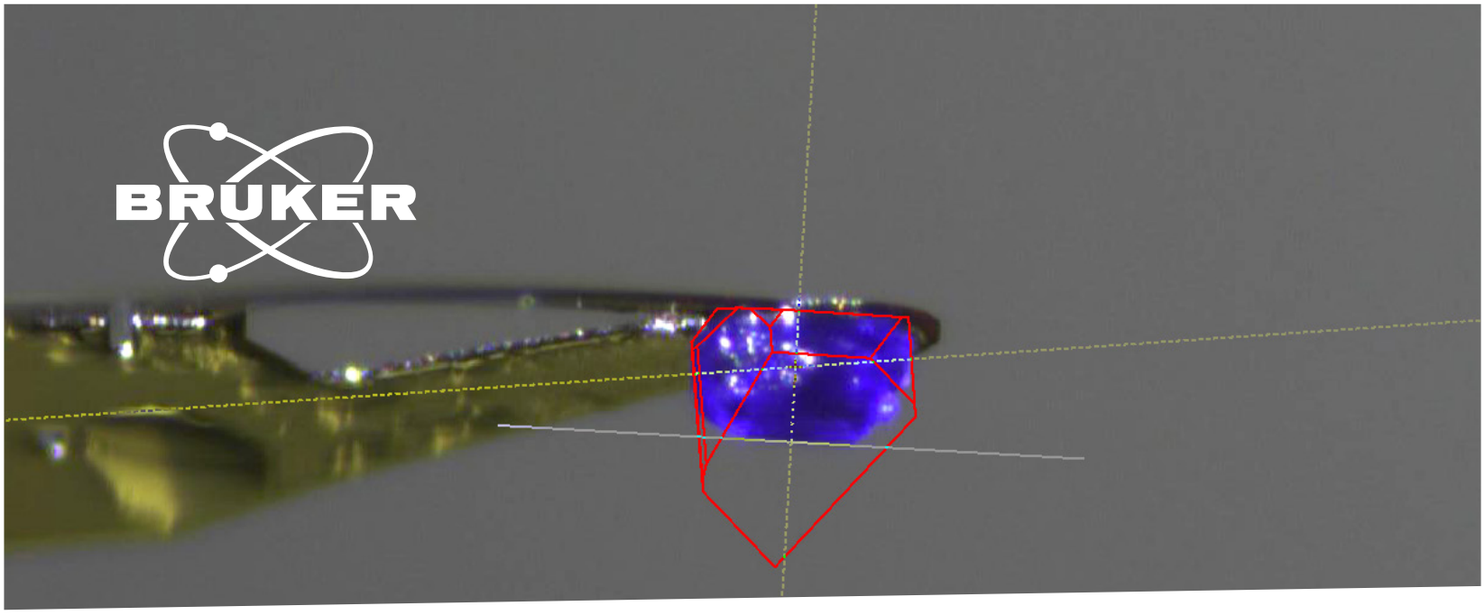

SINGLE CRYSTAL X-RAY DIFFRACTION

Easy and Intuitive: Accurate Numerical Absorption Correction with Crystal Face Indexing

Application Note 374 V2

Introduction

From highly absorbing samples the best achievable data quality greatly depends on an as accurate as possible absorption correction. It is commonly accepted that numerical methods provide the best absorption correction provided the crystal under investigation exhibits a well-defined crystal shape, which allows an accurate description by indexing the crystal's faces.

For face indexing, hkl indices of faces need to be determined and their distances from the center of the crystal measured, thereby describing the crystal's shape by its bounding faces. Traditionally, it was time-consuming to index a specimen with many faces and difficult to determine accurate distances between faces. Therefore, numerical methods were not used as often as empirical methods which were applicable more conveniently.

On the other hand, numerical methods provide the only effective corrections in cases of severe absorption effects. With the crystal's shape defined by its bounding faces, diffraction data can be corrected for absorption effects using numerical methods. From the direction cosines which are provided with the integral intensities, the actual orientation of the crystal for each reflection measured can be determined. The crystal is then divided into small-volume elements. For each reflection, the path length of the incident and diffracted beam through the crystal is calculated for each volume element. Gaussian Integration over all these volume elements gives the reduction of intensity in the form of a transmission factor for each reflection. In the next step, the transmission factors are used to correct each reflection for absorption effects.

APEX4: Face Indexing User Interface

Our modern crystallography solutions are equipped with high-quality video microscopes and highly efficient sample illumination. The combination of excellent hardware with intuitive and easy-to-use software routines in APEX4 has changed the formerly tedious procedure of face indexing into a convenient, simple, and fast method for superb absorption correction. The entire process takes a few minutes only and consists of a small number of intuitive steps:

1. Once the unit cell and orientation matrix for the crystal are determined, the face-indexing plugin automatically collects a 60-second movie from the crystal. Within this time, the crystal is rotated 360 degrees around the instrument's phi axis and 360 images are recorded with the goniometer's axes positions fully encoded.

2. Based on the movie the operator can index the sample, while the instrument can be used for the next measurement.

3. The mouse wheel is used to spin the crystal movie back and forth around the phi axis. If a face of the crystal is viewed "edge on," a line normal to the face appears and a "T-tool" snaps to this line. The operator moves the "T-tool" inwards until it touches the face and, upon a left mouse click, the indexed face and its distance to the center of the crystal are added to a list of face indices. The operator can further rotate the crystal until the next face is edge on and the procedure is repeated until all faces are indexed. As soon as a number of faces sufficient to display a closed polygon have been added, the polygon is overlaid onto the movie and the face indices are displayed.

The face indexing routine comes with a number of helpful tools and options allowing the user to index a crystal within minutes. Display options give color choices for the overlaid polygon and many other configuration parameters, such as font, font size, and line width.

The actual absorption correction is performed in the scaling plugin. In addition to numerical absorption correction, this routine offers the option for empirical multiscan absorption corrections or a combination of both analytical and empirical absorption corrections.

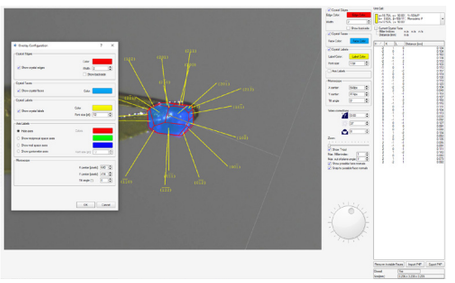

Figure 1
Face indexing plugin in APEX4 – easy and intuitive use. The crystal can be rotated with the "mouse wheel" and faces are added with mouse clicks. The crystal shown is a copper-organic sample with a large number of well-established crystal faces.

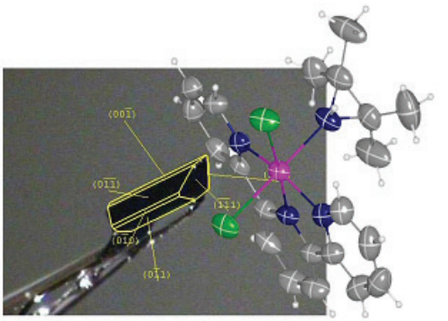

Figure 2
Strongly absorbing ruthenium compound collected with Cu-radiation refines to R1=2.5% against numerically corrected data.
Sample courtesy Bruce Noll, University of Notre Dame

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