But when the photoreaction of **G** was carried out in an inclusion crystal with the optically active host compound (Host:**H**), optically active **P** was obtained. Furthermore, photoirradiation of the inclusion crystals with the molecular ratio of H:G=1:1 and H:G=2:1 gave stereographically opposite **P**. The purpose of this study is to understand the mechanism of this interesting reaction crystallographically. Crystal structure of the H:G=1:1 compound was determined by legacy four circle diffractometer, but the H:G=2:1 crystal would not grow to the suitable size for ordinary X-ray study. So synchrotron radiation and the Vacuum Camera at BL02B1 was necessary to determine the structure of the microcrystal of H:G=2:1.

The energy of the incident beam was set to 15 keV from Si(111), which was focused by mirror. Our beam time began at 15:00 and the first exposure began at 23:30, so we needed 8.5 hours to set up the equipment.

Integrated intensity of the host-guest organic microcrystal, $C_{80}H_{57}O_{10}N_1$, was measured using the Vacuum Camera. The size of the crystal was $100 \times 40 \times 10$ μm^3. Each photograph was taken by rotating the crystal by 5° thirty times at 10°/min. It required about 30 minutes for one photograph. A total of 19 photographs were obtained which covered 76° of ϕ rotation. The ring current was changed from 57.3 to 35.8 mA during the whole measurement and 1-3 mA for one measurement.

The collected data were processed by computer program DENZO installed at BL02B1. Indexing was carried out successfully. Structure determination is in progress.