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 of the precise structure analysis. Specially, the reduction of the extinction correct and the high resolution Fourier synthesis are the most effective factors.

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 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(Crystal Structure Analysis) of SPring-8, the diffractometer and the control system were planed to carry out the high energy X-ray structure analysis. We tried the high energy X-ray structure analysis of $K_3H(SO_4)_2$ crystal, again. This crystal is discussed with the connection of the isotope effect of hydrogen bond dielectric compounds; especially, the difference of electron number around the hydrogen and deuterium atoms.[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 means of the modification of the control program and the optimizing the value of the several parameters, Compensation of the optical axis(setp), Peak Search and Peak Refinement were done with the trouble of the interface, etc. We tried the automatic measurement of the integrated intensities in the reciprocal lattice point. But, we could not collect the effective value of the integrated intensity, because of the poor result of the automatic lattice refinement.

In this experimental beam-time, we confirmed the work of the peak search mode, the automatic peak refinement mode and the automatic collection of the integrated intensities in reciprocal lattice point with some troubles. But, we could collect an automatic measurement of the effective integrated intensity because of the poor result of the automatic lattice refinement. At next beam time, we will done the modification of the automatic lattice refinement mode and the automatic collection mode of the integrated intensities in the reciprocal lattice points, in order to success the high energy X-ray precise crystal structural analysis.

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.