Precise Crystal Structure Analysis of $K_3H(SO_4)_2$ by High Energy X-ray Diffraction Method

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1. Introduction

In order to clarify the mechanism of the structural phase transition, the very precise structure analysis is one of the powerful methods. But, the several factors, for example, absorption and extinction effect and inelastic scattering effect etc. make hard the precise structure analysis. High energy X-ray diffraction technique is the effective method to do the precise structure analysis. The effects of the high energy x-ray are the decrease of the absorption and the high resolution Fourier synthesis. Specially, the reduction of the extinction correct is the most effective factor.

Some years ago, we tried the high energy (50keV) X-ray structure analysis at BL14A of Photon Factory[1]. The diffraction intensity was considerably weak, and the Bragg reflection was too sharp to determine the peak position by the then automatic computer system. As the results, typical integrated intensity at 50keV experiment is few thousands counts, and the R-factor was so poor. It was necessary the more optimized beam line to get much more intensity and proper resolution.

At BL02B1 of SPring-8, the diffractometer and the control system were planed to carry out the high energy X-ray structure analysis. We tried, again, the high energy X-ray structure analysis of $K_3H(SO_4)_2$ crystal. This crystal is given an attention, which the difference of electron number around the hydrogen and deuterium atoms was discussed with the connection of the isotope effect of hydrogen bond dielectric compounds.[2),3)]

2. Experiment and Result

Experiments were carried out at BL02B1 of Spring-8. Spherical shaped single crystal of $K_3H(SO_4)_2$ was used. Previously, UB-matrix of the sample was determined in laboratory experiment. X-ray radiation coming from Bending Magnet was monochromatized by Si(511) and a short distance scintillation counter was used. By modification of the control program, Peak Search and Peak Refinement were partially done with the trouble of the interface, etc. The refinement of the lattice constants could be performed with using the 8 reflections. The ω-scan profile of observed (006) reflection on the way of the peak refinement was shown in Fig.1. The peak width is about 0.013 degrees. Some data points were observed in order to refine the peak position.

In this experiment, we confirmed the work of the peak search mode and peak refinement mode with some troubles. But, we could not carry out an automatic measurement of the integrated intensity because of the time over. At next beam time, we will done the collect the diffraction data utilized high energy X-ray.

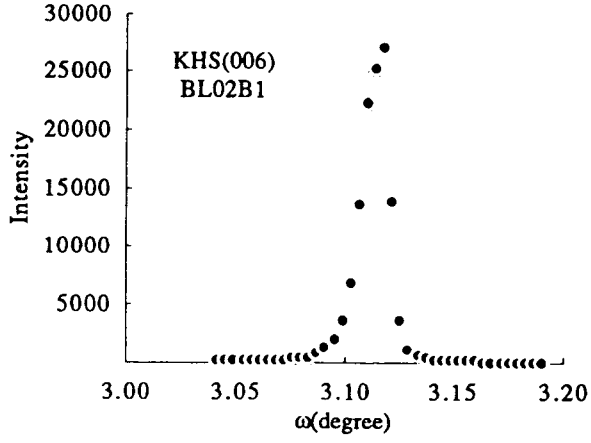

Fig.1 ω-scan profile of observed (006) reflection

1) Y.Noda, H.Kasatani, I.Tamura, Y.Kuroiwa and H.Terauchi: PF Activity report **11** (1993) 297.
2) Y.Noda and H.Kasatani: Kotaibutsuri **28** (1993) 31.
3) Y.Noda and H.Kasatani: J. Phys. Soc. Jpn. **60** (1991) 13.