Highly Accurate Measurement of Electron Density Distribution in Transition-Metal Complexes with SR and Vacuum Camera

Kiyoaki Tanaka(3189)*,

Department of Materials Science and Engineering,Nagoya Institute of Tecnology

Elizabeth A. Zhurova(3499)

Japan Science and Technology Corporation

Yasuyuki Takenaka(3006)

Hokkaaido University of Education at Hakodate

Vladimir V.Zhurov

Karpov Institute of Physical Chemistry, Moscow, Russia

1. Introduction

The results of our experiments for $KNiF_3$ using the vacuum camera(VC) are reported. We call the measurement with a VC as X-ray vacuum camera imaging-plate (VCIP) method. The present measurement is a supplementary one to that in November 1997. The data are now being analyzed together with those measured with a VC installed on our four-circle diffractometer equipped with a rotor X-ray generator. There were many problems for the VCIP method to be refined as an method for accurate intensity measurements. The problems and preliminary results are reported here.

2. Experimental

The experimental condition is the same as the measurement in November 1997. A total of eleven rotation photographs were taken by oscillating the crystal by 7° ninety times. The total rotation angle ranges from 0 to 55°. The rotation angle were overlapped by 2° for the scaling of intensities recorded on separate IP.

3. Indexing and Intensity Integration

Indexing of peaks were done with the program DENZO.[1] However the integration was carried out using the program made by one of us(V.V. Z.). The program permits to assign a relatively small integation box along the direction from the origin to the peak. The background approximation can be done with a formula, $z=ax+by+c$, where z is the background counts, x and y are coordinates from the center of the peak. a, b and c are determined for each reflection by the least-squares method using z values in the background box. This permitted to improve considerably the accuracy of integration, especially for high-angle reflections. It also corrct for the intensity increase due to slantwise crossing of the diffracted beams into IP. R factor of the data measured for $KNiF_3$ by the VCIP method with our laboratory system was reduced to 1.1% after the promolecule refinement. The program for detecting reflections perturbed significantly by multiple diffraction is now being made. The refinement for the Spring-8 data has started and the results will be presented elsewhere.

References

1) Z.Otwinowski, W. Minor, Methods in Enzymology, 276A: Macromolecular Crystaallogaphy, 307-326(1997)