ABSORB-7 and *ABSORB-GUI* for single-crystal absorption corrections

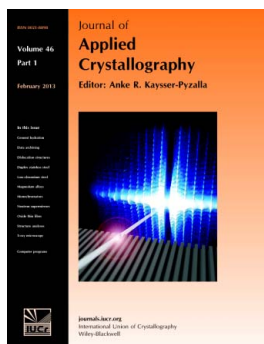**Ross Angel and Javier Gonzalez-Platas**

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computer programs

ABSORB-7 and ABSORB-GUI for single-crystal absorption corrections

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ABSORB is a program to calculate and apply absorption corrections to single-crystal X-ray intensity data from any source, any detector type and any instrument configuration. A new version, ABSORB-7, contains several significant new features. ABSORB-GUI has been developed to allow much easier specification of standard experiments and to enable experimental information to be imported from diffractometer software. The program that calculates the absorption calculations has been reconfigured to allow it to be called directly from other commercial and non-commercial software packages. In addition, a number of new features have been introduced to allow more flexibility in the way the experimental configuration is described and to correct synchrotron diffraction data collected with beams smaller than the sample crystal.

1. Introduction

The correction of single-crystal intensity data for the effects of absorption by the crystal itself and any surrounding materials can be achieved in two fundamentally different ways. One is to use knowledge of the point group symmetry of the crystal to refine a set of correction factors that minimize the internal agreement factor of the symmetry-equivalent reflections after corrections are applied. This empirical approach is perhaps best known in the form of the empirical scaling procedures employed in various data reduction software packages [e.g. DENZO/SCALEPACK (Otwinowski & Minor, 1997), CCP4 SCALA (Evans, 2005), SADABS (Sheldrick, 2003), CrysAlisPro SCALE3 ABSPACK (Agilent Technologies, 2012), XDS XSCALE (Kabsch, 2010)]. The ψ-scan technique (e.g. North et al., 1968) employs the same philosophy but differs in that it minimizes the internal agreement factor of just one or a few reflections collected many times in many different orientations of the crystal in a way that ensures that many path lengths through the crystal are sampled. In both cases the absorption correction is parameterized with a smoothly varying function of either crystal orientation, diffractometer setting angles or detector coordinates (e.g. Blessing, 1995). The empirical approach is routine and very successful for correcting data collected in air from crystals with relatively low absorption coefficients, and has the advantage that it requires very little or no user input or intervention.

However, there are a number of experimental situations for which the empirical method is less effective. As the absorption coefficient or the size of the crystal increases, the effects of the crystal shape become more important and the transmission coefficient no longer varies smoothly as a function of rotation of the sample crystal owing to 'edge' or 'end' effects (e.g. Burnham, 1966; Wuensch & Prewitt, 1965; Sheldrick & Sheldrick, 1970); in such cases representing the absorption corrections as a smooth function of crystal orientation is no longer physically appropriate. Neither is an empirical correction appropriate if the crystal is held within an environmental device such as a pressure cell or cryostat, and the device results either in rapidly changing absorption corrections as a function of crystal rotation or in different correction factors for the same beam path to different points on the crystal. The other drawback of the empirical approaches based solely on the minimization of R_{int} is that they will not necessarily correctly model absorption that has a strong 2θ dependence, for example as generated by the anvils of a diamond-anvil cell (DAC). In these cases the absorption corrections may be more accurate if they are based on a physical representation of the experiment (diffractometer, crystal and environmental device), from which path lengths of the incident and diffracted beams in the various materials can be calculated and the absorption corrections thus determined.

The absorption by the crystal alone can then be calculated by a number of methods that employ direct integration over the crystal volume by dividing it into Howells polyhedra (e.g. de Meulenaer & Tompa, 1965; Blanc et al., 1991). If the absorption of any given beam by a component of an external device, such as a diamond anvil, is the same for all parts of the crystal, the factor due to absorption by this device can simply be applied to the calculated crystal absorption by multiplication (Santoro et al., 1968). However, that is often not the case for crystals in DACs in which, for some reflections, some portion of the crystal may be shadowed by parts of the device. In such cases, the path lengths of each beam through each component of the device as well as the crystal can only be calculated to a grid of points in the crystal. This was the method implemented in the original ABSORB program (Burnham, 1966) for crystals in air. The program was subsequently developed to handle data collected from crystals held in various types of environmental devices, including diamond-anvil pressure cells (Angel, 2004). It can be used to correct single-crystal diffraction data collected with either an area detector or a point detector from any type of diffractometer and with any type of radiation. It handles a variety of data file formats, including SHELX hkl files and incommensurate data. In this paper we describe the significant changes to the ABSORB program that have culminated in the release of version 7. The next section describes the changes that have been introduced into the ABSORB program that allow the user to define more experimental configurations. The reconfiguration of the program to allow it to be called from other software packages is

described in §3, and the features of the new *ABSORB-GUI* follow in §4.

2. Changes and new features

ABSORB-7 incorporates a large number of small changes and new features compared to the version described by Angel (2004). Many of these are trivial but are intended to make the program easier to use. For example, the units for absorption coefficients have been changed to mm^{-1}, and the sizes of DAC components are now in mm simply because this has become the most common set of units used in other software. Similarly, for consistency with the dominant *SHELX hkl* file format for intensity data (Sheldrick, 2008), all structure factor data are now reported in terms of F^2 rather than F. More options have also been provided for handling input data to make the program more compatible with data sets produced by various integration programs. Incommensurate data files can be read, and the user can control explicitly the processing and fate of individual data such as those with negative intensities and/or uncertainties. At the same time we have taken the opportunity to improve the clarity of information provided to the user in the output files and error messages.

Several new options have been provided for describing the crystal, in particular in DACs. Many diffractometer control systems include video capture software that can be used to define the crystal size and shape in terms of the Miller indices of its faces. The previous incompatibility in *ABSORB* of such a specification of the crystal with corrections for a DAC has been removed in *ABSORB-7* by allowing the origin of the coordinate system used to determine the face indices to be specified. While it is normal to set up the DAC so that when the diffractometer angles are zero the DAC is face on to the beam, this is not always possible. In order to accommodate different orientations of the DAC with respect to the diffractometer, a distinction is now made between the sample coordinate system and the φ-axis axial system (Busing & Levy, 1967). If the DAC is face on to the beam when the diffractometer circles are zero then this sample coordinate system coincides with the Busing–Levy φ-axis system. If the DAC is rotated around the φ axis when the diffractometer circles are all at zero, the sample coordinate system does not coincide with the Busing–Levy φ-axis system; the Z axes remain coincident, but the X and Y axes are rotated around the φ axis, with the Y axis remaining parallel to the load axis of the DAC. The crystal is then described on this sample coordinate system as before, and in addition the rotation angle of the DAC is specified to the program.

The normal mode of calculation in *ABSORB* (see Burnham, 1966; Angel, 2004) is based upon the assumption that the incident beam intensity is constant across the crystal and that the beam diameter is therefore much greater than the maximum dimensions of the crystal (and indeed the gasket hole of a DAC). This is true for laboratory X-ray measurements with conventional sealed-tube sources. At synchrotrons it is possible to collimate the beam down to a size much smaller than the crystal and thus avoid generating extra background in the diffraction pattern by diffraction from the gasket or backing plates of the DAC. *ABSORB-7* now makes the corrections for this situation, assuming that the crystal is a flat plate with parallel faces. The relative absorption for each crystal reflection then depends solely on the angles of the incident and diffracted beams to the load axis of the DAC, and the diffracting volume of the crystal depends only on the angle of incidence of the incident beam.

3. Interaction with external programs

The most significant change from the previous version of *ABSORB* is that the program has been reconfigured so that it can be called from any external program. The description of the crystal, the instrument and any environmental device along with instructions for data handling are still provided to the *ABSORB* program through a text file known as the 'experiment file'. This file consists of instructions and information specified by keywords that are completely documented in the user's manual. External programs, such as commercial diffractometer control or data reduction programs, that wish to use *ABSORB* must create this experiment file and then write into it all of the necessary information about the experiment, as well as the instructions for the corrections to be performed. Only relevant instructions need be written, as the program has sensible default settings for all instructions.

The external program then also has to write a 'configuration file' that holds the names, formats and locations of both the experiment file and the data files to be used, after which the external program can run the *ABSORB-7* executable. That program reads the experiment file and performs the absorption corrections on the data, and writes the results to the 'print file' and the 'output data file'. If warnings or errors are generated, these are written into an 'error log file'. In order to avoid system calls in the calling programs, the status of the *ABSORB* program is indicated by the existence or absence of the error log file and a 'lock file'. The calling program has simply to wait until the deletion of the lock file indicates that *ABSORB* has finished, and then has to check if an error log file exists. If it does, it is up to the external program as to how that information is interpreted and whether it is presented to the user. Full details of this interaction, and further details of the *ABSORB* program itself, are provided in the programmer's manual.

4. *ABSORB-GUI*

ABSORB is not just a program for routine data processing; it is also a research and development tool for single-crystal diffraction under non-ambient conditions. Therefore it has a lot of features and modes of operation that are not used for routine experiments. The availability of these many features, and the complexity of possible interactions between them, is often overwhelming for the new user. We have therefore developed a graphical user interface (GUI) that guides the user through the description of the more common experimental configurations for diffraction experiments, and imports as much information as possible directly from files generated by commercial diffractometer control programs. *ABSORB-GUI* interacts with the main *ABSORB* program *via* text files in exactly the same way as other external programs do, as described in §3 above, but this interaction remains hidden unless the user specifically wishes to manually edit the experiment file.

ABSORB-GUI has been created using the Fortran90/95 language with two external libraries: CrysFML (Crystallographic Fortran95 Modules Library, https://forge.ill.eu/projects/show/crysfml) and a Fortran9X GUI toolset library called Winteracter (http://www.winteracter.com). *ABSORB-GUI* provides four dialogs (Diffractometer, Experiment, Crystal Model and DAC; Fig. 1). In the Diffractometer dialog the user provides basic information about the diffractometer, the wavelength and the conventions used in the diffractometer software for describing the orientation matrix of the crystal. The Experiment dialog allows the user to provide the cell parameters or the orientation matrix that indexes the intensity data set. Other options on this dialog allow further controls on which reflections are processed. For example, reflections that are written to the output data file can be limited by the 2θ value, and whether the program keeps or rejects reflections with either negative intensities or negative $\sigma(F^2)$ can be specified.
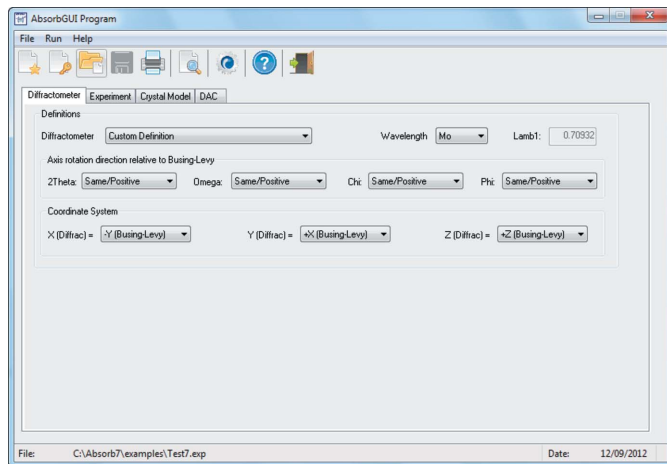
Figure 1
The interface of *ABSORB-GUI*, showing the Diffractometer page. Users can set up their experimental description for absorption corrections by simply working through the four tabs shown.

The corrections to be made for absorption are specified in the last two dialogs. In the Crystal Model dialog, the user can specify the type of model to be used for the description of the crystal. The dialog prompts the user for the information appropriate for the chosen type of model by only providing relevant input boxes. This dialog also allows input of the absorption coefficient, either by specification of the contents of the unit cell (only available for Mo, Cu, Fe, Cr and Co $K\alpha$ radiations) or by the input of the value of the absorption coefficient directly (for example, for synchrotron data). The last dialog guides the user through the description of transmission-geometry DACs, if required. There are two basic ways to describe the absorption by the DAC components; by specifying their sizes and absorption coefficients, or by specifying a measured absorption curve. Both cases are handled by the GUI, which again prompts the user for relevant information through the provision of the relevant input dialogs.

Many diffractometer control or data reduction software packages routinely write a description of the crystal and other parameters of the experimental configuration to a text file (*e.g.* `*.cif_od` from Agilent, `*.p4p` from Bruker or `*.sum` from Stoe systems). *ABSORB-GUI* includes an import utility that reads all of these file formats, extracts all of the relevant information and deduces the conventions necessary for the interpretation of the orientation matrix. The information is then displayed in the dialogs described above. This represents a significant simplification in the workflow for the user, who then only has to review the information and sometimes provide minimal additional information about the experiment in order for the absorption corrections to be calculated.

Once the experiment has been described in the dialogs of *ABSORB-GUI*, the data can be saved from the GUI to an experiment file, and the *ABSORB* program can be run from a dialog that incorporates file browsers to select input and output data files. The provision of a separate 'run' dialog also allows the *ABSORB* program

to be run with pre-existing experiment files and allows the expert user to edit into the experiment file additional instructions not available from the GUI. Both the experiment file and all of the files created by the *ABSORB* program can be viewed by an internal editor by simply clicking on the respective button located in the main toolbar.

5. Availability

ABSORB-7 and *ABSORB-GUI* run under the Windows 2000, Windows NT, Windows XP and Windows 7 operating systems. Both programs are available in a single installation package from http://www.rossangel.net, which includes full documentation in a user's manual, an online help system and example files. A programmer's guide is also provided to describe how to call and interact with *ABSORB-7* from other software. Programmers and users who do not need the GUI can download *ABSORB-7* and the manuals as a zip file without the GUI.

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