Structure Determination of Small Crystals of Organic Functional Compounds

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Crystal structure determinations are essential for many studies on the mechanisms of functional appearances in the organic crystals or the determination of the reaction path in the organic syntheses. The size of single crystals, however, required with the conventional X-ray source is about a few hundred μm. For example, in the case of crystals of charge-transfer complexes especially grown electrolytically, their size tend to be very small and their shape will be thin plates or fine needles so that the structure analyses of these crystals were often very difficult. The high flux X-ray beam from SPring-8 will make possible the structure factor measurement of crystallites with dimensions less than 100 μm. In this study, three kinds of small organic crystals were subjected to the measurement of diffraction data using the vacuum camera implemented on BL02B1. Each crystal is so small that the measurements of diffraction data were failed by the conventional X-ray source. The wavelength was corrected by the K edge of Zr (17.998keV) and set to 17.5keV (0.7085Å). Diffraction patterns were recorded on the cylindrical Imaging Plates (radius of 75mm) using the oscillation method. The ring current was ranged from 17.0 to 19.0mA. The Bragg spots upto about 2θ of 55° were clearly seen in each exposure. The experimental conditions are summarized in Table 1. For the crystal **I**, which is the precursor of a certain chemical luminescence compound, the integrated intensities were successfully obtained by the program DENZO The structure analysis with these data gave a reasonable result. Large R factor can be ascribed to the large thermal displacement of the terminal carbon atoms. The crystal **II** is a CT-complex crystal grown electrolytically. In this case, the specimen was found to be twinned in the course of the integration procedure. The diffraction spots of the crystal **III** are too closed because the unit cell, in which the long alkyl chains with 10 to 15 carbon atoms exist, must be very large. So that the integration processes of these crystals are still in progress.

Table 1. Experimental conditions

Crystal	I	II	III
Formula	$C_{38}H_{17}N_3O_3$	$C_{16}H_{10}S_{12}I_3$	$C_{31}H_{64}N_2Br_2$
Crystal size /μm	230×130×50	200×80×30	110×40×2.5
Δφ for each IP /°	6	6	3
No. of exposures	20	20	19
φ range /°	101	101	37
a,b,c /Å	12.657, 19.763, 14.067	-	-
β /°	109.27	-	-
No. of refs			
measured	19416	-	-
unique	6512	-	-
R	0.101	-	-

The authors are grateful to Dr. Konishi and Dr. Yamagata of SPring-8, JASRI for their kind support in using the beam line.