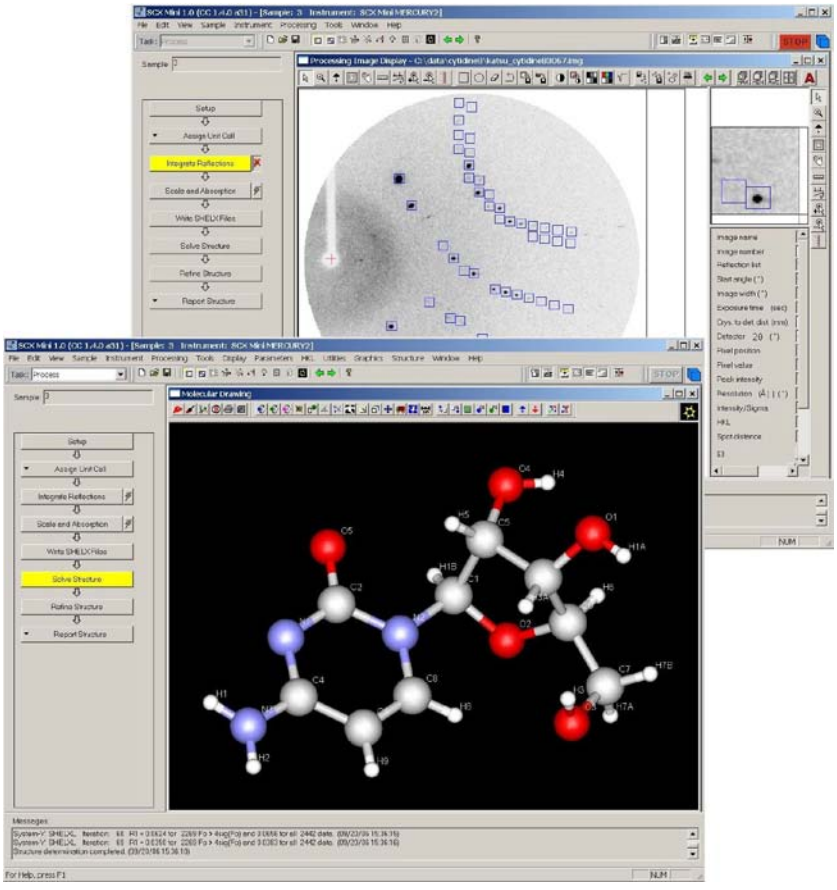
Rigaku

Leading With Innovation

CrystalClear™ 2.0

User Manual

August 31, 2009



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1. Overview

CrystalClear is a program for collecting and processing crystallographic data for Rigaku single crystal detectors and goniometers. CrystalClear has been used for over a decade by more than 500 crystallographers worldwide to successfully collect and process data. Version 2.0 of CrystalClear adds new features and fixes earlier problems.

Supported Hardware

Supported Detectors for Data Collection

Support is provided for all current and many former single crystal instruments produced by Rigaku. These include:

- R-AXIS IV, IV++, and HTC (R-AXIS VII)
- RAPID, RAPID II (SPIDER)
- Mercury, Saturn, Jupiter, and A200 CCDs
- SCXmini and XtalLABmini

CrystalClear supports any of the Rigaku goniometers for these detectors. It also supports any of the Rigaku X-ray generators that are provided with these detectors. The huge number of permutations of detector, goniometers, and X-ray generators supported by CrystalClear is made possible by a flexible Instrument Server and Frame Grabber architecture and a set of carefully designed configuration files.

Image Format Support

In addition to Rigaku detectors, CrystalClear processes data from a large number of 3rd party vendors. These include

- Bruker
- MAR CCD
- MAR IP
- ADSC CCD
- MACScience
- Brandeis CCD
- Pilatus

This allows you to use CrystalClear not only for your home lab data collection and processing, but also for processing data from most beam lines.

Computer Requirements

CrystalClear is installed on a Control computer which interfaces either with the instrument directly, or through a Frame Grabber computer. The Control and Frame Grabber computers must both use Windows® XP. These computers are normally supplied by Rigaku with the instrument. Using this

computer for other purposes, especially those involving heavy computations or high network traffic is not recommended since timing for instrument control can be critical.

CrystalClear can also be installed on additional computers that are not connected to any instruments for the purpose of processing data that has already been collected. These must also be Windows computers, but both XP and Vista can be used for a processing computer.

CrystalClear comes with a dongle that is used for installing licenses. The number of licenses for your site is determined by your purchase agreement. You have a dongle for each license of CrystalClear.

Using this Manual

This manual can be used as an introduction and as a reference. It is mostly task oriented rather than tool oriented. It presents the various features and tools in the program in the context of their normal use and in the order that they are most often used. Once you learn to use these tools in this recommended way you will soon understand them well enough to be able to adapt them to your own style of work.

Earlier versions of CrystalClear had extensive online and interactive help. But this feature was actually used very little in practice and was very time consuming to maintain, so it has been removed. It has proven more effective to spend time designing user interfaces that are easier to use than to maintain online help. The documentation for CrystalClear has been re-organized to focus on a coherent single user manual rather than fragments of online help. If you click the **?** icon in the main toolbar or on **Help Topics** under **Help**, this manual opens as a pdf file using Acrobat Reader. You can use the Acrobat indexing to navigate to the relevant section.

Chapter Outline

1. **Overview**.
2. **Installation and Administration**. This is primarily for the user who is responsible for installing and updating CrystalClear for your site. It covers the procedures for installation and for configuring various files to reflect the actual instrument setup. Version 2.0 has a different directory structure, so updating to 2.0 is a bit more complex than previous updates. Even if you have upgraded CrystalClear earlier (e.g., from 1.3.6 to 1.4.0) it is worth referring to this Chapter as you update to 2.0. This chapter also covers administration functions such as setting up new users.
3. **Getting Started**. As in any complex computer program, it is important to understand a few fundamental concepts in order to effectively use CrystalClear. If you don't read anything else in this manual, you should read Chapter 3.
4. **The Toolbar and Views**. The main window tool bar provides access to the most common functions. CrystalClear provides a set of viewers to allow you to examine the data images, the state of the hardware, the cell and other processing information, etc. These views usually pop up when they are relevant to the task at hand, but they can also be selected via the **Windows** menu. Since they are used in many different tasks, we will illustrate their use first so that we can concentrate on the flow of the task itself.
5. **Small Molecule Tasks.** For small molecule work you have the option of running in **Auto** mode where the entire process of collecting and processing data is highly automated or in **Expert** mode where you have detail level control as you work through the process manually. In addition, in Expert mode, you have the choice between d*TREK, FS_PROCESS, and TwinSolve. You may safely skip this chapter if you are a macromolecular user.
6. **Macromolecular Tasks.** For protein and other macromolecules, you can either process the data with d*TREK or FS_PROCESS. The flow bars and approaches associated with each of these

tasks are described here. The individual steps are described in chapters 8 through 11. You may skip this chapter if you just work on small molecules.

7. **Collection Steps**. This chapter concentrates on the process of collecting data. The collection steps are the same regardless of the processing suite and very similar for small molecule and macromolecule work. **Strategy**, which might be considered a collection step, is described in the next chapter since it is only provided if d*TREK is the processing suite and it is intimately tied in with indexing and other processing steps.
8. **Processing Steps with d*TREK**. You can either process data as it is collected or wait and process it later. This chapter covers both approaches and explain the steps provided by CrystalClear when you have selected the d*TREK processing suite to integrate, scale, and analyze your data.
9. **Processing Steps with Fine Slice Process**. The chapter describes the processing steps provided by FS_PROCESS. They are similar to those provided by d*TREK, but there are differences both in the flow and the parameters.
10. **Processing Steps with TwinSolve**. The chapter describes the processing steps provided by TwinSolve. TwinSolve requires a separate license and is only applicable in Small Molecule Expert mode.
11. **Reciprocal Lattice Viewer**. This chapter describes the Reciprocal Lattice viewer which can be used to examine reflections in reciprocal space and provides tools for manual indexing.
12. **Tools**. This chapter covers menu accessible tools that are provided by CrystalClear for specialized functions. If you can't find what you need in the previous chapters, you will probably find it here.
13. **FAQs**. This chapter has a list of Frequently Asked Questions.

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2. Installation and Administration

Most of the information in this chapter is intended for the CrystalClear administrator. If you are the administrator or a joint administrator and are doing software updates, adding users, etc., then you should read the whole chapter.

CrystalClear has its own definition of users and their privileges which is independent of Windows. Everyone in your lab can use the same Windows account to log on the Control computer and you can still set up separate CrystalClear users. Conversely, you could all have separate Windows accounts but run as the same CrystalClear user. Generally, since the login screen defaults to the Windows user name, it is most convenient if you set up CrystalClear users to match the Windows users.

Similarly, a user who has administrative privileges in Windows need not have administrative privileges in CrystalClear and vice versa. The directory structure for CrystalClear has been modified significantly starting in version 2.0 to allow users to run without Windows administrative privileges. In newer versions of Windows this means moving any file that can be created or updated while running CrystalClear (e.g., log files, configuration files, etc) out of C:\Program Files. This is described in more detail in the section on **File Structures**.

Installation

Downloading the Installer

You will have been given a link with a user name and password for the CrystalClear installer. Once you log into this web site, find the link to download the installer. Simply click this link and the installation downloads to your computer. This is a large file (more than 80 MB) so this may take a while.

If the computer on which you are going to install or update CrystalClear is not on the net, then you can download the CrystalClear installer onto another PC that is on the net and then copy the installation file to a USB memory stick or some other medium and carry it by hand to your target computer.

Running the Installer

For CrystalClear 2.0, the released version of the CrystalClear installation file is called CrystalClear2.0.0r1. If the file name is something like CrystalClear2.0.0b7, then it is a beta version and if it is something like CrystalClear2.0.0rc4 it is a release candidate.

You must have Windows administrative privileges to run the installer.

To run the installer, simply double click this installation file. Depending on your protection schemes, Windows or your anti-virus software may want you to confirm that you really do want to install this software.

New Installations or Major Version Upgrades

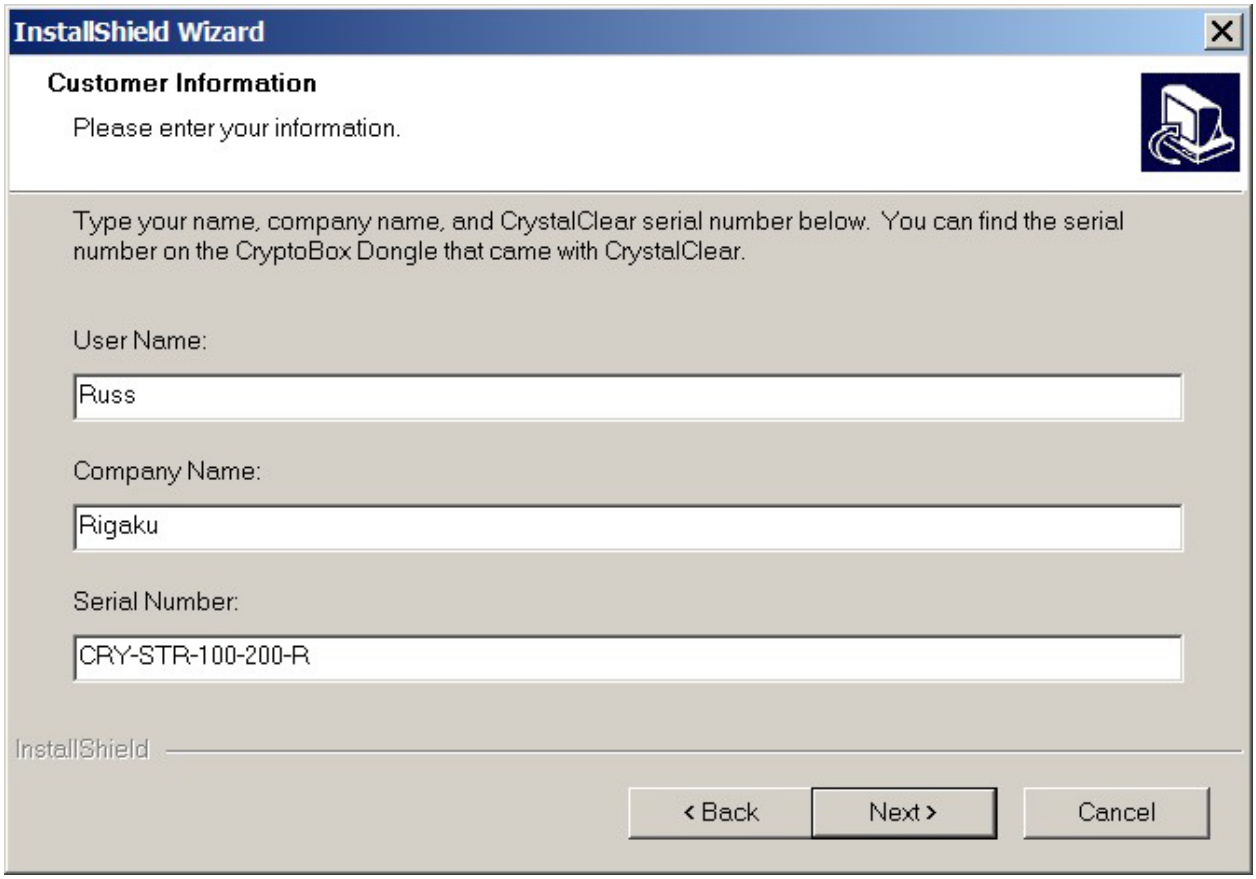
First Installation

Major new versions (e.g., CrystalClear 2.0) are treated as different programs in the installation process. So installing a major version is similar to a new installation even if an older version of CrystalClear has

been installed. However even for major version updates the installer looks for previous versions of CrystalClear and uses as much information (users, configuration, etc) as it can find.

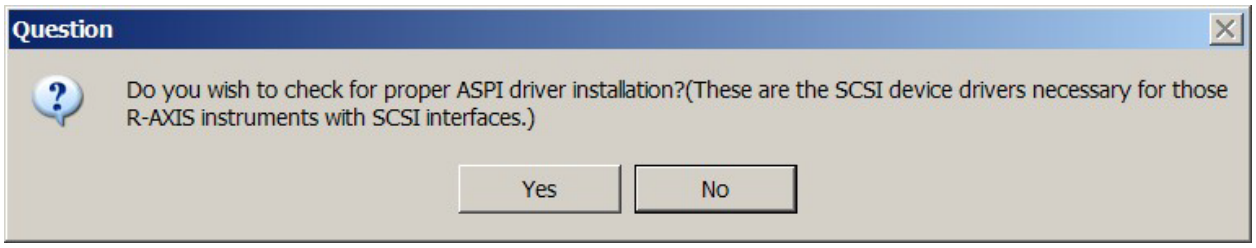
If this is the first time that any CrystalClear or a major new version has been installed on this computer, the installer starts walking you through a wizard to complete the installation. In general it is best to accept the defaults for the installation directory and other defaults, so you can just click through **Next** and then **Finish** in the final screen.

You have to enter you identification and dongle ID even if this is a major version update.



If you see a serial number (i.e. Dongle ID) that does not match the one on your dongle itself, you may need to run the old version of CrystalClear and go to **Edit|Set Dongle ID**. This brings up a dialog with the current dongle ID. Make a note of this and then click **Cancel**.

There are some post installation processes that run after the **Finish** screen. For example:



Click **Yes** if you have an R-AXIS with a SCSI interface, otherwise click **No**. There can be other processes that run at this time to install system dlls, etc.

It is important that you wait for the post installation processes to complete and that you do not do anything else on your computer until they do. It could take several minutes. Also, the USB dongle driver installation occasionally hangs. When this happens stop CBSetup.exe with the task manager and the installation usually completes successfully after that.

Dongle Installation

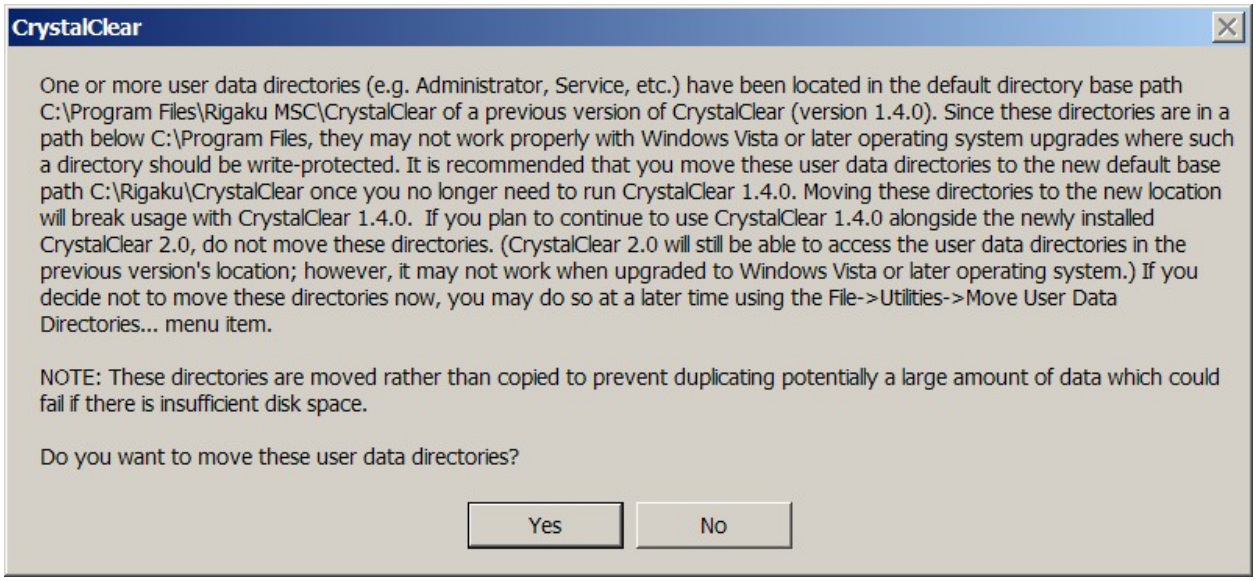
CrystalClear requires a dongle. If you are doing a new installation, this is a USB dongle. For an old system you might have a parallel port dongle. If you are replacing your computer and have a parallel port dongle on the old computer, contact Rigaku about replacing it. The driver for the USB dongle is installed during the CrystalClear installation process. If there are problems and you need to re-install, this file is in C:\Rigaku\CrystalClear20\CBSetup.exe.

Under normal circumstances you just have to plug your dongle into an available USB 2 port.

Special Issues for CrystalClear 2.0 Updates

Between CrystalClear 1.4.0 and 2.0 there were major changes in the directory structure (see Directory Structure section) that were necessitated by the evolving security requirements in Windows XP and Windows Vista. This meant that everything had to be moved out of C:\Program Files. Rigaku has made every effort to automate this process back to CrystalClear 1.3.5. If you are updating an older version of CrystalClear or discover that your configuration is not updated automatically as it should be, please contact your Rigaku support person.

There is an additional complication if any of your users have their **User Data Directory** (the root directory for all their projects and samples) in Program Files. This was a default for the Administrator account in earlier CrystalClear versions. These should eventually be moved out of program files if you are ever going to update your operating system or want to run as other than a Windows administrator. You cannot just move these files with Windows Explorer since they contain full file paths that need to be updated when the data is moved. If CrystalClear detects that you have users in Program Files, it brings up a message:



If you click **Yes** all user data directories in Program Files are moved to **C:\Rigaku\CrystalClear**. If you do not want to move the data at this time, you should click **No**. You can always go back to **File>Utilities>Move User Data Directories** to move them. However, at that stage you are already logged in as a user which opens some of the files that need to be moved. Windows does not allow you to move open files, so you have to log in as a different user to move your user data directory. You may have to create a temporary user with administrator privileges to make the move at a later time (or get your CrystalClear administrator to do it).

First Run After a New Installation

When you click CrystalClear you see a splash screen which asks you for a user name and password. After installation of the first version of CrystalClear there is an **Administrator** user with no password. You

have to log on under Administrator the first time. Once you get in, you can setup new users and modify user accounts as explained in the **User Administration** section. If this is a major version upgrade, all your old users are still there.

If this is the first installation or a major version upgrade of CrystalClear you are also asked questions about the instrument you are using and whether you are running mostly small molecule or macromolecule samples. Only those files that are needed for your site are loaded into the installation directory. If this is an upgrade, the installer makes every effort to find your configuration files from a previous version.

Reverting to an Old Version

Once the defaults are updated, you are ready to run under the new version. However, if you need to revert to an old version, it still works. Note however, that if you have moved the User Data Directory you are not able to run the older version of CrystalClear. This is one reason that you may not want delay moving the User Data Directory until you are sure you no longer need to run the old version.

Updating CrystalClear to a New Minor Version

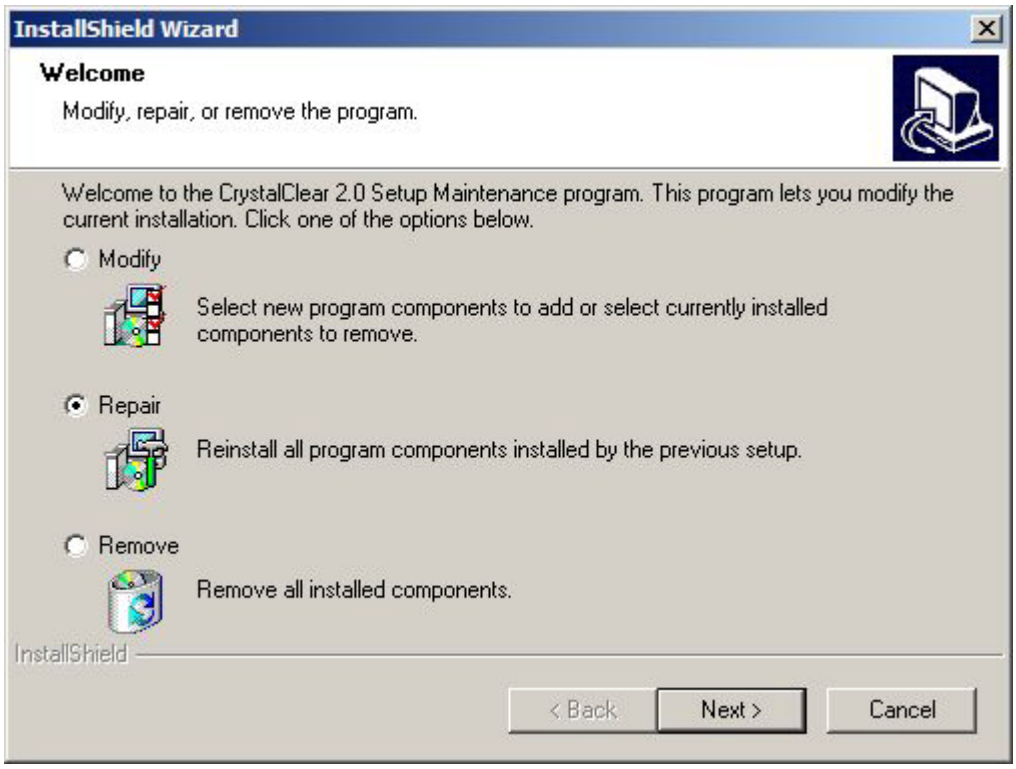
Installing Updates

Installing a minor version update is generally very simple, but still requires Windows administrative privilege. The recommended first step is to copy the following directories into some other location as a backup in case you have to recover.

- C:\Rigaku\CrystalClear
- C:\Rigaku\CrystalClear20 (or whatever the current version is)
- C:\Rigaku\Config

In general you should not uninstall CrystalClear nor delete any of the files in C:\Rigaku. If you do this you could lose a lot of information about your configuration, users, projects, samples etc. However, if you must do this, refer to the following section on uninstalling and reinstalling CrystalClear.

When you double click the CrystalClear installer, you are presented with the following screen:



Make sure **Repair** has been selected and then click **Next** on the wizard screens. Even though this is an update, the CrystalClear installer still runs several processes after the final **Finish**. So it is important that you wait for these to complete and do nothing else on your computer until they have.

When you update CrystalClear your user information, projects, samples, and settings are not overwritten.

First Run after a Version Update

CrystalClear defaults usually change between versions. Even though none of the existing parameter defaults may have changed, there are often new parameters that require a default. When CrystalClear runs the first time after an update, it usually asks you if you want to update the defaults and bring up the **Default Manager** described in Chapter 3. Unless you have modified the defaults extensively, it is a good idea to accept these new defaults and apply them to your site, users, and projects. It is not usually necessary or wise to apply the defaults to individual samples.

If you are an administrative user, you are allowed to apply these defaults to other users, but they have the chance to review this decision for themselves when they first log on after an update.

Uninstalling CrystalClear

Uninstalling and then re-installing CrystalClear is not generally recommended. It is much better just to use the **Repair** option. If you must uninstall CrystalClear, you can either double click the Installer and then select **Remove** or use **Add/Delete Programs** in the Windows **Control Panel**. If you want to save configuration setup and user information, you need to make copies of some of the directories in C:\Rigaku in order to restore your settings and users. You should copy:

- C:\Rigaku\Config
- C:\Rigaku\CrystalClear\Administration

Once you have re-installed CrystalClear, you can copy the saved versions of these directories into C:\Rigaku to restore your configuration settings and restore your users.

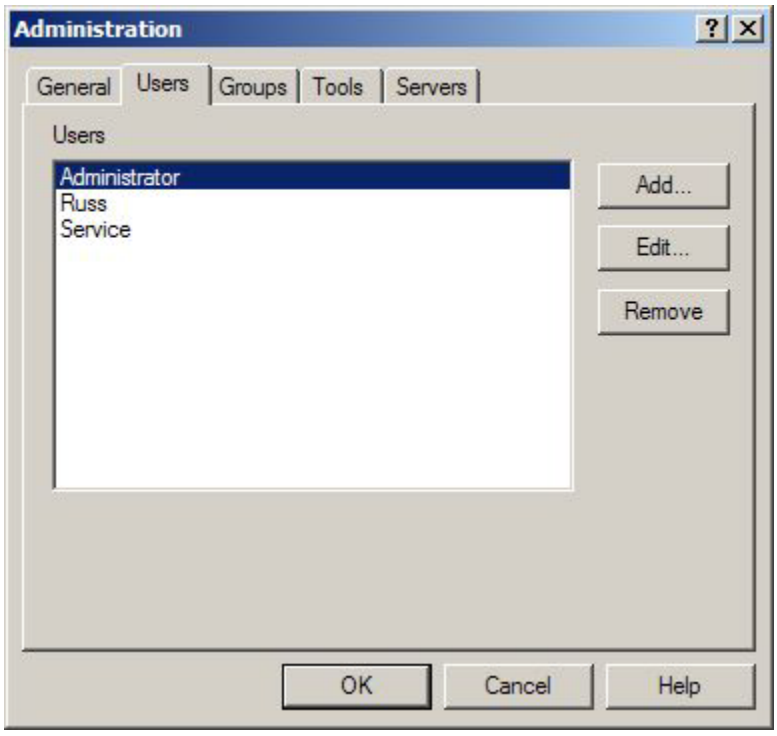
User Administration

CrystalClear provides support for separate user accounts each with their own data directory, defaults, preferences, projects, and samples. A user without administrative privileges can only modify their own account settings. Administrative users can add new users and modify all user accounts.

Creating and Editing User Accounts

To setup or modify a user account select **Tools>Administration** and click the **Users** tab. The other tabs in the **Administration** dialog are rarely used and should only be changed as instructed by Rigaku support personnel.

Administration Users Tab

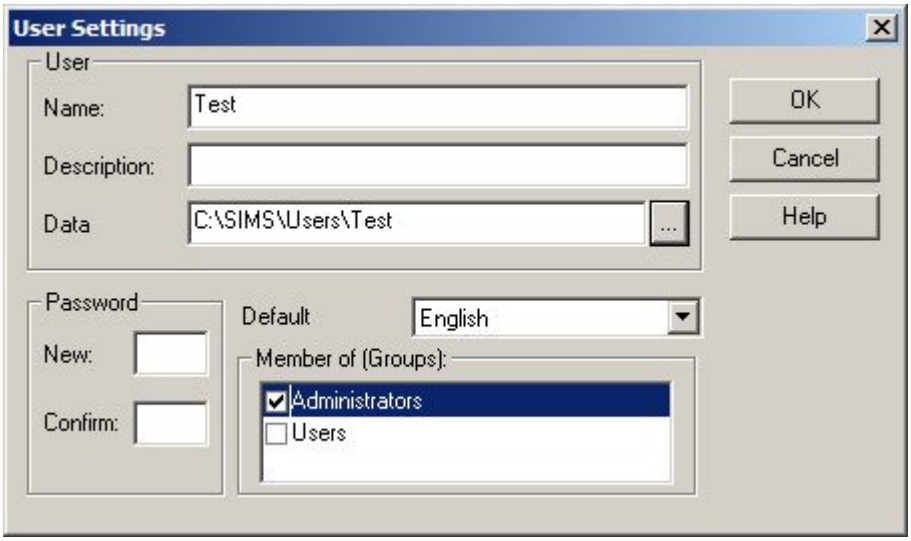


Parameter	Description
Users	A list of the current users is selected. Click the user if you wish to edit or remove them. If you are not an Administrative user, you can edit only your own account and you cannot add new accounts.
Add	This brings up the dialog **User Settings** shown below and then adds the new user.
Edit	This also brings up the **User Settings** dialog but only the user name is read and the existing user account is updated.
Remove	Deletes the selected user. You are asked to confirm the deletion of this user. You are also informed that the files associated with this user have not been deleted and given the path for doing it yourself. This is to prevent accidental deletion of important data. Note that if your image files are not in a subdirectory of the Sample, they are not deleted.

Parameter	Description
OK, Cancel	Both of these simply close the dialog. **Cancel** does not restore the previous state (i.e., it is not an "undo").

User Settings Dialog

This dialog comes up when you click Add or Edit in the Users tab.



Parameter	Description
Name	The user name. It cannot contain spaces. Names are not case sensitive.
Description	This is redisplayed when this user account is edited. It is for record keeping purposes only and not otherwise used.
Data	The **User Data Directory** is the root directory for all of this user's information. If the user information is to be shared (e.g., to be able to run CrystalClear on another computer to process data etc) it is best to put this on a shared drive or shared directory.
Password	Passwords are case sensitive. Unless you have an environment where you are worried about security it is usually not necessary to supply a password.
Default	At this time there is only an English version of CrystalClear at this time, so leave this as English.
Member of (Groups)	Check Administrators if this user is to have administrative privileges. For non-administrative users check **Users** and uncheck **Administrators**. There is a way to create new groups with the **Groups** tab, but it has not been fully developed and should not be used.

After setting up a new user account, click OK, log out of CrystalClear and finally log back in as the new users.

First Run as a New User

The first time you run as a new user you are asked if you are connected to a real instrument or wish to run in simulation mode. If this is the control computer connected to the instrument, you should check **Real**. If you are running CrystalClear on another computer that is not connected to an instrument but will be used

for processing data, then you should click **Simulator**. These settings can be changed later in the **Server** tab to the Preference dialog (see Chapter 12 **Preferences**).

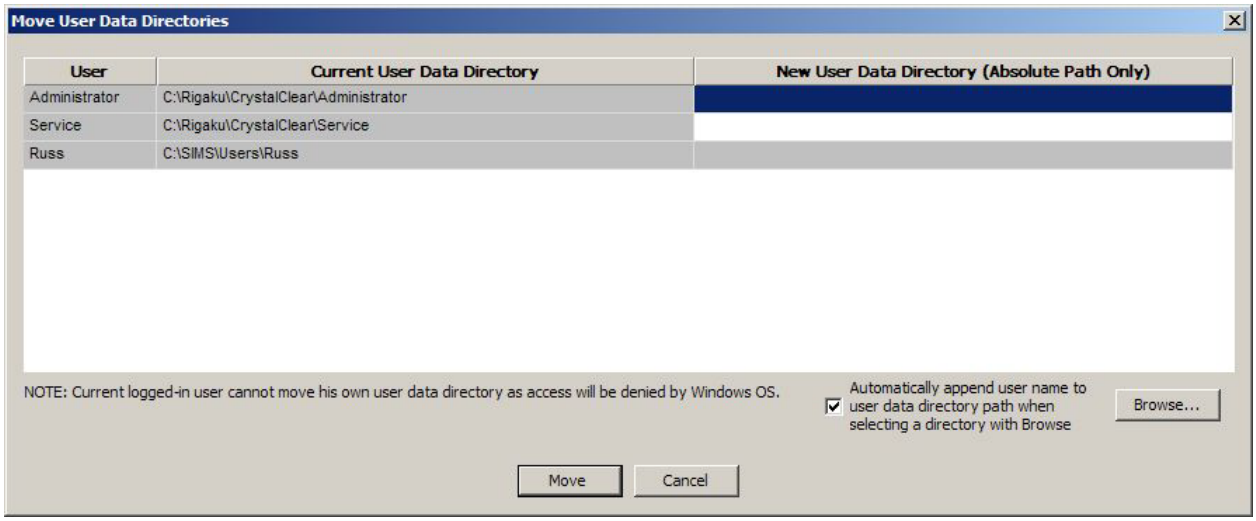
Moving User Data Directories

All user information, projects, and samples are stored in the **User Data Directory** and its subdirectories. Since some of these files contain absolute references to other files, you can't just copy them to another directory without breaking these links and causing CrystalClear to crash. But starting with CrystalClear 2.0, you can select **File>Utilities>Move User Data Directory** to move the whole user data directory and automatically repair all links. If you are moving the directory to a different disk, then Windows interprets a move as a copy and you get a copy, but if it is on the same disk then the operation is a move rather than a copy and you do not get copies when you are done. This is an important consideration if you have a lot of data.

In early versions of CrystalClear the Administrator **User Data Directory** defaulted to a subdirectory of C:\Program Files. Newer versions of Windows XP and all versions of Vista do not like data to be stored in Program Files which is normally write protected for non-administrative users and even for some administrative access. So it is strongly suggested that you move any user data directories you have in program files to somewhere else when you install a new version of CrystalClear. If you have administrative privileges (in CrystalClear not Windows) you can also move other users.

Once you log into CrystalClear as a user, some of the files in the **User Data Directory** are opened for updating as you do your work. This prevents them from being moved. So you either need to log in as another user with administrative privileges or get your CrystalClear system administrator to do this for you. If you want to move the Administrator user and you do not have another use with administrative privileges you need to create a new account with administrative privileges and then log into that account to move the Administrator account.

Move User Data Directories Dialog



Parameter	Description
User Table	This is a table with a list of all the current **Users** (if you have CrystalClear administrator privileges). It shows the **Current User Data Directory** for each user and provides a column to enter a **New User Data Directory**. All User Data Directories are full, absolute paths. You can select the user you want to move and then either enter the new directory path or click **Browse**…
Browse…	This brings up a directory browser. If you check **Automatically append user**

Parameter	Description
	name to user data directory path when selecting a directory with Browse, the current user name is added to the directory you select. This is useful if you want to put all users under a common directory.
Move	Once you have filled out the table, click **Move**. Any user that does not have a new directory path in the **New User Data Directory** is not changed. Since quite a few files have to be updated if you have a lot of projects and samples, this process may take some time.

Directory Structure

Starting with CrystalClear 2.0, there is a new directory structure.

C:\Rigaku – All Rigaku software will eventually be in this directory.

ASPI – Only used for SCSI R-AXIS systems

Config – Contains configuration file for all Rigaku applications. Some of the config files have been renamed (see Configuration Files below). This only contains configuration files for your instrument type.

CrystalClear – Contains shared information that all versions of CrystalClear need.

Administration – Contains the administration database with the user information.

Administrator – The Administrator user data directory is typically kept here. If you are going to use it to actually collect data other than just for testing, it may be good to move it outside C:\Rigaku with the Move Directories command.

Data – Site defaults

SiteDefaults – Current site defaults.

CrystalClear20 – This is the installation directory and contains the programs executables, dlls, and other files needed to run CrystalClear. A different version of CrystalClear (e.g., 2.1) has a different installation directory (e.g., CrystalClear21).

Factory – All the factory configuration and default files are kept here. You should not generally modify anything in this directory.

Mask Files –Current mask files

Process/Database –This has various data files used by FS_PROCESS.

TwinSolve – The error text files and other files needed by TwinSolve are installed here.

Doc – This manual and various other documents are kept in this directory. This includes html files which document the various configuration files.

Log – The CrystalClear and instrument server logs are stored here. However, the html versions are now in **Web**. Some log files are still kept in the sample or image directory for each sample.

RAXWish – These directories contain the code and support files for RAXVideo and RAXShape.

Scripts – Not currently used.

Web – The html version of log files are kept here unless you have set another path in your preferences.

Configuration Files

In CrystalClear 2.0 and above, the configuration files are in the C:\Rigaku\Config directory. For most of these files, there is a corresponding html file in the C:\Rigaku\Doc directory that gives you the details you need for maintaining these files. These configuration files are critical to the operation of your system, so you should only change them if you know exactly what you are doing or have been instructed to make the change by your Rigaku support person.

Only some files should be edited with a text editor such as Notepad or Wordpad. Others are maintained through the user interface. Write the files as simple text files with no formatting information and remember to save before you run CrystalClear again. You need to restart CrystalClear for the change to become effective.

The list of files below is an example with AFC-11 with a Saturn 724 CCD so that we can use actual file names. If you have a different instrument the names change to reflect that instrument.

system.configuration. This file contains the IP address and the port for the Frame Grabber PC for CCD systems.

CrystalClear.configuration. This has information about the mode (macro, small molecule expert, small molecule auto) and configuration information about JXG for managing the X-ray generator and RMailer for sending out emails when there is a failure or a data collection finishes.

CrystalClear_Instrument.configuration. To update this file, log in as an administrative user and go to the Setup dialog and select the X-Ray Source Tab. The **Save to Config** updates this file with information about the X-Ray source. (In certain circumstances for small molecule Auto mode there may also be a sm_xxx_instrument.configuration that is used instead of this file.) This file should not be edited manually.

MSCServDetCCD.configuration. This has information that the instrument server needs about the goniometer type, overload handling, trigger delays, wavelengths etc. There are RAXIS, RAPID, and RAXISHTC versions of this file if you have an image plate instrument.

AFC11_RCD3.configuration. This has the port for connecting to the goniometer controller (RCD3) and datuming and limit information.

AFC11Saturn24.xml. This has a link to the collision files that are usually kept in the Factory directory. You should not normally modify this file nor the collision files themselves.

RAXVideo.configuration. If you have a video camera and capture card and use RAXVideo, this file stores the RAXVideo version. The version can be set in CrystalClear by going to the Instrument>RAXVideo Version menu.

Director.configuration. This is only used if you have an ACTOR system and are using Director.

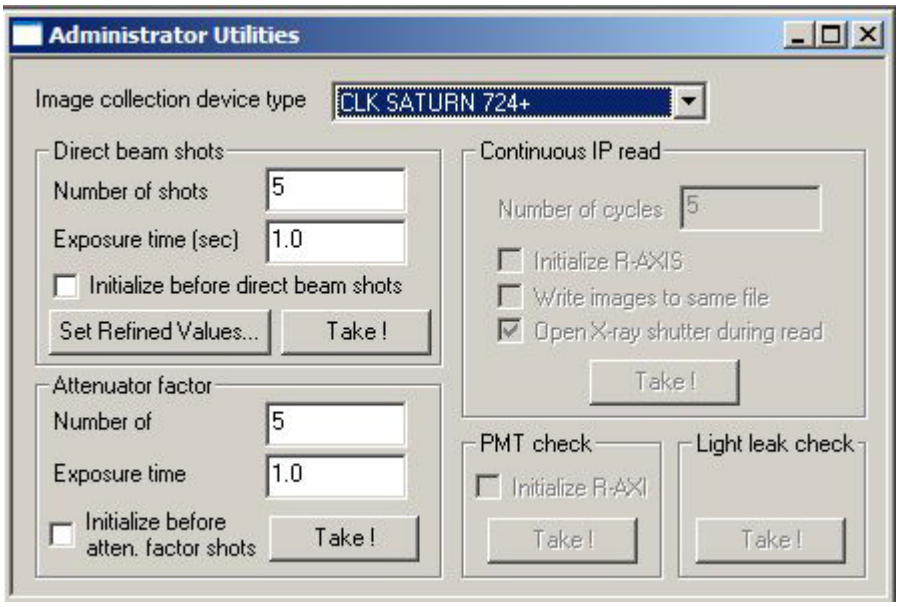
Instrument Setup and Maintenance

To modify the instrument type (rare) or take direct beam shots use the following function from the main menu.

Instrument>Administrator Utilities

The administrator utilizes are for setting up the instrument type and performing certain maintenance functions. They require administrator privileges.

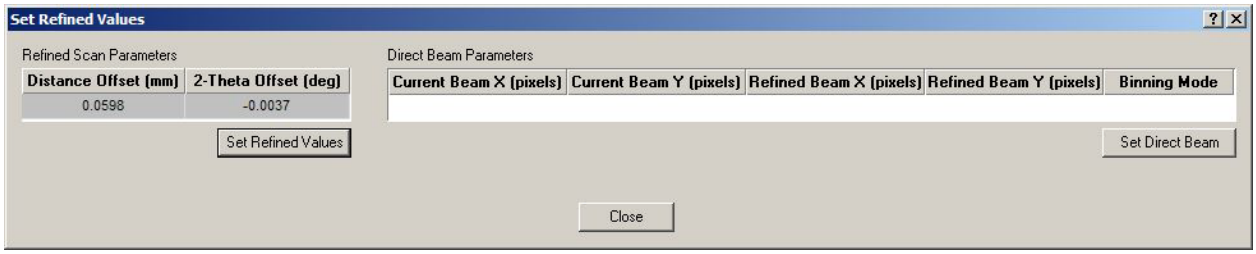
Administrator Utilities View



Parameter	Description
Image collection device type	This is normally setup for you by your Rigaku installation engineer and you seldom need to change it. But if you do, there is a drop down list of all the many available instrument types that CrystalClear recognizes. Note that you do **not** need to change your instrument type in order to **process** images from a different instrument (even a non-Rigaku instrument) since the information for processing is derived from the image itself. If you do change the instrument type you need to restart CrystalClear.
Director beam shots	This is the area of the view that you use to take direct beam shots. You should be trained by Rigaku service personnel or a Rigaku application scientist in how to take these images. The parameters for this are: • **Number of shots**. How many direct beam images to collect. • **Exposure time**. The exposure time for each direct beam shot in seconds. • **Initialize before direct beam shots**. This check box forces an initialization of the instrument when you click **Take!** and before the direct beam images are collected. • **Set Refined Values**… This is a new feature in CrystalClear 2.0 and above that allows you to update the Distance, 2 Theta and direct beam with refined values (e.g., from indexing and refining a known crystal). This can be especially useful for the SCXmini and XtalLABmini to update these refined values. There is a dialog that pops up which allows you to do this that is described below. • **Take!** This activates the collection of the direct beam.
Attenuation Factor	This activates the collection of the images used to calculate an attenuation factor for a given attenuator. • **Number of shots**. How many attenuation images to collect. • **Exposure time**. The exposure time for each attenuation image.

Parameter	Description
	• **Initialize before atten factor shots**. Checking this causes the instrument to be initialized when you click **Take!** • **Take!** Starts the collection of attenuation factor images.
Continuous IP read	This is a maintenance function for R-AXIS image plate detectors. It is disabled for CCDs. • **Number of cycles**. How many times to repeat the read • **Initialize R-AXIS**. Check to initialize before the reads start. • **Write images to the same file**. If this is checked all the images go into a single file. • **Open X-ray shutter during read**. • **Take**! Starts the continuous read.
PMT Check	The R-AXIS has two photo multipliers and they have to be very carefully aligned. This section is used to run a diagnostic on the PMTs. • **Initialize R-AXIS**. Check to initialize before the PMT test starts • **Take**! Run the PMT test.
Light Leak Check	This is another R-AXIS diagnostic check. • **Take**. Run the check.

Set Refined Values Dialog



Parameter	Description
Refined Scan Parameters	Before you click Set Refined Values in the Administrator Utilities View you should have collected data and processed data on a high quality crystal with well known cell parameters with the options checked to refine the Distance, 2 theta, and Direct Beam position. If you have done that, these values are automatically entered into the **Distance Offset** and **2-Theta** Offset fields. You can then click **Set Refined Values** to set the distance and 2 theta offsets.
Direct Beam Parameters	Similarly after you have run the refinement, the direct beam parameters in this table are updated. You should review these to make sure they are reasonable and then click **Set Direct Beam**.

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3. Getting Started

This chapter tells you how to start up CrystalClear and explains the central concepts that are necessary to understand in order to use CrystalClear effectively. If you are new to CrystalClear, this is the most important chapter in the manual for you to read.

Starting CrystalClear

Icons

When CrystalClear is installed it puts an icon on the desktop. To start CrystalClear simply double click this icon.

Login Screen

When CrystalClear starts up it presents a login screen as follows

The Login Name defaults to the current Windows user. If this is the first time you have run CrystalClear after a new installation, the only user is Administrator and there is no password. If your Login Name has not yet been setup, then you should login as Administrator. Login Names are not case sensitive, but passwords are.

Once you click OK, CrystalClear should start up. See the instructions in Chapter 2 if you need to add a new user or edit your user setup.

Users, Projects, and Samples

CrystalClear organizes the data collection and processing into a hierarchy. Each user has a collection of projects and each project has a collection of samples. Understanding this user/project/sample hierarchy and how it affects defaults is vital to using CrystalClear effectively. If you understand and use projects and samples they can provide a flexible and useful way of organizing your data.

Users

When you log into CrystalClear you specify a user name. The procedure for creating users and editing user information is described in Chapter 2. Each user has a User Data Directory. This is the root directory for all of the data that CrystalClear maintains for this user. The exception to this rule is that images can optionally be stored in an image directory that can be anywhere on disk. The User Data Directory contains:

- User preferences (.ho and .xml). These are preference for display options and other program features that are stored for each user. The8 can be set in the Preferences Dialog described in the chapter 12 on Tools.
- User defaults (.ho and .xml). These are the default parameters for the various dialogs that correspond to steps in CrystalClear (e.g., Index, or Collect). When the user is created, the site defaults are copied into the User Data Directory and become the user defaults. These defaults can then be updated by the user by using the Save button provided on most dialogs. Note that since you copy rather than refer to the site level defaults, changing the site level defaults does not update the user defaults. However, a default update mechanism described later in this chapter allows you to update selected levels of your defaults at the same time.
- Project List (.ho and .xml). These are the projects that have been created for this user.
- Project Directories.

For the examples in this section we assume that the User Data Directory is C:\UserData\Russ, but of course in practice it can be any accessible directory.

Projects

A project is a collection of samples that are related in some way. Each project has a name. The name must be unique for the current user. A directory is created under the User Data Directory that corresponds to this project. For example if the project name is HSP90, then the directory in our example is C:\UserData\Russ\HSP90. This directory file contains the sample directories and a set of default and other files with .ho or .xml extensions.

Project Directories contain:

- Project defaults (.ho and .xml). These are the default dialog parameters for this project. When the project is created, the user defaults are copied into this directory as the project defaults. Since they are inherited at this point, changing the user defaults does not change the project defaults.
- Sample List (.ho and .xml). The samples in this project

Samples

A sample generally corresponds to a physical sample crystal. A sample also has a name and a directory is created to store the log files, reflection files, and other data relevant to the sample. For example if the sample name were Soak0246, then the directory in our example would be:

C:\UserData\Russ\HSP90\Soak0246.

The sample directory by default includes an Images subdirectory where the images are stored, but it is possible to specify that the images are stored in a separate directory anywhere accessible to the control computer. For Process only samples you set the image directory to refer to the already collected image files.

Sample directories contain:

- Sample files (<sample>_20.ho and .xml). This is sometimes referred to as the sample database. This file contains the current status, dialog parameter defaults, and a history of the sample. Older versions of this file are also retained. If you install a new version of CrystalClear and reprocess the data you get new versions of the <sample>_xx.ho file and the old files are maintained. This allows you to revert to an older version of CrystalClear and still access this sample.
- Log files for d*TREK , FS_PROCESS, or TwinSolve. These are the logs that are created by indexing, refinement, and other processing steps.
- Header (.head and .dat) files. These are used by d*TREK or FS_PROCESS to pass sample information from one step to the next (e.g., from index to refine).
- Reflection and CIF files. These are the output files that are used for structure determination and refinement.

The images directory may or may not be included in the sample directory. In either case the image directory contains:

- Image (.img or .osc) files. These are your data images.
- Collision.xml File. The collision file used to prevent goniometers collisions while collecting data.
- Instrument Server log file. This can be useful in diagnosing any problems and your Rigaku support person may ask for this file.

Each time you mount a new crystal, you should create a new sample to hold the data associated with that crystal. The normal and default practice is to store the images you collect in the Images subdirectory of this sample.

If you need to screen a lot of samples to find one that you can collect, you can also open up a sample and then just add a screen scan for each new crystal changing the image name template each time. Then when you find a good crystal you can go on with the sample.

You can reopen a sample at a later time after the data has been collected and reprocess some or all of the data. The complete state and history information of the sample is maintained in the various files, so that nothing is lost by stopping CrystalClear and restarting at some later time.

Process Only Samples

It is sometimes useful to process the data in a sample without reopening the original sample. For example, if you have processed a data set with d*TREK and suspect that it is twinned, you should normally open up a process only sample for the TwinSolve processing. Or you may want to process the data on a different computer which has its own installation of CrystalClear and its own directory structure for each user. When you open a process only sample, you are asked to browse to where the data images are kept. This can be on an external drive, but you need to make sure the drive stays accessible during the time you have the sample open.

Sample names must be unique to the project, so if you open a process only sample in the same project as the original sample, give it a different name.

The Flow Bar

Each sample has an associated task. In order to explain what these tasks mean, you must first understand how the flow bar works.

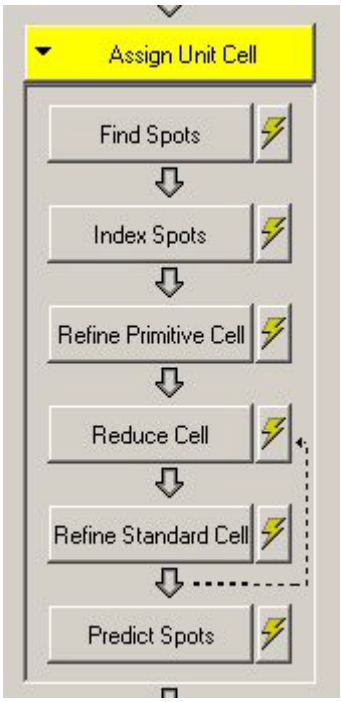
There are various ways to navigate to a particular step and it associated dialog in CrystalClear. Almost all steps are accessible through the menus. Some of these steps are only accessible through the menus. Other features are accessible through the toolbar at top of the user interface. But the most common steps are arranged in a flow bar along the left side of the user interface. The goal of the flow bar is not only to provide a convenient way to navigate to a particular step, but to guide you through the steps for a particular task.

Using the Flow Bar

Most flow bar steps bring up a dialog when you click them. You then fill in the parameters for that dialog and click OK. Once that step is done the next step in the flow bar is activated automatically. If you want to skip that step, you simply click another step in the flow bar. A unique feature of CrystalClear is that most dialogs associated with flow bar steps are not modal. This means the rest of the user interface is still active while the dialog is up. There is also an implicit cancel features which means you do not need to close a dialog before you click another flow bar button. If there is a dialog active when the flow bar button is pushed it closes automatically before the next dialog comes up.

Some flow bar steps are containers for other steps. For example the **Assign Unit Cell** step appears as follows when it first comes up.

The down arrow on the left indicates that this is a compound step. When you click the box it expands to reveal:



The **Find Spots** and other flow bar boxes are actual steps.

The previous example illustrates some other flow bar concepts. Normally when you click the flow bar button a dialog pops up. However, if you click the lightning bolt to the right of the button, CrystalClear executes the step immediately with the current parameters for that dialog.

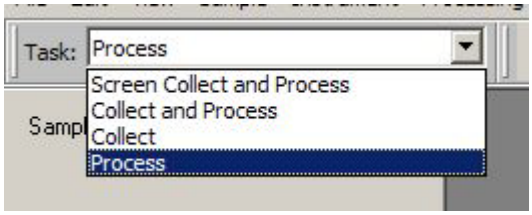
You will also notice the down arrows and the dotted arrow looping back to a previous step. The down arrow indicates the next normal step in the flow. This is usually the step that is activated automatically when the current step is completed. For manual processing the arrow is just a visual guide to the next step, so changing it into a stop sign has no effect. The dotted arrow is merely to indicate that you may want to return to a previous step if the results of the current step are not satisfactory for some reason.

 You can click the down arrow and it turns into a stop sign. This stops any automatic processing of the flow bar (e.g., in small molecule Auto mode) at the step before the stop sign.

 Many of the flow bar buttons have a lightning bolt to their right side. If you click this lightning bolt you immediately start executing the step using the current default parameters rather than bringing up the dialog associated with that set. While the step is executing this arrow has a red X on it. If you click this X it aborts the step. Alternately you can click the **Stop** button in the upper right corner of the screen.

Tasks

There are actually four different flow bars that correspond to each of four different tasks. You can select the task and its associated flow bar using task drop down in the upper left corner of the user interface.



When you click a task in this dropdown the sample task is set and the associated flow bar is displayed. These tasks include:

- **Screen Collect and Process**. This is the most common task for collecting data even if you do not plan to integrate the images during data collection. In order to be able to use this task successfully, you need to be connected to either a real or a simulated instrument.
- **Collect and Process**. For those who use the "Wild West" method (shoot first and ask questions later) to collect the data. This is not used very often in practice since it is often useful to collect screen images and index in order to decide how best to collect the data.
- **Collect**. Even more bare bones for those who simply want to collect the data and plan to process it later.
- **Process**. This is often referred to as **Process Only** and is used quite commonly to process or re-process data that has been previously collected.

These are same selection of tasks that are provided in the dialog to create a new sample.

Processing Suites

A processing suite is a program or set of programs used to process either screened images or collected data. These run as separate programs, but are integrated into CrystalClear's user interface. CrystalClear

comes standard with two state of the art processing suites with an optional third choice for twinned small molecule samples.

d*TREK. This was originally written by Dr. James W. Plugrath with more recent contributions from other members of Rigaku staff in the US. In addition to the standard processing operations, d*TREK includes features for ranking screen images and for automatic strategy calculations. d*TREK tends to work better for narrow images (< 2 degrees) than for wide images.

FS_Process (FS_PROCESS) Fine Slice Process was written by Dr. Tsuneyuki Higashi with contributions from other staff members of Rigaku Japan, FS_PROCESS is the successor to the PROCESS program. This program is popular in Japan and works well for both narrow and wide images. It does not currently include ranking or strategy.

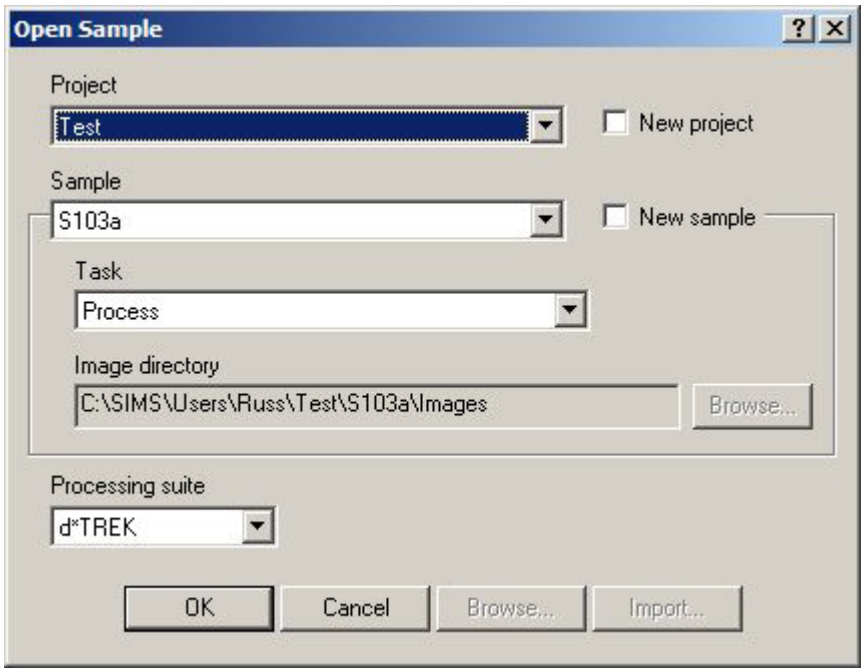
TwinSolve. Under a separate license CrystalClear, also provides an interface to Dr. Christer Svensson's TwinSolve program. As its name implies, it is particularly well suited to twinned small molecule crystals. If you have a small molecule system from Rigaku, this probably includes a license to TwinSolve.

It is possible to mix and match the processing suites to a limited extent. In particular, you can use d*TREK up through data collection and then process with FS_PROCESS. This is a fairly common approach for small molecule work especially on RAPID and SPIDER instruments.

Working with Samples

Opening a Sample

Many CrystalClear operations require a current sample open. When you start CrystalClear it automatically presents the Open Sample dialog.



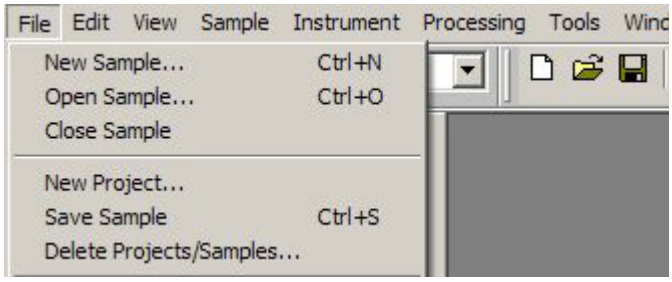
Certain operations (e.g., **Manual Instrument Control**) can be done without an open sample. So if you really don't want to open a sample, you can just click **Cancel**. But without an open sample, many functions are disabled.

Parameter	Description

Parameter	Description
Project	The existing projects are listed in the Project drop down list. If you want to open a new project, click the **New project** check box. Of course, if you open a new project you also have to open a new sample.
Sample	All the current samples in the selected project are listed in the Sample drop down list. Again check the **New sample** box if you want to open a new sample. This can be done either in an existing project or a new project.
Task	If you are not connected to either a real or simulated instrument, it is best to change the task to Process otherwise CrystalClear tries to connect to the Instrument Server for that instrument which fails if there is no instrument setup. See the Task section for more detail.
Image directory	If you are creating a sample with any of the Collect tasks, then the Image directory defaults to <sample directory>/Images, but you can browse to another directory if you wish. If this is a Process sample, then you need to click **Browse…** and navigate to the image directory. You can select any image in this directory that matches the image template you want to use.
Processing suite	Each sample has one of the available processing suites associated with it. The default is d*TREK.
Import	If New Sample is checked, the **Import** button is enabled. You can navigate to any sample in either your user area or in another user's area. You should select the <sample>_20.ho file in this sample directory. All of the files in the sample directory are copied to your new sample directory except the Image subdirectory. You are also given the opportunity to reset the path to the image directory since it could be on an external drive that has a different letter on your computer.

Sample Manipulation Commands

There are other ways to get to the Open Sample dialog.



New Sample… This pops up the Open Sample dialog with New sample checked.

Open Sample… This pops up the Open Sample dialog with nothing checked.

Close Sample This closes the currently open sample (usually close is automatic).

New Project… Pops up the Open Sample dialog with New project checked.

Save Sample. Save the sample database (.ho, xml) file.

Saving a Sample

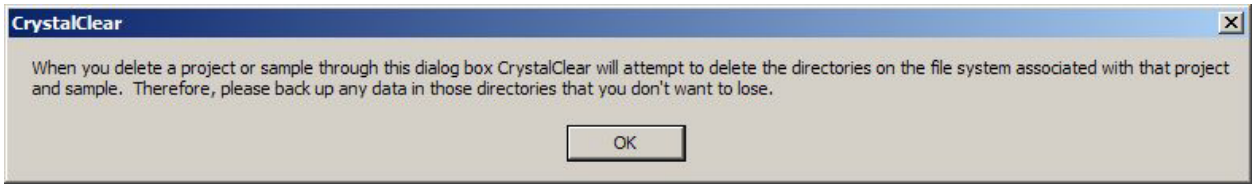
Sample information is kept updated on disk at the end of each step in the flow bar and whenever significant changes are made through toolbar or menu functions. When you exit CrystalClear, close a sample, or open a new sample, you are asked if you want to save the sample information. If you say **No** then any changes you have made to open dialogs and other temporary information is lost. But the changes made to the sample by each step are saved. Log, reflection, and other files are also updated as the processing steps are run. They are versioned. The database (.ho) file is also versioned up to 5 levels in case you need to back up. You can also save a sample explicitly with either the toolbar icon or the menu command.

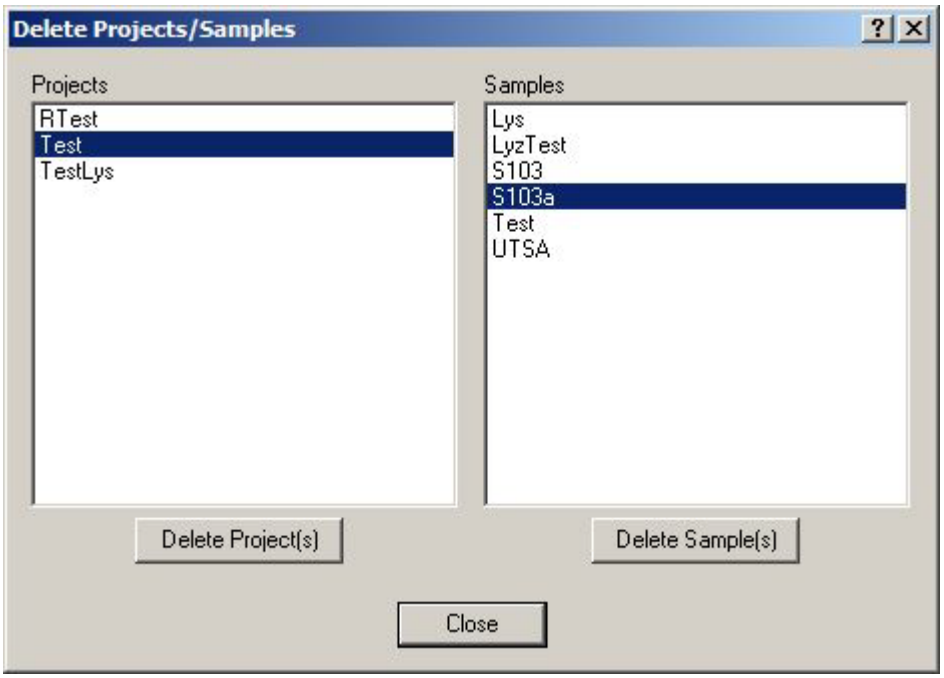
Deleting Projects and Samples

Since CrystalClear keeps an internal list of projects and samples, you should delete them through CrystalClear rather than just deleting them with Windows Explorer or other file manipulation program. If a project or sample is not actually found on disk in the expected path, you are asked if you want to update the internal list. If you really wanted to delete it, you should say yes, but if you are going to restore it later on, it is best to say no.

When you delete a project or a sample you are warned that this involves deleting directories and files and you are asked to confirm that you really want to do this. If the images are kept in an Images subdirectory of the sample, they are deleted with the sample, but if they are not in the project/sample directory structure, they are not be deleted. So you can, for example, delete a process only sample that refers to images collected in a different sample without deleting the images.

Use the following command and dialog to maintain your projects and samples. The menu entry is **File>Delete Projects/Samples.**





Parameter	Description
Projects	This is a list of your current projects. When you select one of these projects the samples associated with this dialog are listed under Samples.
Samples	These are the samples in the currently selected project.
Delete Project(s)	Delete the currently selected project and all the samples that it contains.
Delete Sample(s)	Delete the currently selected sample(s).

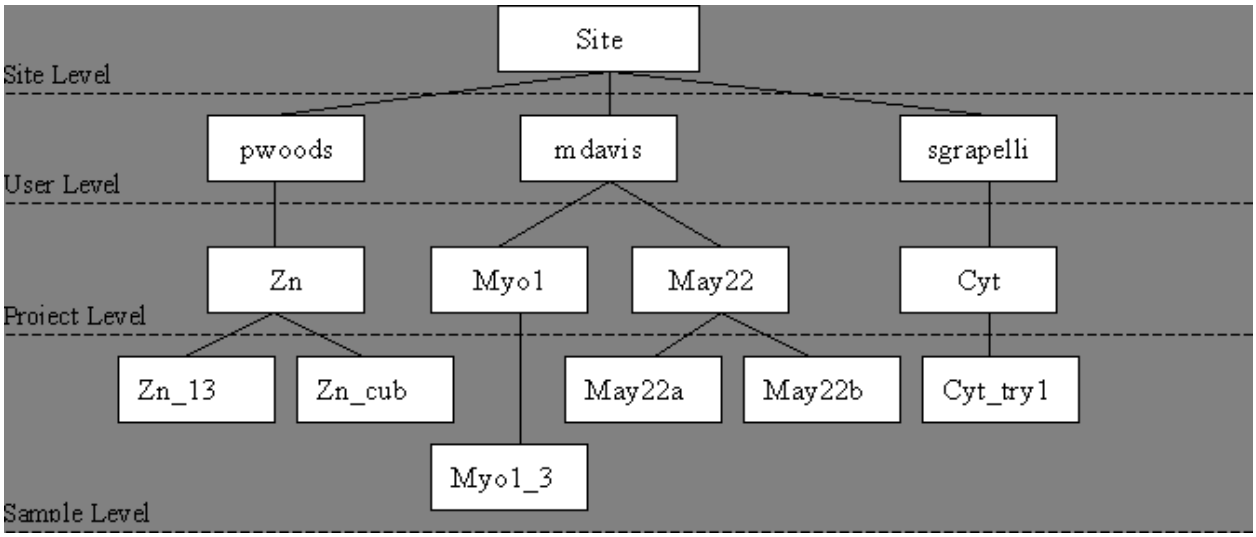
Defaults

Dialog Defaults

Overview

CrystalClear comes with a set of defaults that were determined by the application scientists at Rigaku to be the recommended values. These are based on instrument type and the mode (small molecule or macromolecule). Furthermore, within a sample, even if you close and then re-open a sample or exit CrystalClear altogether, the last values you set for a dialog are retained. So it is not usually necessary to change the defaults. In any case, this is not recommended until you have become familiar with CrystalClear and understand thoroughly how it works.

The chart below illustrates a site installation with three users and several projects and samples in order to illustrate how defaults are set and updated in CrystalClear.



When a new user is created, their defaults are taken from the site defaults. Note that the defaults are copied from the next higher level when the user (or project or sample) is created, so *changing the defaults at a higher level does not automatically change them at the lower levels.*

When a project is created, its defaults are taken from the user defaults.

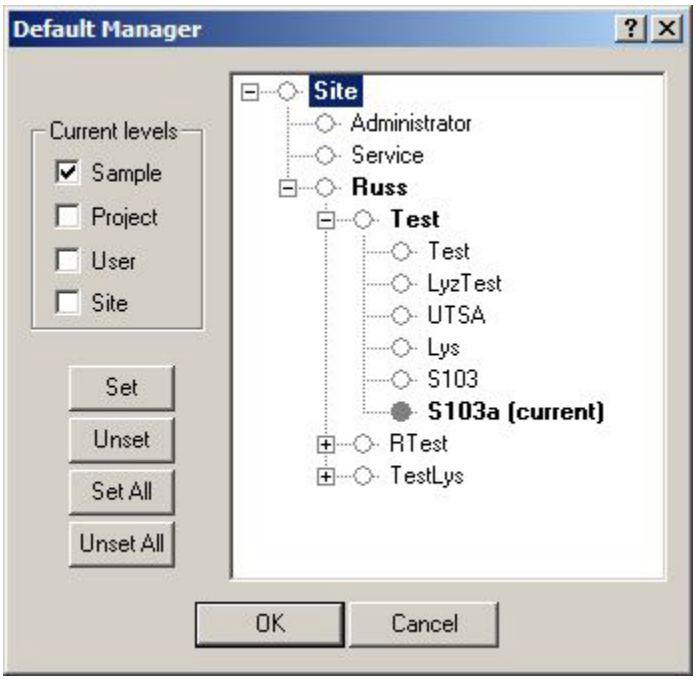
When a new sample is created, its defaults are obtained from the project under which it is created.

Example: When sample `Zn_13` was created, a copy of the defaults from project `Zn` was made and assigned to `Zn_13`.

Each CrystalClear dialog has a **Save** button. This button brings up the **Default Manager** dialog shown below which allows the user to save the defaults for that dialog to the selected level. When the user saves defaults at the project level, the changes are *not* automatically reflected in other, existing samples in the project, but the **Default Manager** allows defaults to be propagated to other samples, projects (and if you are an administrator, other users and the site defaults). In any case, any new samples created reflect the new defaults.

NOTE: Only the administrator can save defaults at the level of other users and the site. Any user can save up to their own user level.

Default Manager Dialog



This dialog allows you to choose both the level and the individual objects you want to update. The general practice and recommendation is that you save defaults to the project, user, and site level only. Once a sample has been created, it is a little misleading to go back and change the defaults that brought you to the current state. The exception to this rule is that you may want to save the defaults for a particular dialog to the current sample so that they are retained for later use.

The tree control on the right side of this dialog is what actually controls which samples, projects, and users have their defaults updated. If the circle is filled in, the associated level is updated. The controls at the left of the dialog are only shortcuts for selecting or deselecting elements in the tree control.

You have to have administrator privileges to set the site level or to set anything for another user. Note that this means that you are in the administrator group for CrystalClear which was specified when your CrystalClear user account was added, not that you have administration privileges in Windows which is entirely separate.

Parameter	Description
Current levels	This series of check boxes just selects the current sample, project, user, or the site level. It does not affect how the buttons below it work.
Set/Unset	These two buttons just set or unset whatever is highlighted in the tree control. Since you can toggle the selection state simply by clicking on the circle next to the object name, these are not heavily used.
Set All/Unset All	These buttons set or unset everything in the table. This can get you closer to the final selections that you want, but you usually have to go through and select objects one at a time.
OK	Click OK when you have selected the items to be updated by the new defaults.
Cancel	Exits without updating any defaults.

Collection Schedule Defaults

There are some additional steps to create collection schedules that can be shared with other samples, projects, or users. The best way to do this is from the **Scan State Display** which is described in the next Chapter in order to create and store new schedules.

CrystalClear Databases

The CrystalClear database files are binary files with a .ho extension that contain information that CrystalClear needs to maintain from one run to the next. These files are also stored as xml files, but the xml files are read only if the .ho file cannot be found. Normally these fails are maintained entirely by CrystalClear and you need not worry about them. However, they can occasionally become corrupted (e.g., if the computer has a problem during the middle of a write operation) and you are asked by your Rigaku service person to delete the .ho file to force CrystalClear to revert to the xml file.

The database files in CrystalClear are listed in the following table.

Database	Directory	File Name
Administration (Users)	*C:\Rigaku\CrystalClear\ Administration	*AdminDatabase_20.ho *AdminDatabase_20.xml
Site defaults	*C:\Rigaku\CrystalClear\Data\ SiteDefaults	<instrument name>_20.ho <instrument name>_20.xml
User preferences	<user data directory>	*UserPrefs_20.ho *UserPrefs_20.xml
User defaults	<user data directory>	*CrystalClearUserDefaults_20.ho *CrystalClearUserDefaults_20.xml
Project list	<user data directory>	*CrystalClearProjects_20.ho *CrystalClearProjects_20.xml
Project defaults	<user data directory>\<project>	*CrystalClearProjectDefaults_20.ho *CrystalClearProjectDefaults_20.xml
Sample list	<user data directory>\<project>	*CrystalClearSamples_20.ho *CrystalClearSamples_20.xml
Sample (cell parameters, history, dialog defaults, collection information)	<user data directory>\<project>\ <sample>	<sample name>_20.ho <sample name>_20.xml

* - Directory or Filename for this database does not change.

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4. The Toolbar and Views

CrystalClear includes a set of views (also referred to as displays). The toolbar is described briefly and the most commonly used viewers are discussed in detail in this chapter. Specialized viewers that are used less often are covered in Chapter 12 on **Tools**.

Some of these views also allow you to interact directly with the instrument or modify parameters and settings used data collection or processing, so they are interactive views rather than simply passive views.

The viewers covered in this chapter include

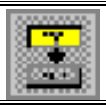
- **Image Viewer**. For examining the collected images.
- **Processing State Display**. Cell parameters and other processing information
- **Scan State Display**. For monitoring and managing collection schedules
- **Instrument State Display**. Axis positions and image collection monitoring
- **Manual Instrument Control**. Control functions for low level operations on the instrument
- **Log File Viewer**. To review processing log files

Toolbar Overview

The **Toolbar** displays buttons which are easily accessible to complete common activities. As you move your mouse cursor over each button, a Tool Tip flyover helps you identify the button function. Many of these functions can also be accessed using the menu.

	**New Sample**. Accesses the **Open Sample** dialog, allowing you to create and name a new sample.
	**Open a Sample.** Accesses the **Open Sample** dialog, allowing you to open an existing sample.
	**Save Sample.** Saves the current sample.
	Use **d*TREK** as the processing suite.
	Use **TWINSOLVE** as the processing suite.
	Use **FS_ PROCESS** as the processing suite.

	**View Image Collection Updates**. When toggled on, image updates are displayed during collection. Toggle ON position shows as the button pushed in.
	**View Image Processing Updates**. When toggled on, processing results are displayed during processing. Toggle ON position shows as the button pushed in. If **View Image Processing Updates** is toggled ON, the reflection in the reflection file is represented on each image using the following color scheme: • Included = blue circles, • Excluded = green circles, • Rejected = red circles. Adjusting **Resolution Range** fields, and I/σ includes/excludes reflections. Adjusting the rejection limit, (X and Y in mm, Rot in Degrees) includes/rejects peaks). Also displayed on each peak is a difference vector representing the difference between the peak top on the image and the center of the predicted reflection related to the current values of the orientation matrix.
	**View Image.** Accesses the **Open Image File** dialog, allowing you to select an image to view.
	**View Processing State Display**. Accesses the **Processing State Display** dialog, allowing you to make adjustments in the processing state.
	**View Scan State Display**. Accesses the **Scan State Display** dialog, allowing you to make create, edit, and examine scans within named schedules.
	**View Instrument State Display**. Accesses the **Instrument State Display** dialog.
	**View Manual Instrument Control.** Accesses the **Manual Instrument Control**, allowing you to control the instrument.
	**View Log File**. The **Open Text File** dialog appears, allowing you to select the log file to view in text format. The file is then opened in the Log File Editor.
	**Set Resolution**. Access the **Set Resolution** dialog, allowing you to set resolution limits.
	The **RAX Shape** crystal viewer are displayed. This is available for small molecule only.
	**Open Reciprocal Space Viewer.** Initiates the **Reciprocal Lattice Viewer** which uses the current cell parameters (including any twin information found by **TWINSOLVE**). The **Lattice Viewer** can also be used to help diagnose and even index problem samples.
	**Display Summary of Indexed Twins**. Accesses the **Twin Summary (TwinSolve)** table.
	**Previous Window.** The previous window is displayed. (Note there is a separate green arrow for the next image in the **Image Display**.)
	**Next Window**. The next window is displayed.

	**About**. The **About** dialog displays information about the current version of *CrystalClear*.
	**Pause.** Pause data collection after the current image has been collected. This turns into a continue button once you have paused. These controls are also in the instrument state control.
	**Stop After Image.** Stop after the current image has been collected. The advantage of this over the full immediate stop below and the Abort button that is always visible is that since it allows the current image to finish, the system state is known and you do not have to re-initialize when you restrart.
	**Toggle Task List**. Toggle ON/OFF to display/remove **Task List** on **Toolbar**. Toggle ON position shows as light, recessed frame.
	**Toggle Standard Tools.** Toggle ON/OFF to display/remove **Standard Tools** on **Toolbar**. Toggle ON position shows as light, recessed frame.
	**Toggle Flow Bar**. Toggle ON/OFF to display/remove the **Flow Bar** on *CrystalClear* interface. Toggle ON position shows as light, recessed frame.
	**Toggle Command Bar.** Toggle ON/OFF the **Command Line** to display/remove it from the *CrystalClear* interface. Toggle ON position shows as light, recessed frame. The **Command Bar** is not used currently, so this should be left off.
	**Toggle Message Bar**. Toggle ON/OFF the **Message Bar** to display/remove it from the *CrystalClear* interface. Toggle ON position shows as light, recessed frame.
	**Toggle Status Bar**. Toggle ON/OFF the **Status Bar** to display/remove it from the *CrystalClear* interface. Toggle ON position shows as light, recessed frame.
	Not used
	**STOP** (shown inactive) Close the shutter and shut down the device driver or processing server immediately.
	**Cube** The cube rotates to show the activity of processing and collection data.

Activating Views

Toolbar Icons

The viewers can be activated by the icons toward the center of the toolbar.

They are also activated automatically when they would be useful to display the results of an operation. For example, if the View Image Collection Updates is active, the image viewer is started when the first image has been collected. More than one view can be open at once, but only the front view is active.

You can use the Windows menu or the green forward and back arrows to bring views to the front. And of course, you can move views around within the CrystalClear main window.

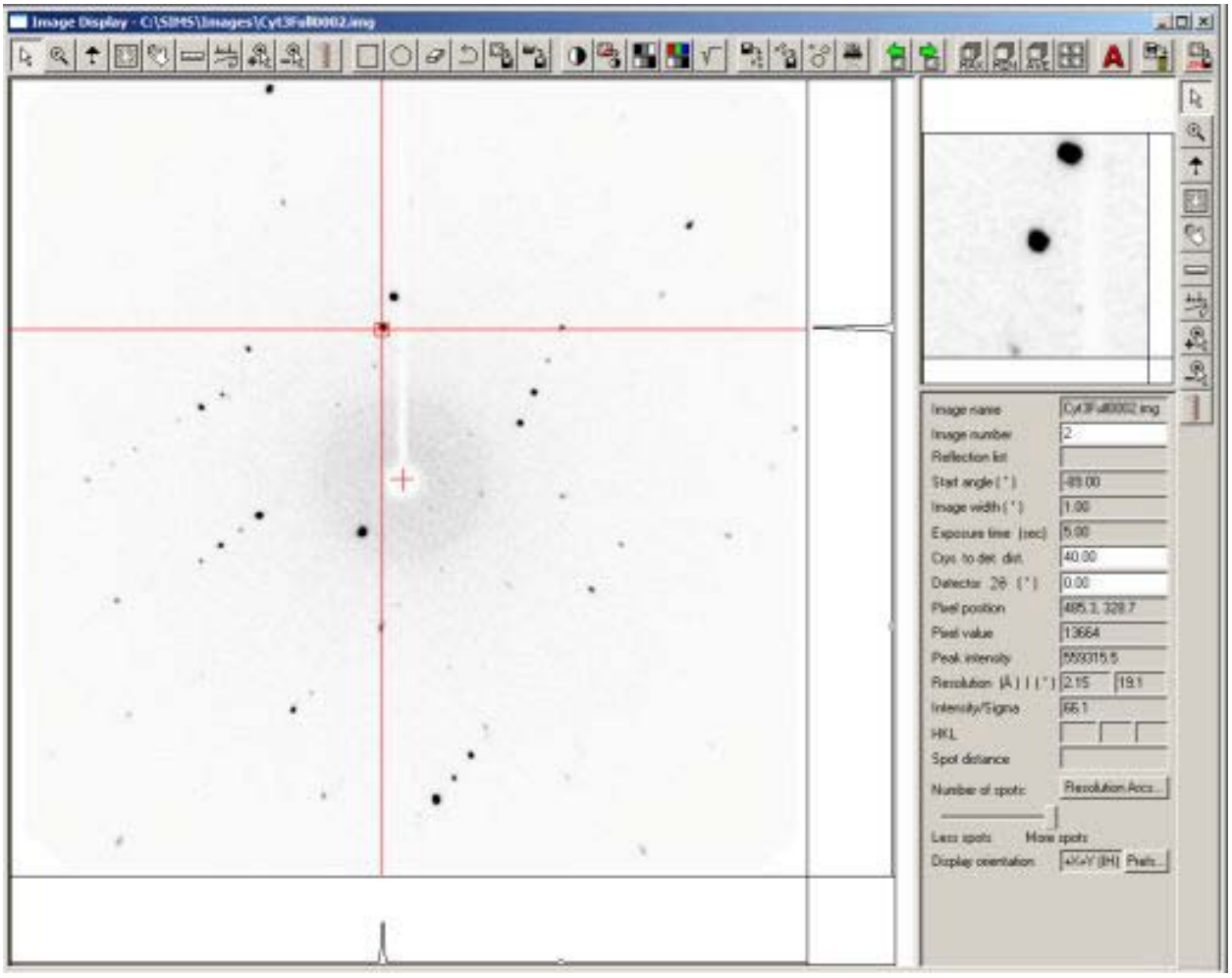
Image Display

Overview

 The Image Display is activated by selected by clicking on this icon. Alternately you can select File|Open Image… from the menu. The Image Display may also be opened automatically if you have selected either View Image Processing Updates or View Image Collection Updates from the toolbar.

The Image Display provides many options for displaying the image in various ways at various scales and position. It also provides an interface for defining masks and selecting reflections. Most of these functions are documented in the section on the main toolbar.

The basic layout is show below.



There are several areas of this viewer.

- **Main Display**. This is the large image display area which is shown with a red cross in the above view. The tool bar icons at the top of this display area operate on this display.
- **Profile Display**. These are the two areas along the bottom and to the right of the main display which display profiles of the cross section of the image along the red cross.
- **Zoom Display**. This is the smaller image display to the upper right which shows a zoomed in view of the part of the image selected by the red cross.

- **Image Information**. This is the form at the lower right which contains information from the image header.
- **Main Toolbar**. The toolbar at the top of the display operates on the main display.
- **Right Toolbar**. This vertical toolbar operates on the zoom display.

Image Controls

The icons in the main toolbar allow you to manipulate the main display. The icons in the right toolbar have the same function as they do in the main toolbar except they apply to the zoom display rather than the main display. For most of these icons you click the icon to select the desired control and then click the image to perform the function associated with that tool.

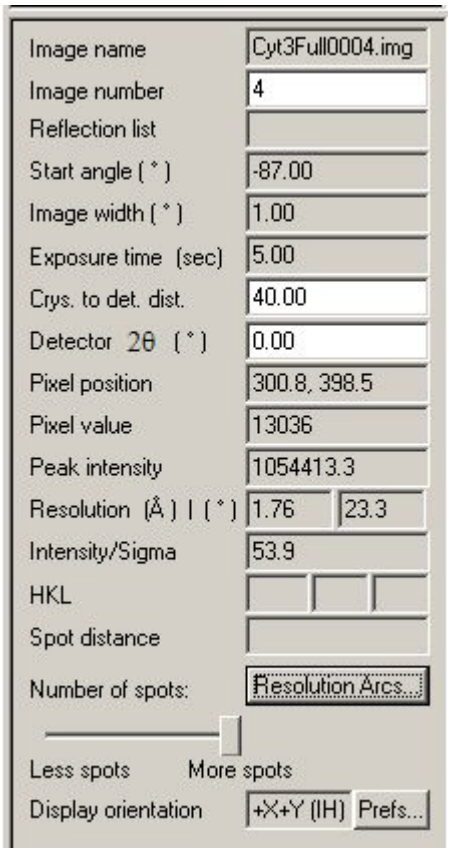
	**Select**	You can left click anywhere in the main display. This centers the red cross and the zoom display on this pixel and show information about that pixel in the Image Information window. The horizontal and vertical profiles along the red cross are displayed automatically in the profile display. You can also right click and drag out a box. This zooms into the area selected by the box preserving the aspect ratio of the main display.
	**Magnify**	With the Magnify tool you can left click the image and the area immediately under the cursor is magnified. You can drag the cursor to scan across regions of the image.
	**Zoom**	Generally the best way to zoom is to use the right click feature under **Select** which changes both the zoom level and the center of the image. If you want to change the zoom level and leave the center as it is, you can left click Zoom and click anywhere in the display and drag the cursor up for zooming in and down for zooming out.
	**Full View**	This tool returns the display to the original image display size which fits the entire image in the space available in the main display. You can also double click the main image display.
	**Pan**	Left click and then drag the image. Note that this does not change the selected pixel on the image which means that the zoom display, red cross, and profile display do not move when you pan.
	**Measure**	This tool displays a measurement in Ångstroms between two points. Click a point, then drag to a second point.
	**Profile**	Use this tool to get a Profile of a cross section. Unlike the **Select** profile, which gives only horizontal and vertical profiles, with this tool you can get a profile of pixel values in any arbitrary direction. Click a point, then drag to a second point to display the profile.
	**Add Spots**	Click a spot to add it to the displayed reflection list.
	**Delete Spots**	The spot closest to where you click is removed from the reflection list that is being displayed.
	**Measure Pixels**	By clicking on a point in the display and then dragging to another point in the display, you can determine the number of pixels between the points. Note that these are pixels in the original detector image and not simply display pixels so they are independent of the zoom level.
	**Quad Erasure**	This is used to create a mask region for a mask file. You can click four different points which defines a quadrilateral (the line from the fourth

		point back to the first point is implicit). Any reflections within this quadrilateral are masked out for integration. After you have used this tool and the circle tool to define the mask areas, you must save the mask file.
	**Circle Erasure**	To define a circular mask region you need to click first on the center of the circle and then drag the radius out as far as you would like.
	**Free-hand Erasure**	This tool is not currently active.
	**Undo Last Erasure**	Undo the last erasure. This tool only allows a single undo of the quad and circle tool actions.
	**Write File**	Save an image that has been modified.
	**Write Mask File**	Click this tool to save a mask file. The mask file is an image file that includes any erasures you might have made. You are prompted for the location of the mask file.
	**Contrast**	This brings up a control which lets you interactively adjust the contract of the image.
	**Reverse**	Reverse the pixel values (i.e. show the negative) of the image.
	**Color**	Toggle between gray scale and color display.
	**Toggle Square Root Display**	Displays the image with each pixel value being converted to its square root. This gives more contrast to the image, truncating the strong pixels and raising the display of weak pixels.
	**Reset Local Contrast**	Sets the contrast of the displayed portion of the image. This is useful in zoomed areas where there is a smaller or greater difference in minimum and maximum pixel intensities than the current contrast values.
	**Load Reflection List**	Loads a reflection list into memory. If you would like to add or remove reflections from this list you can use the Add Spots and Delete Spots tools, then click Write Reflection List to save the edited reflection list.
	**Write Reflection List**	Saves the current reflection list with any additions or deletions.
	**Set Reflection Size**	Adjust the size of the circle drawn around each spot. Note that this circle is for display purposes only and does not reflect the integration area or background/peak separation.
	**Toggle Filtering**	Filter out spots from reflection list that are not located on the current image. For example, a spot that appears in Image 10 may not intersect Image 1. This tool filters out all spots whose rotation range does not intersect the displayed image.
	**Previous Image**	Display the previous image in the series. For example, if the current image is Img004.osc, clicking this tool displays Img003.osc . This allows you to click through the images in a data set very quickly.
	**Next Image**	Display the next image in the series. For example, if the current image is Img004.osc, clicking this tool displays Img005.osc .
	**Overlay Images**	Display an image created from a specified series of images. Each pixel in the resultant image is generated by using the maximum value occurring in the images for that pixel position. This is generally used to display the image that would result from a wider rotation/oscillation angle.
	**Underlay Images**	Display an image created from a specified series of images. Each pixel in the resultant image is generated by using the minimum value occurring in the images for that pixel position. This is generally used for examining the background over wide rotation/oscillation angle or for examining the

		extent of a reflection across several images and rotation angle values.
	**Average Images**	Display an image created from a specified series of images. Each pixel in the resultant image is generated by using the average value occurring in the images for that pixel position.
	**Tile Images**	Displays an image created from tiling a specified series of images.
	**Image Font**	Clicking on this tool brings up a dialog which allows you to change the font used in the image viewer.
	**FSP Mask Delete**	This deletes the current FS_PROCESS mask file (blind.dat) and is only useful when FS_PROCESS is the current processing suite.
	**Write JPG File**	This writes the current image as a jpg file. Images are quite large. Being able to write them as a jpg file compresses them and allows them to be included in documents and viewed by standard image viewers.

Image Information

The image information area contains vital information about the current image, the current selected pixel on that image, and controls for filtering the reflection list and controlling display options.



The image name and associated image information for the current image are display in the top section of this panel. The values relating to the current pixel are:

Pixel position. Pixel position (in the original image space, not the image display space – so this does not depend on zoom).

Pixel Value. The value of the current pixel in the image file.

Peak Intensity. The intensity of the nearest peak to this pixel.

Resolution. The resolution of the current pixel in Angstroms and degrees.

Intensity/Sigma. The I/Sig of this pixel.

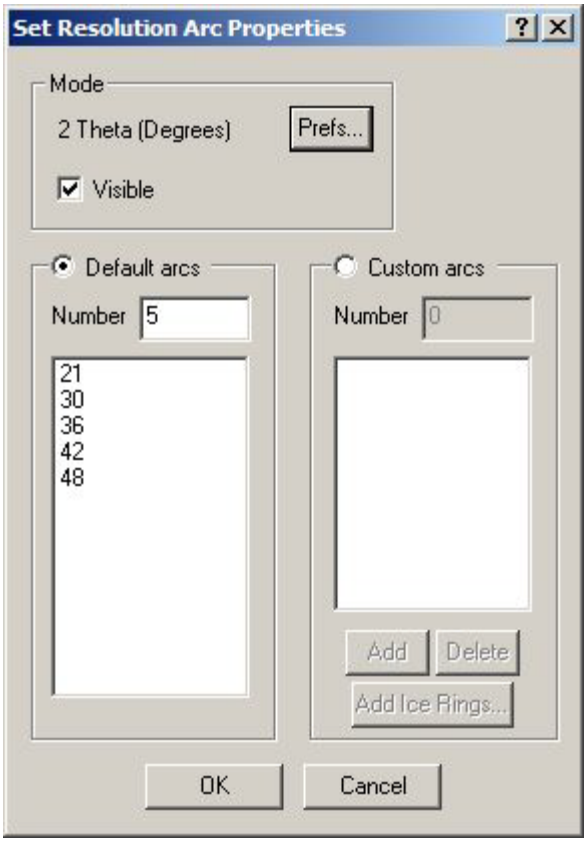
HKL. If the sample has been indexed and there are cell parameters, and you are reasonably close to the reflection center, the integer HKL values are reported.

Spot Distance. If you are using the Measure tool, the current distance between spots is display in this area.

Number of spots. This text control is actually replaced with the number of spots in the current image.

Spot Slider. The spot slider allows you to filter out the weakest spots. This is only really useful after Find and before Index. You can then save the spot list to the sample directory using the Write Reflection List tool in the main toolbar and then use this reflection list for indexing.

Resolution Arcs. This brings up a dialog for setting resolution arcs.



To display arcs you need to click the **Visible** check box. You can then control the number of Default arcs, or select Custom arcs that you define yourself. The **Prefs**… button brings up the preferences dialog which is described in Chapter 2 on installing CrystalClear.

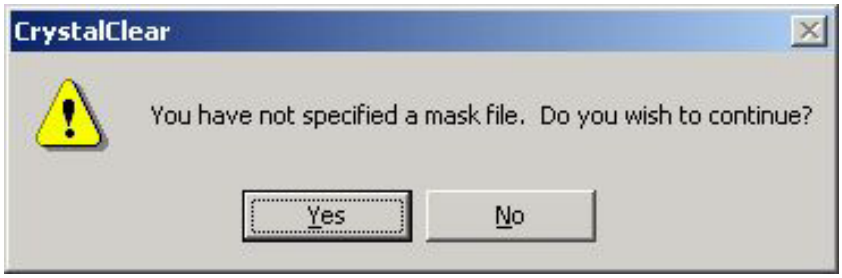
Display Orientation. This shows you the display orientation. To change the orientation, you must click the **Prefs…** button which brings up a dialog described in Chapter 12. In the example above the image orientation is taken from the image header which is the normal and recommended setting.

Creating a Mask File

A **Mask File** may be used to define the shadow of the beam stop on images and mask the pixels in the beam stop shadow. Reflections overlapping with the shadow are excluded during **Integrate Reflections.** d*TREK and FS_PROECSS, and TwinSolve use mask files to improve the quality of the integration.

The mask file is usually defined interactively in the **Image Viewer** with the tools described below. You may do this at any time, but it might be best to do it after you have collected the screen images. Note that mask files are dependent on 2 Theta and to a lesser extent on distance, so if your scans have multiple 2 Theta and distance values, you should do a mask file for each scan.

If you have not specified a mask file prior to clicking the **Run** button on the **Integrate Reflections** dialog, the following *CrystalClear* message appears:



- Clicking **No** discontinues the **Integrate Reflections** so you may go back to the **Image Viewer** to create and specify a mask file before continuing.
- Clicking **Yes** continues with the **Integrate Reflections** without a mask file.

1. Select and display any image in the **Image Window** by clicking the **View Image**  on the CrystalClear **Toolbar**. Select any image.

2. Click **Circle Erasure**  on the **Image Window Toolbar** to define a circular area to be masked during processing. To use, click the center of the circular area, then drag to the outside edge of the area. When the mouse button is released, the circular area are defined. This is usually used to define the mask for the cup of the beam stop.

3. Click **Quad Erasure**  on the Image Window Toolbar to define a four-sided area to be masked during processing. To use, click in four corners of the area to be removed. This created 3 lines since the 4th line is automatically connected back to the first point. This is usually used to define the mask for the support of the beam stop from an image.

4. Click **Write Mask File**  on the **Image Window Toolbar** to save a mask file. The mask file is an image file that includes any erasures you might have made.
The **Save Image File As** dialog appears.

5. In **File Name** type a unique name for this mask file.

NOTE: You may create multiple mask files by repeating steps 1-4, then using a unique name for each mask file.

6. Click **Save**.
The following CrystalClear message appears:

7. Click **Yes** to use this mask file for all scans. Click **No** to use different/multiple mask files for various scans.

All Rigaku CCD detectors now create an **Active Mask** which is encoded at the end of each image file which marks bad pixels. This is all handled automatically, so that you do not need to worry about it.

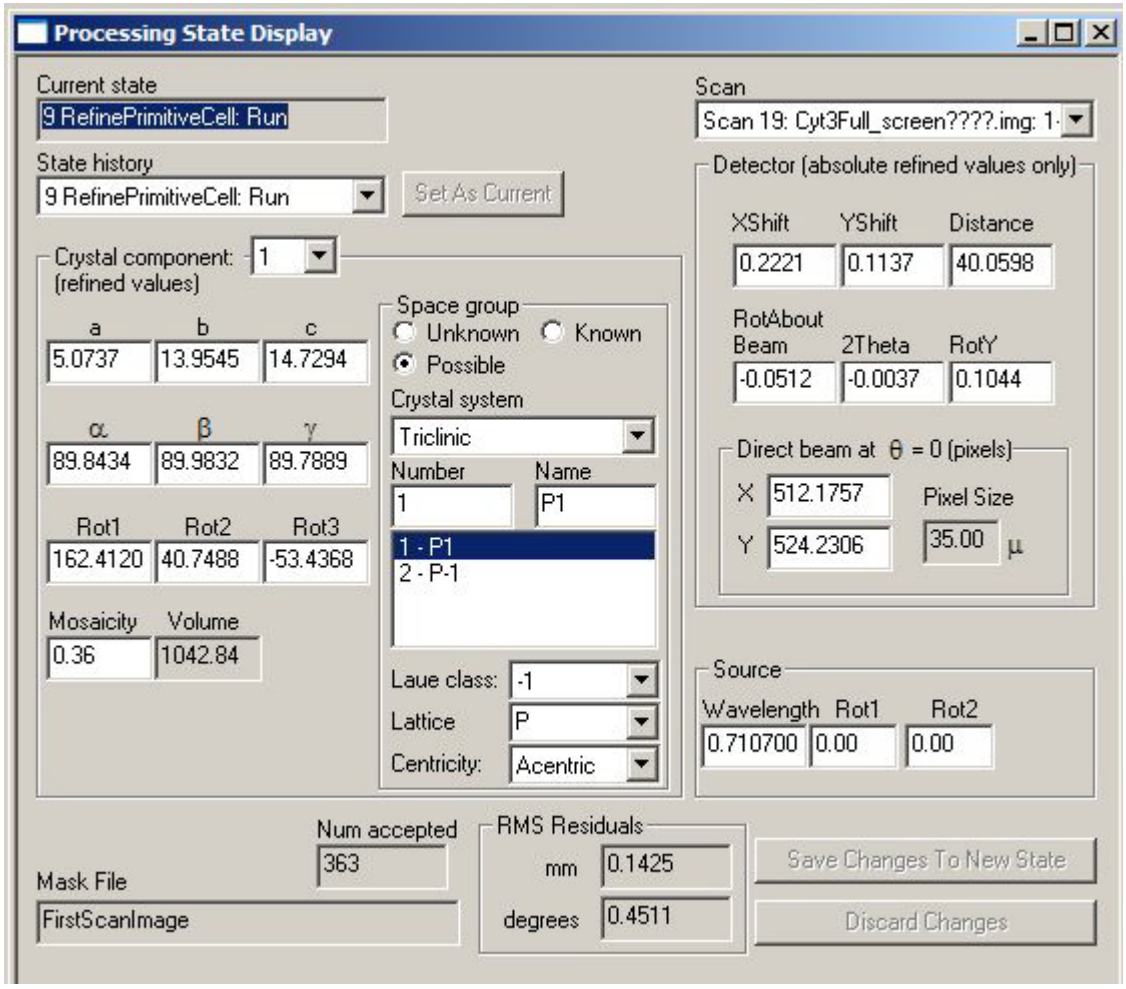
Processing State Display

Overview

 The processing state display is used to show the current cell parameters and other information related to the current sample. It can be activated at any time by clicking on the icon in the top tool bar shown here. It can also be activated by clicking Processing State Display… in the View menu. And finally, it pops up on its own after indexing or other processing steps that change the state.

CrystalClear maintains a full copy of the state of the sample after each processing step. These states are numbered and stored in a stack (most recent on the top of the stack). This complete state history allows you to go back to any previous step (e.g., if the last refinement messed up the cell parameters beyond repair). It is even possible to edit parameters (e.g., the cell values) by hand and create a new state to be used for further processing. This state history is stored when you Save the sample and is restored when the sample is re-opened even if you have exited CrystalClear.

Display Parameters



Parameters	Description
Current State	The number and description of the step that created the current state. In this example this is the 9th state and was created by **Refine Primitive Cell**.
State History	This is drop-down list that contains the history of all states of the sample with the most recent at the top. When you select a state, the values associated with that state are shown in the **Processing State Display**.
Set As Current	Although the new state values are displayed when you select the state in the **State History**, they are not set as the current state for further processing unless you click the **Set As Current** button.
Save Changes To New State	To create a new state, select an existing state that is near to what you want and then edit the parameters you want to change (e.g., change a cell axis that is twice as long as it should be). You can then click this button and it creates a new state with the current values and make this the current state for further processing.
Discard Changes	If you have made edits to the parameters in the **Processing State Display** and decide you do not want to keep them, you can click **Discard Changes** and the original values associated with the selected state is restored.

Parameters	Description
Crystal Component (refined values)	The crystal component can be used for selecting the current twin component. This is most useful when using the TwinSolve option. Otherwise this is just 1.
a **b** **c** **α** **β** **γ**	The current unit cell parameters.
Rot1, Rot2, Rot3	These are the orientation angles determined during indexing.
Mosaictiy	The current mosiacity as calculated by the current processing suit.
Volume	The crystal volume.
Space group	The first group of radio buttons has one of three states depending on the extent to which the space group is known. • Unknown • Possible • Known
Crystal System	This drop down shows the current crystal system and allows you to select a different one.
Number	The most likely space group number
Name	The most likely space group name
{possible space groups}	This is a list of possible space groups starting with the most likely.
Laue Class	The current Laue class with the option to select others through a drop down list
Lattice	The current lattice with a drop down list
Centricity	Centric or Acentric
Scan	This shows the scan that was used to in the last step. All the current scans are in the drop down list, so you can select other scans.
Detector (absolute refined values)	This area displays the refined detector settings.
XShift **YShift** **Distance**	Refined detector shift and distance
Rot About Beam **2 Theta** **Rot Y**	Refined rotation and 2 Theta shifts.
X **Y** **Pixel Size**	The direct beam position (calculated back to 2 Theta = 0 even if the current 2 Theta is something else). Pixel size for this detector
Source	The source settings are for information and should not usually be changed here. It is best to change them in the Setup dialog and then save them to the configuration file.
Wavelength **Rot1** **Rot2**	Source parameters
Mask File	Shows the mask file currently used for integration. This can be changed in the integration step.

Parameters	Description
Num Accepted	This shows the number of reflections accepted in the last step (e.g., refinement).
RMS Residuals	This is a summary of the RMS residuals that came from the last refinement step in millimeters and degrees.

Scan State Display

Overview

 The Scan State Display can be activated by clicking on this icon in the top toolbar or by selecting **Scan State Display** in the **View** menu. This view shows what is collected and what has already been collected. It also allows you to create and store collection schedules so that they can be used in other samples. You can use this view for several different purposes:

- Review the current status of your data collection. This can also be done through the Instrument State Display which gives you a more detailed view.
- Review the status of your data collection if it has had to be interrupted for some reason. The scan state display tells you exactly what you were collecting and how far it got.
- Review the scan information from a sample that you re-open or create as a process only sample.
- Create standard schedules that you can re-use at other times or share with other users at your site.

Before getting into the details of this view, it is necessary to define some of the terminology used by CrystalClear which is adopted from d*TREK conventions.

Images

An image is a single exposure from the detector. For image plate detectors (e.g., R-AXIS, Rapid, etc), these have a .osc extension (since historically these devices have fairly long exposure times and oscillate at least once back and forth during the exposure). For CCDs the images have a .img extension. Images correspond to what are sometimes called “frames” in other crystallographic software.

Scan

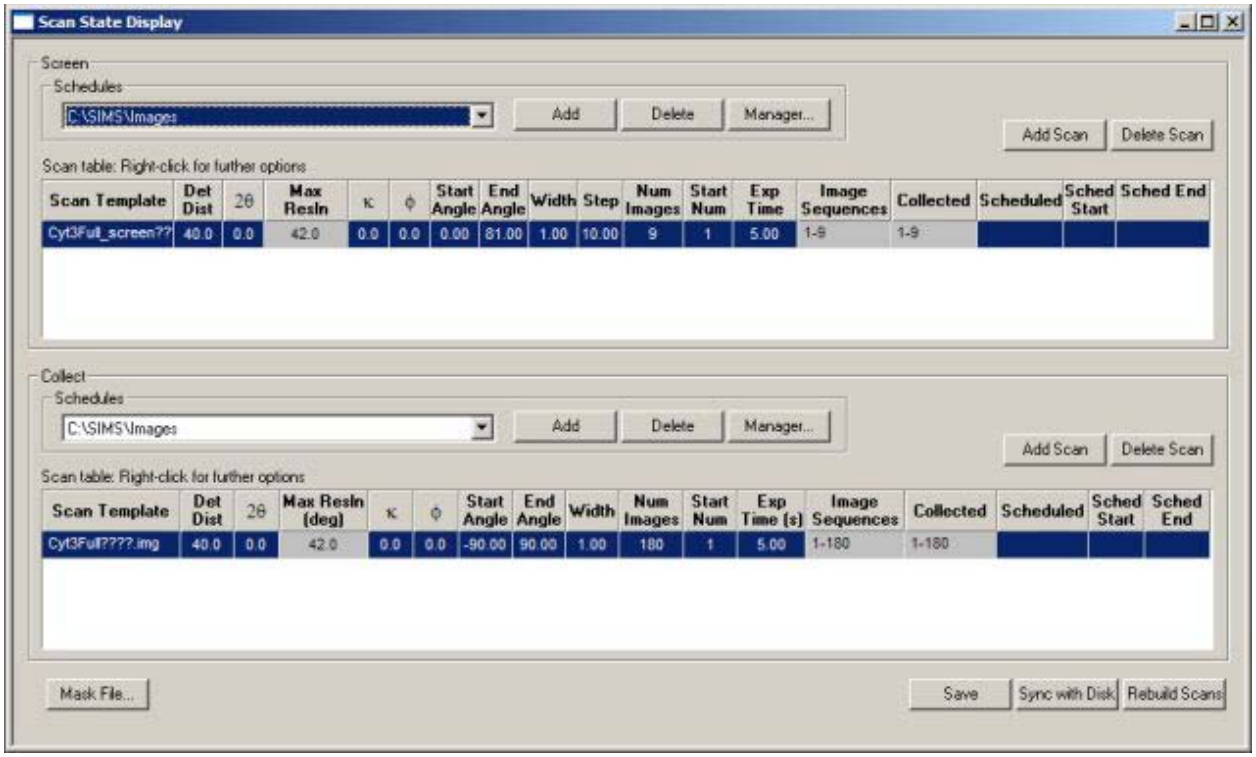
A scan is a collection of images that differ from each other only in the setting of a scan axis. For example, the scan may be from 0.0 to10.00 degrees. Each image in the scan has a **width** which is the amount the axis rotates during the exposure and a **step** which is the distances from the start of one image to the start of the next image. For screening small molecule data you might have a scan with a 1 degree width and a 10 degree step that would go from 0.0-81.00 degrees in 10 degree steps, so that there would be 9 images. Note that the end angle is the angle at the end of the last image, not at the beginning.

If a set of images do not meet the rules for a single scan, they are divided into two scans. For example, if you collected four 0.5 degree images at 0, 0.5 and 90.0, 90.5, this becomes two scans. Similarly if another axis changes (e.g., chi or kappa) from one image to the next, a new scan is generated.

Schedule

A schedule is a collection of scans that usually defines a complete data set. There are **Screen** schedules and **Collect** schedules maintained separately by CrystalClear. Schedules have names and can be made accessible to other samples and even other users.

Controls



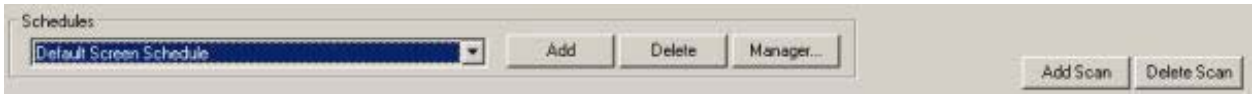
There are several regions of this display that are dealt with separately.

- Screen
  - Schedule Management
  - Scan Table
- Collect
  - Schedule Management
  - Scan Table
- Miscellaneous Controls

The schedule management and scan tables are the same for both screen and collect, so we only describe these controls once. The exact scan tables and similar schedule management is also used in the separate screen and collect dialogs that are associated with the **Initial Images** and **Collect** steps in the flow bar.

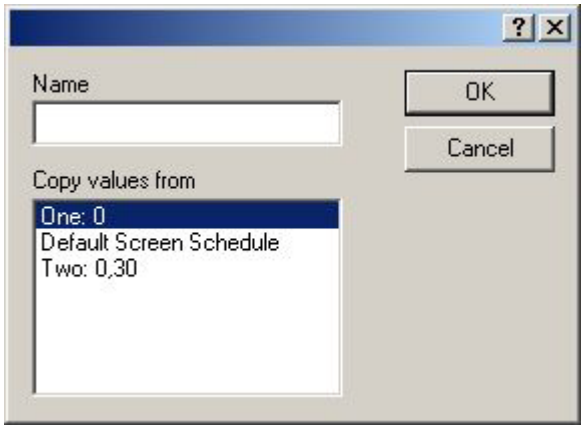
Schedule Management

The controls for schedule management are shown below.



Schedule Drop Down. This is a drop down list of pre defined schedules for either screen or collect. These are stored in the defaults and so your sample inherits the schedules that are in the project defaults. If this is a process only sample, then there is a derived schedule which was constructed when the sample images were read in. The name for this schedule is a path to a directory. When you select a schedule, the scans associated with that schedule are shown in the scan table.

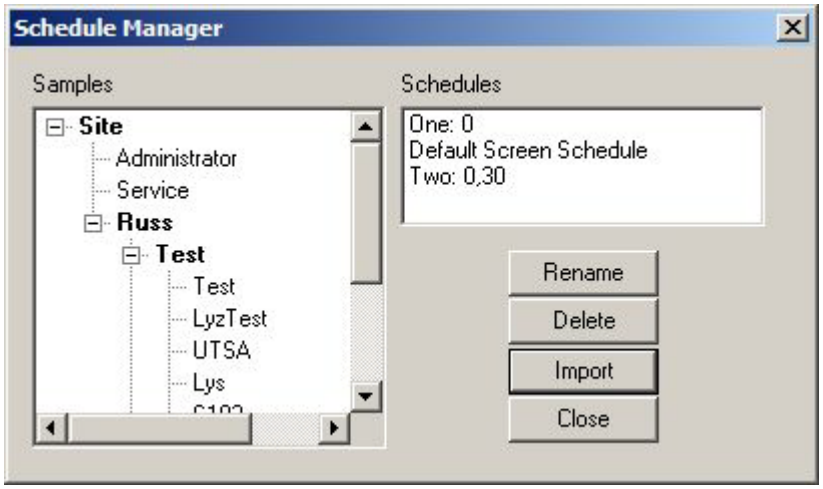
Add. This button allows you to add a new schedule to the screen or collect schedule list. The dialog that comes up when you click this is:



You should enter a Name for the new scan and choose one of the existing scans as a starting point for deriving this scan. If you want to use this schedule in other samples, you need to click the Save button at the bottom of the dialog after you have added the schedule. This brings up the standard Default Manager described in Chapter 2 which allows you to select the levels and objects to be updated with the new schedules. Note that this saves the entire current scan table list to each of these rather than just adding the new sample you have created.

Delete. When you no longer need a schedule, you can delete it with this button.

Manager. For more complex schedule management, this button brings up a separate dialog.



The **Samples** tree control allows you to navigate to a user, project, and sample. The current schedules for the selected level are shown in the **Schedules** list. You can navigate to the schedule you want to operate on and then use one of the following buttons

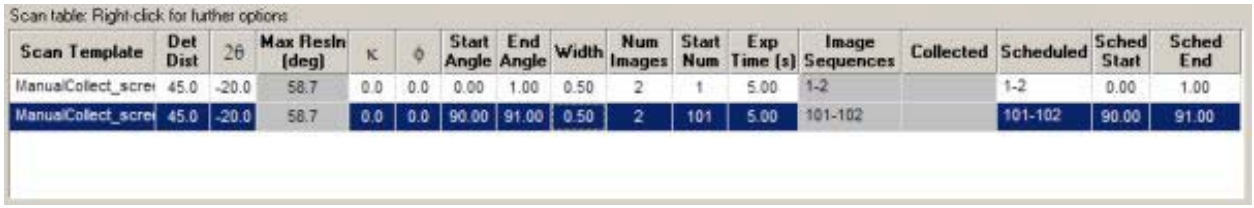
- **Rename**. This allows you to rename the schedule. You should not rename schedules that are not in your current sample, project, or user.
- **Delete**. Delete removes the selected schedule. Again you should restrict this to your own sample, project, and user.
- **Import**. If there is a schedule that you would like to use which is not in your current schedule list, you can navigate to this schedule and then import it.
- **Close**. Closes the schedule manager.

Add Scan. This adds a scan to the screen or collect schedule. Its initial settings are copied from the last scan.

Delete Scan. If you want to remove a scan, select it and the click Delete Scan.

Scan Table

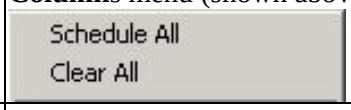
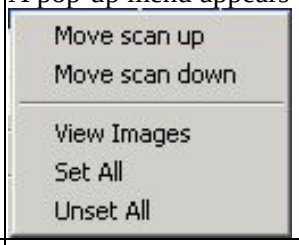
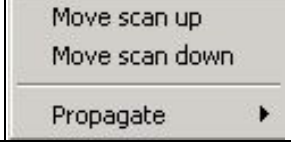
Scan Tables are used in the Scan State Display and in the screen and collect dialogs. It works the same way in all these environments.

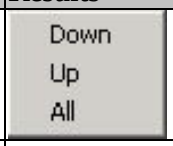

Scan table: Right-click for further options

Scan Template	Det Dist	2θ	Max ResIn (deg)	κ	ϕ	Start Angle	End Angle	Width	Num Images	Start Num	Exp Time (s)	Image Sequences	Collected	Scheduled	Sched Start	Sched End
ManualCollect_scre	45.0	-20.0	58.7	0.0	0.0	0.00	1.00	0.50	2	1	5.00	1-2		1-2	0.00	1.00
ManualCollect_scre	45.0	-20.0	58.7	0.0	0.0	90.00	91.00	0.50	2	101	5.00	101-102		101-102	90.00	91.00

The Scan Table is very flexible and can be customized to your particular needs. The table below describes the interaction with this table. Many of these functions are activated by right clicking, so they are not always obvious, so it is useful to read the following section carefully.

Mouse Action	Results
Right-click any column heading	To display a column, just click the column name. If you click a checked column, it toggles to unchecked and is not displayed. This allows you to customize the scan table to fit your needs. To close this dialog, just click somewhere outside this list.

Mouse Action	Results
	
Right-click "Images Scheduled" column heading	Right-click the column heading named **Images Scheduled**. In addition to the **All Column**s menu (shown above), a **Schedule All** menu appears (shown below). 
Right-click "Images Scheduled" field entry	A pop-up menu appears which allows you to: 
Right-click any other field entry	A pop-up menu appears. This feature is helpful in moving scans and when setting similar parameters for several different scans. 

Mouse Action	Results
	
Click and drag heading lines	Dragging a vertical column line allows the user to extend the column widths to view an entire columnar heading. Making columns narrow allows the user to fit more columns into the viewing area. Dragging on horizontal lines expands depth of the table.
Double click a field	In order to edit a field you should double click the field you want to change.
Tab	To get to the next field, you can just tab. Note that some of the fields are gray since they are read only fields.

The meaning of each field in the Scan Table is given below. Chi is replaced by Kappa for AFC-11K and AFC-12.

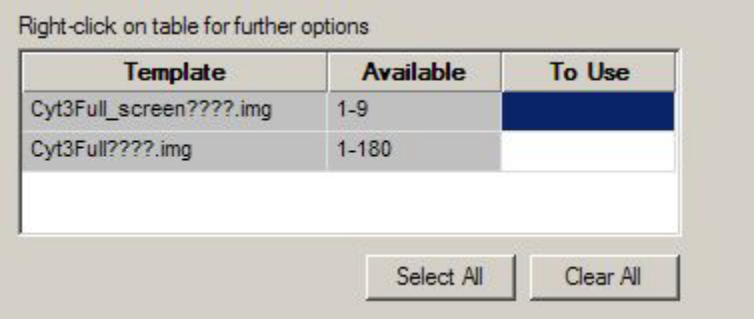
Column Heading	Description
Scan ID	Scan identification number.
Scan Template	A separate dialog pops up in which you enter the template *without* the ????.
Detector Distance	Value of the distance axis of the detector goniometer. Also referred to as the crystal-to-detector distance (in mm).
2θ	Value for 2θ axis of the detector goniometer (in degrees).
Max Resln (deg)	Maximum resolution at edge of detector for specified distance (in Å or degrees depending on your settings in Preferences).
λ	(Wavelength) λ of the X-ray source specified in Å.
Beam X	Position of latest direct beam shot along the X-axis.
Beam Y	Position of latest direct beam shot along the Y-axis.
ω	Position of ω (Omega) axis of crystal goniometer (in degrees).
χ	Position of χ (Chi) axis of crystal goniometer – Replaced by **K** (Kappa) for AFC-11K and AFC-12 (in degrees).
ϕ	Position of ϕ (Phi) axis of crystal goniometer (in degrees).
Rotation Axis	Scan axis which is usally Omega (ω), or Phi (ϕ**)**. NOTE: Phi (ϕ) is normally used on the R-AXIS. Omega (ω) is used on the AFC8 and AFC9. Omega (ω) and Phi (ϕ) are used on the AFC7, AFC11, and Kappa goniometers.
Start Angle	Angle of scan start position for the rotation axis (in degrees).
End Angle	Angle of scan end position for the rotation axis (in degrees).
Width	Width of one image (in degrees).
Step	The angular distance between the start angles of two consecutive images in the scan. Step usually has the same value as Width, but it may different in for collecting separated screen images or other purposes.
Num Images	The number of images to be collected in the scan.
Completeness	Percent completeness of the data in that scan.
Start Num	Image number to assign to first image of scan.
Exp Time	Exposure time (in seconds or minutes).
Image Sequences	Images in sequence groups.

Column Heading	Description
Collected	Images already collected, if any.
Scheduled	Images currently scheduled for collection during next collection run.
Sched Start	Scheduled start angle position for images (in degrees).
Sched End	Scheduled end angle position for images (in degrees).

Scan Selection Table

Scan Selection Table Dialog Component

The Scan selection table does not appear on its own, but is a common component of many dialogs. Since it is closely related to the Scan State Display, it is described here.

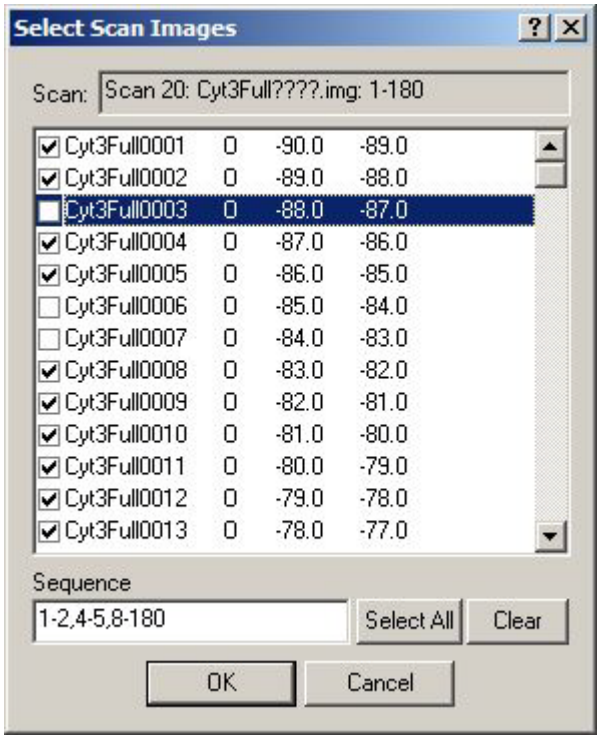


Each scan is represented by a row in this table. For this example there is one screen scan and one collection scan.

Parameter	Description
Template	This is the file name template for the particular scans. More than one collect scan may have a given template, but the **Available** numbers are different.
Available	Image numbers within the scan template are displayed in the **Available** column.
To Use	This is where you specify which of the images within this scan you want to use for the particular operation. For example if you wanted to use all the images in the collect scan in this example, you would type in 1-180 in **To Use**. There are other ways to fill in To User • You can double click the **Available** cell for that scan. This selects all the images that are available. • You can right click the **To Use** cell and click **Select Images**. This gives you the dialog shown below from which you can select the images you want • Another option for right click is **Select All** which selects all the available images for that scan. • The final right click option is **Clear** which clears the **To Use** cell for this scan.
Select All	Most instances of this dialog component have a **Select All** button which selects all available images from all scans.
Clear All	Most instances also have a **Clear All** button which clears the entire **To Use** column.

Select Images Dialog

This dialog is activated when you right click a **To Use** cell and then click **Select Images**.



Parameter	Description
Scan	This shows the scan number (an arbitrary integer identifier), the scan template, and the available images.
Image List	Each image is shown in a row of this list. Each row has a check box and includes the scan information (the O in the table above is for Omega, the scan axis it is not a numeric 0). You can check individual files, but if you just want to leave out some images, it is better to click **Select All** and then uncheck the images you don't want.
Sequence	This is the sequence of images that have been checked. When you click OK, this goes into the **To Use** column in the **Scan Selection Table**.
Select All	Check all the images.
Clear All	Uncheck all the images.

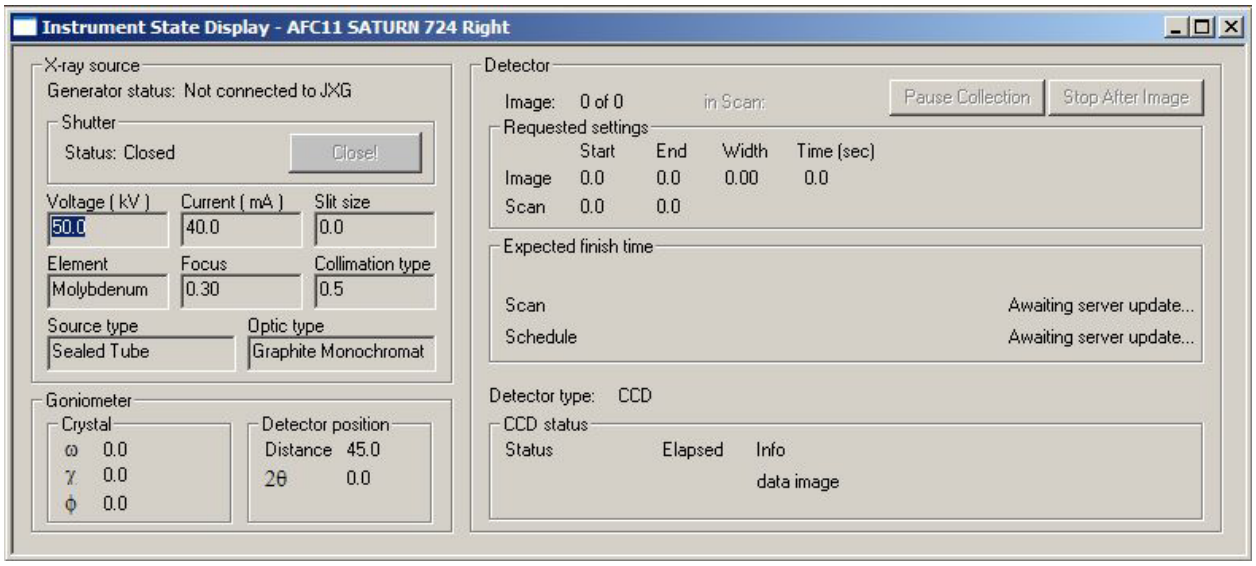
Instrument State Display

Overview

 The **Instrument State Display** is purely a viewer and has no interaction controls that allow you to change the state of the instrument. The one exception to this is that there is a button to allow you close the shutter in an emergency. It is common practice to display this viewer during data collection since it also gives expected finish times for the current scan and schedule. The instrument state display is somewhat different for CCD and image plate (R-AXIS, RAPID, etc) instruments.

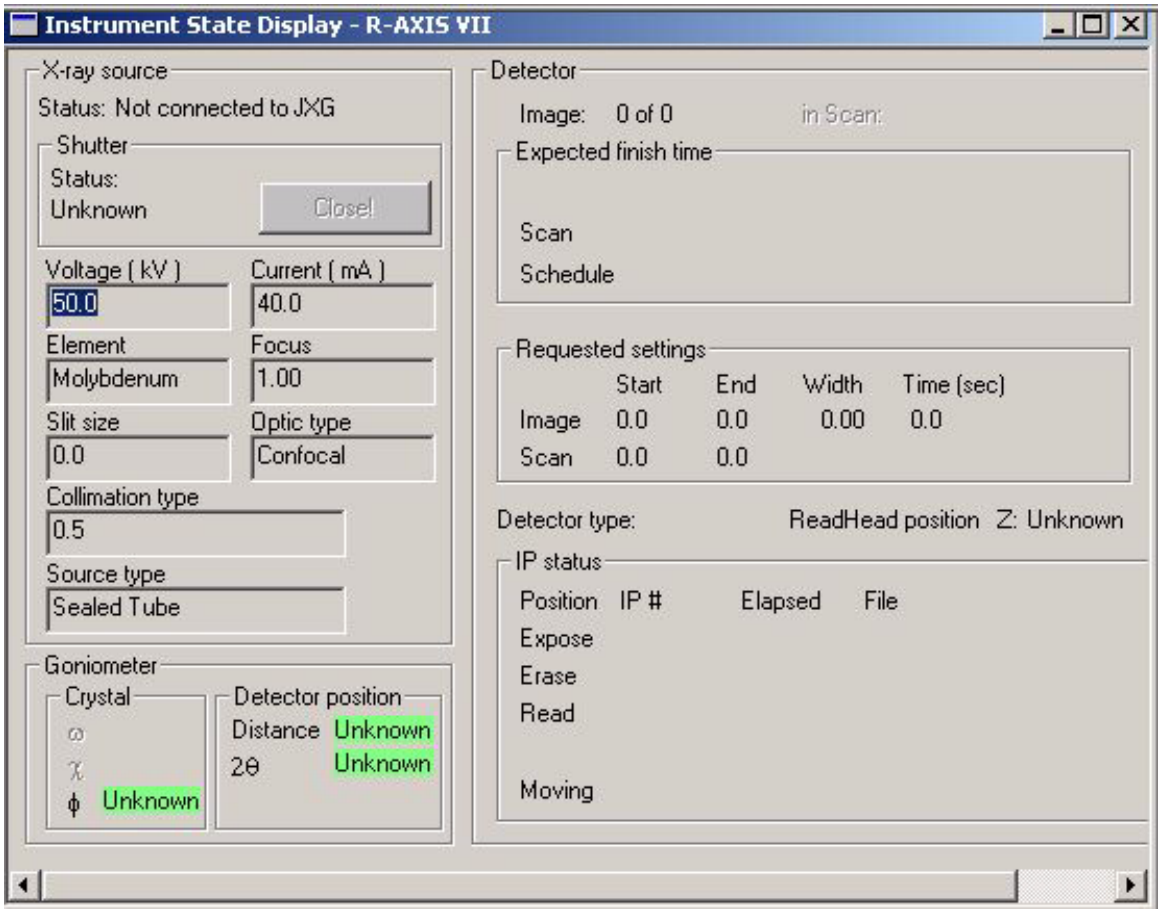
To control the low level operations of the instrument (e.g., to rotate a goniometer axis), use the **Manual Instrument Control** icon described in the next section.

CCD



Parameters		Description
X-Ray Source		
	Shutter Status:	• Open: Elapsed Exposure Time displayed and Close button enabled. • Closed: Enabled when Shutter Status Open. • Unknown.
	Voltage (kV)	Voltage setting of X-ray goniometer.
	Current (mA)	Current setting of X-ray goniometer.
	Slit Size	Size of Slits.
	Element	Target element producing X-radiation.
	Focus	X-ray generator focus type.
	Collimation Type	Type of collimation of attached optics.
	Source Type	Type of X-ray generator.
	Optic Type	Type of optics system.
Goniometer		
	Crystal	Current positions and names of crystal goniometer axes.
	Detector Position	Current names and positions of detector axes (e.g., Distance and 2Theta)
Detector		
	Image	Current image being collected and total images in current scan.
	Requested Settings	Various information about current image and scan being collected.
	Expected Finish Time	Expected finish time for current image and current scan.
	Pause Collection	This pauses data collection after the current image has completed. The button is then renamed and allows you to resume data collection. You do not have to re-initialize to resume.
	Stop After Image	This stops the current schedule after the current image has been collected. This is the preferred way to stop a data collection (e.g., the crystal has died or slipped out of center) since you do not need to re-initialize to continue on with the next sample or to restart the collection on the current schedule.
	Detector Type	Type of detector.
	CCD Status	Current action being performed by CCD, elapsed time of action, miscellaneous information.

R-AXIS



The parameters for this are similar to the CCD version, but there are additional parameters for the status of the image plates.

Parameters		Description
X-Ray Source		
	Shutter Status:	• Open: Elapsed Exposure Time displayed and Close button enabled. • Closed: Enabled when Shutter Status Open. • Unknown.
	Voltage (kV)	Voltage setting of X-ray goniometer.
	Current (mA)	Current setting of X-ray goniometer.
	Slit Size	Size of Slits.
	Element	Target element producing X-radiation.
	Focus	X-ray generator focus type.
	Collimation Type	Type of collimation of attached optics.
	Source Type	Type of X-ray generator.
	Optic Type	Type of optics system.
Goniometer		
	Crystal	Current positions and names of crystal goniometer axes.
	Detector Position	Current names and positions of detector axes
Detector		
	Image	Current image being collected and total images in current scan.
	Requested Settings	Various information about current image and scan being collected.

Parameters		Description
	Expected Finish Time	Expected finish time for current image and current scan.
Detector Type		Type of detector.
	Read Head Position	Current X and Z positions of read head.
	IP Status	Current status of IP: position, expose, erase, read, decay, and moving elements.

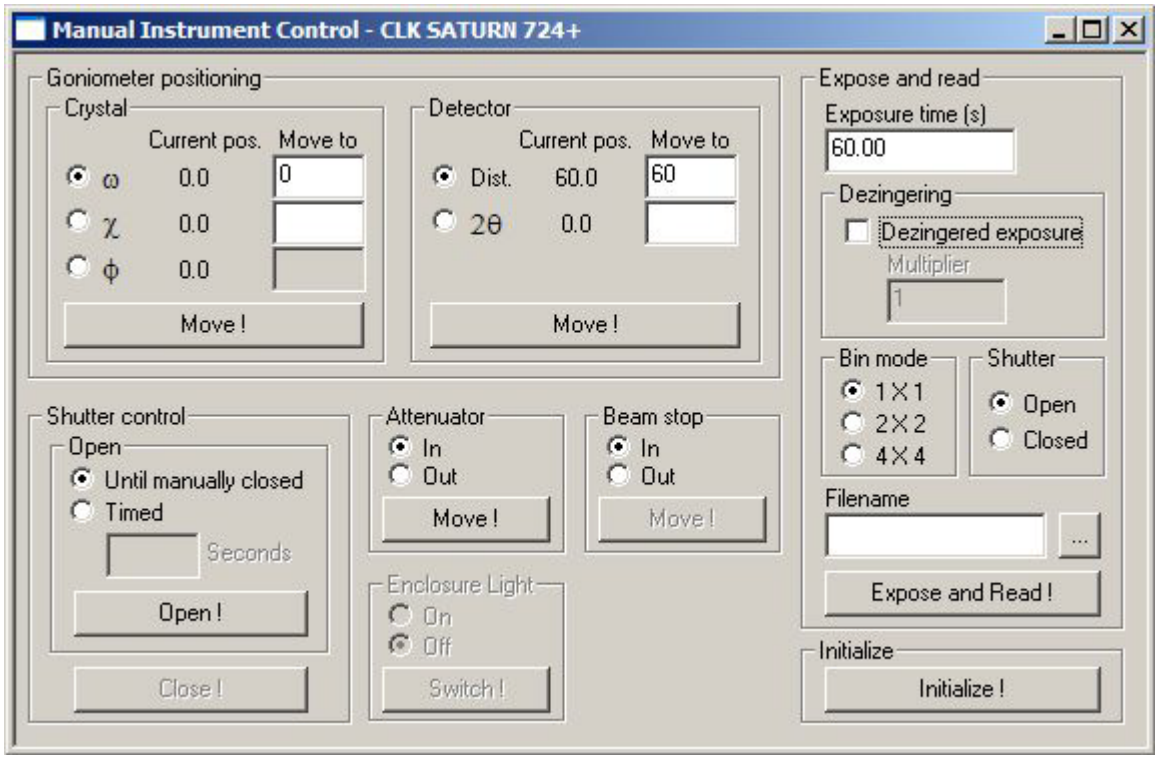
Manual Instrument Control

Overview

The Manual Instrument Control viewer functions both to report the current status of the instrument and to control the settings of the goniometers and detector positions. It can also be used for collecting single images. Like the Instrument State Display, there are different forms of this viewer depending on your instrument type.

This viewer is intended primarily for installation and maintenance functions, but depending on your instrument it can be useful to move to a position where it is easier to mount the crystal or perform some other function. You have to have administrator privileges to use the manual instrument control.

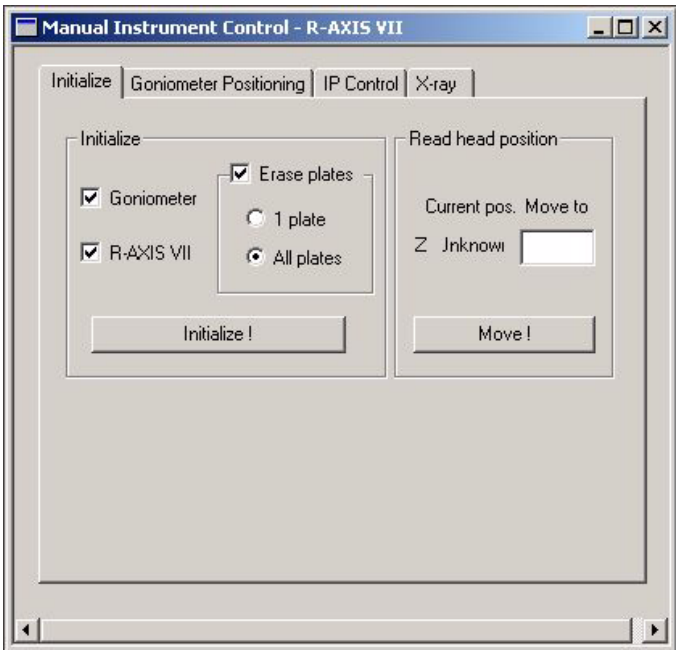
CCD



Parameter		Description
Goniometer Positioning		
	Crystal:	Displays current positions of crystal goniometer axes and allows for positioning of goniometer axes. You can only move one axis at a time, so you first need to select the axis you want to move by clicking on the radio button next to it. You then need to enter the new value for that axis. Note that the **Move!** button does not become active until you have entered a value into the edit box. So if you have initialized and it is still deactivated you just need to enter a value and it becomes enabled.
	Detector	Displays current positions of detector axes and allows for positioning of detector axes.
Shutter Control		
	Open	Specifies Open Shutter control • Until manually closed - the shutter remains open until **Close!** button is clicked. • Timed - X-ray shutter remains open for specified time (in seconds) or until the **Close!** button is clicked.
	Open!	Click button to open the shutter.
	Close!	Click button to close the shutter.
Attenuator		Indicates if the attenuator is placed In or Out of X-ray beam.
Beam Stop		Indicates if the beam stop is placed In or Out of X-ray beam.
Expose and read		
	Expose time	Allows a change in the Expose time (in seconds).
	Dezingered exposure	Allows Dezingered exposure. Stipulate the Multiplier.
	Bin mode	Specify the binning mode. • 1X1 • 2X2 • 4X4
	Shutter	It is sometimes useful to take images with the shutter closed, so this control gives you the option.
	Expose and Read!	This activates the exposure cycle.
Initialize		Nothing else in this dialog works until the instrument has been initialized.

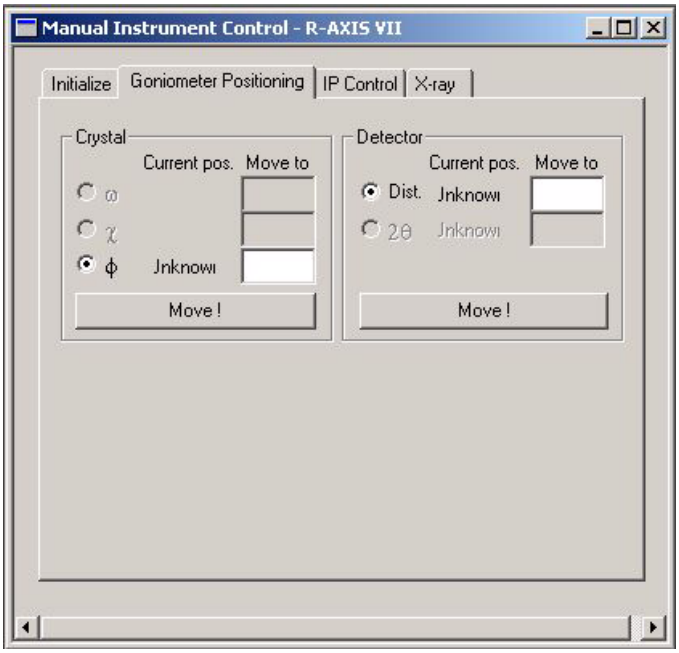
R-AXIS HTC

Initialize Tab



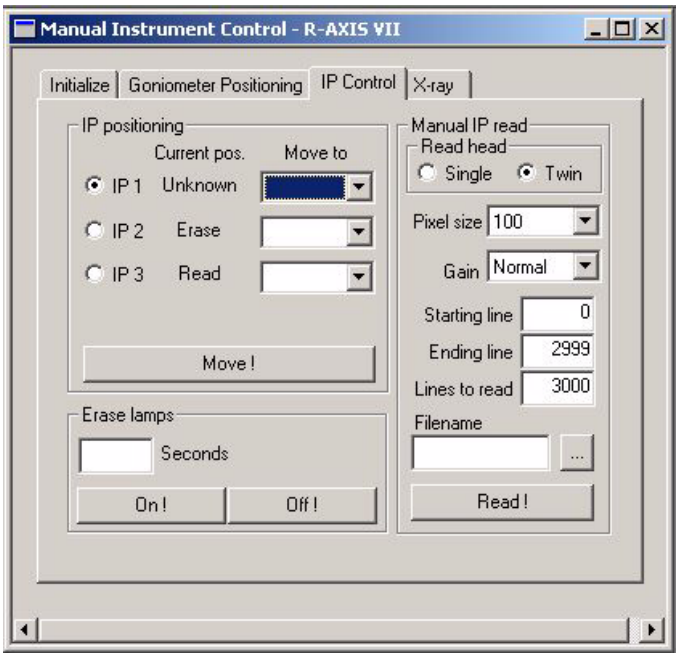
Parameter		Description
Initialize		Displays Initialize parameters to be included. The Instrument State Display appears during initialization
	Goniometer	If Goniometer is selected, it is included in Initialize
	R-AXIS	If selected, the detector is included in Initialize.
	Scanner*	If Scanner is available and selected, it is included in Initialize.
	Erase Plates	If Erase Plates is selected, plates are erased during Initialize. Specify: • 1 plate • All plates
	Initialize!	Click the button to complete the initialize.
Read head position		Displays Current Position of Read Head. Allows for input of a position to move to. This should only be done by a trained Rigaku service person.
	Move!	Click button to complete the move.

Goniometer Positioning Tab



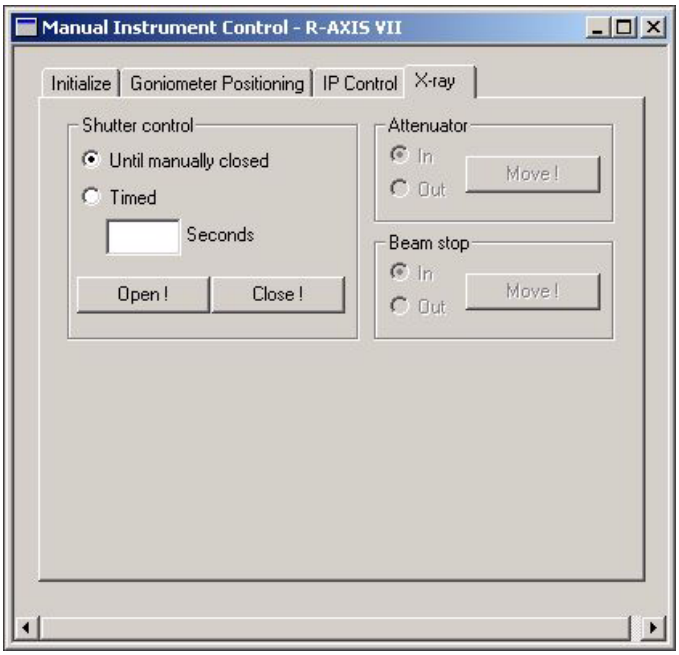
Parameter		Description
Crystal		Displays Current positions of crystal goniometer axes and allows for re-positioning of goniometer axes.
	ω	Move the omega axis of crystal goniometer (in degrees). The new position is entered in the edit box next to the selected axis.
	χ	Move the chi axis of crystal goniometer (in degrees).
	ϕ	Move the phi axis of crystal goniometer (in degrees).
	Move!	Perform the move.
Detector		Displays Current positions of detector goniometer axes and allows for re-positioning of goniometer axes.
	Dist	Move the distance axis of the detector goniometer. Also referred to as the crystal-to-detector distance (in mm).
	2θ	Move the value of 2θ axis of the detector goniometer in degrees).
	Move!	Perform the move.

IP Control Tab


Manual Instrument Control - R-AXIS VII
Initialize
Goniometer Positioning
IP Control
X-ray
IP positioning
Current pos.
Move to
IP 1
Unknown
IP 2
Erase
IP 3
Read
Move !
Erase lamps
Seconds
On !
Off !
Manual IP read
Read head
Single
Twin
Pixel size
100
Gain
Normal
Starting line
0
Ending line
2999
Lines to read
3000
Filename
...
Read !

Parameter		Description
IP positioning		
	IP 1*	Click one of these buttons to select the IP to be controlled. The current position of the IP is displayed. The **Move** drop down selects a new position for the IP. You and also select an **Erase** or **Read** operation from these drop downs.
	IP 2*	
	IP 3*	
	IP 4*	
	Move!	Click button to perform the actual move and selected operation.
Erase Lamps		Display Erase lamps (in seconds).
	On!	Click button to turn Erase lamps on.
	Off!	Click button to turn Erase lamps off.
Manual IP read		Make desired changes to the Manual IP read parameters.
	Read head	Specify Read head: • Single • Twin
	Pixel size	Indicates Pixel Size of image to be read. Click the down-arrow to change the selection: • 100x100 • 100x150 • 200x200
	Gain*	• Normal • High
	Scan area*	Displays Scan area. Click the down-arrow to change the selection: • Narrow • Wide
	Starting line	First line in image read.
	Ending line	Last line in image read.
	Lines to read	Number of lines in image read.
	Filename	Name of file written for image read. Click the  button to access the Open Image File dialog.
	Read!	Click the **Read!** button to complete the read.

X-ray Tab



Parameter		Description
Shutter Control		
	Until manually closed	Shutter remains open until **Close!** button is clicked.
	Timed	X-ray shutter remains open for specified time or until **Close!** button is clicked. Specify time (in seconds).
Attenuator*		Indicates if the **Attenuator** is placed **In** or **Out** of X-ray beam.
	Move!	Click button to complete the **Move!**
Beam stop*		Indicates if the **Beam stop** is placed **In** or **Out** of X-ray beam.
	Move!	Click button to complete the **Move!**

R-AXIS II Controls

Parameter		Description
Laser shutter*		Click to Open Laser shutter (R-AXIS II)
Oiler*		
	Rotate*	Specifies Counterclockwise or Clockwise rotation of read head. Also indicates number of Pulses of oiler (*R-AXIS II only*).
	Check Oil Status*	Click to Check Oil Status to see if the read head needs to be oiled.
	Oil Read Head*	Click to Oil Read Head when necessary.
Initialize*		Initializes the instrument.
	Erase Plates*	If Erase Plates is selected, IP plates are erased during initialization.

** Parameter included only on some instruments.*

Log File Viewer

Overview

 Log files are shown when the various steps are run by the processing suites. However, it is sometimes useful to go back to look a specific log file to analyze the processing results. When you click this button, a file open dialog comes up and allows you to choose the log file you wish to open. You can simply double click this file and it is opened in a viewer that allows you to scroll through the file.

*d*TREK*

OPERATION	LOG FILENAME
Find Spots	dtfind.log
Refine Spots	dtrefine.log
Index Spots	dtindex.log
Predict Spots	dtpredict.log
Integrate Reflections	dtintegrate.log
Num Abs Corr	
Merge Reflections	dtreflectionmerge.log
Scale and Average	dtscaleaverage.log
Rank Crystal	dtranker.log
Strategy	dtmulitstrategy.log
Laue Check	dtcell_laue.log
Centricity	dtcell_cent.log
Spacegroup Check	dtcell_sgrp.log

TwinSolve

OPERATION	LOG FILENAME
Find Spots	tsFind.log
Refine Spots	tsRefine.log
Rinal Refine	tsFinalRefine
Index Spots	tsIndex.log
Predict Spots	tsPredict.log
Integrate Reflections	tsiIntegrate.log
Num Abs Corr	tsNumabs.log
Reject & Average	tsRejectAverage.log
Scale & Shape	tsScaleShape.log
Laue Check	tsLaueCheck.log
Spacegroup Check	tsSpacegroupCheck.log
Get Twin Relations	tsTwinRelations
Update Reflection File	tsUpdateReflectionFile.log

FS_PROCESS

OPERATION	LOG FILENAME
Find Spots	prfind.log
Refine Spots	prrefine.log
Index Spots	prindex.log

OPERATION	LOG FILENAME
Predict Spots	prpredictspots.log
Integrate Reflections	printegrate.log
Num Abs Corr	
Merge	merge.log
Scale	scale.log
Post Refine	post.log
Average	average.log
Peak Search	psearch.log
Refine	refosc.log
Integrate	integ.log

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5. Small Molecule Tasks

There are two different small molecule modes. **Auto** mode is designed for automatic data collection, processing, and structure solution. It is targeted especially for the SCXmini and XtalLABmini and is only enabled for those two instruments. **Expert** mode is designed for the crystallographer who wants more detailed control of the instrument and procedure. In previous versions of CrystalClear what is now Auto mode was called SCXmini mode. In CrystalClear 2.0 Expert mode works on any instrument including SCXMinin and XtalLABmini.

Auto mode uses d*TREK to index the cell and then FS_PROCESS to integrate the data. There are four different tasks available in Auto mode.

The four tasks are Screen Collect and Process, Collect and Process, Collect and finally Process

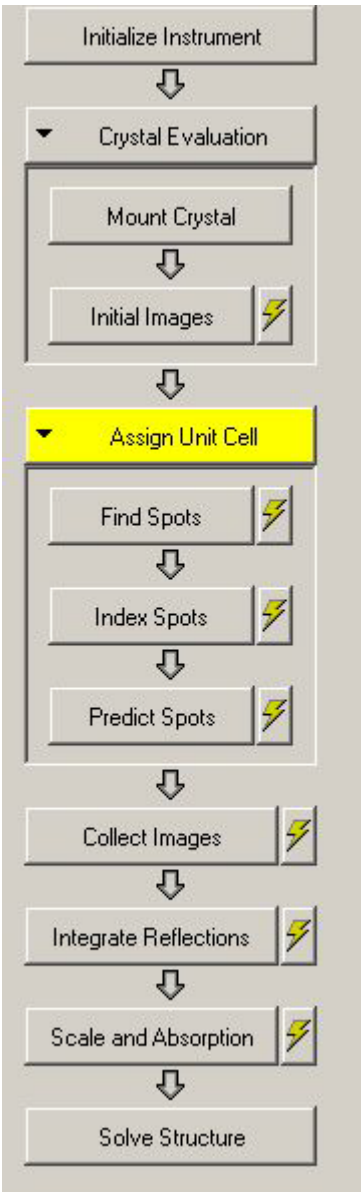
In Expert mode you have your choice between d*TREK, FS_PROCESS, and TwinSolve to process the data. TwinSolve is available under a separate license and is most suited to processing data from twinned crystals. You also have the choice of four different tasks.

This chapter contains quick start guides for Auto mode and Expert mode for d*TREK on a CCD, d*TREK on a Rapid, and TwinSolve.

Auto Mode

Screen Collect and Process

The **Screen Collect and Process** task is used when you want to automate the entire process of collect and processing you crystal. The flow bar is shown below.

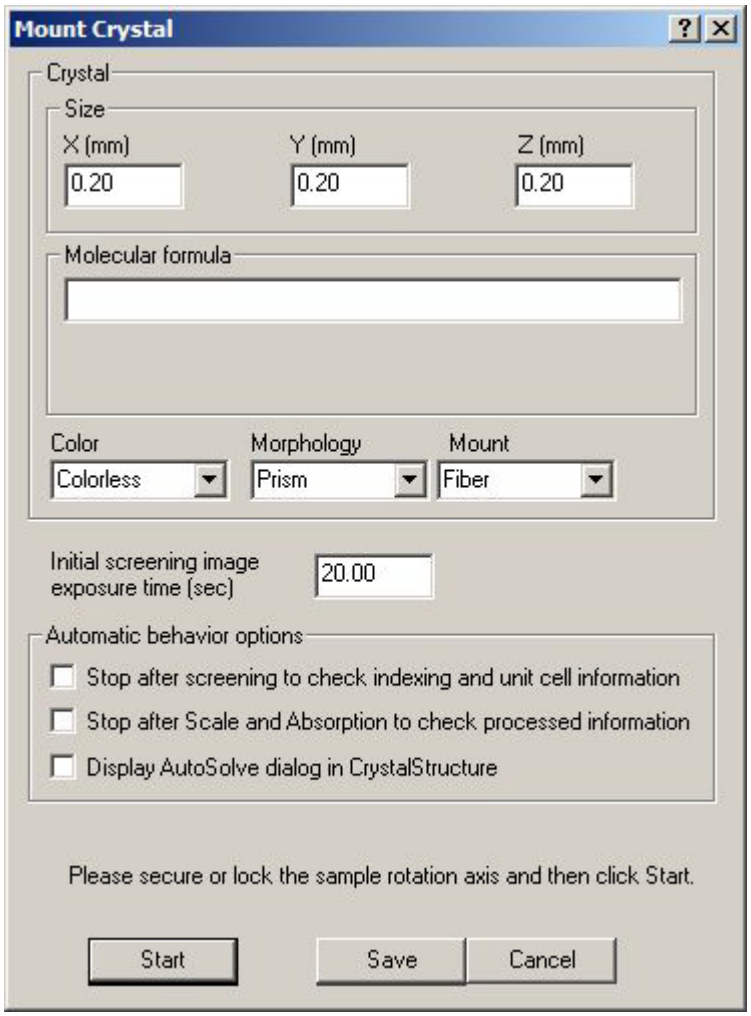


The steps in this flow bar are executed automatically using the default parameters for each step. If you want to change these default parameters you can just click the step manually, enter the new parameters, and then use the **Save** button to save the defaults to the desired level. See the section in Chapter 3 on Defaults.

The **Mount Crystal** step contains the controls for automated processing. This step is just a reminder for other modes, but for Auto mode it is critical. Note also that there is no **Setup** step in Auto mode since a simplified version of this is found in **Mount Crystal**.

Initialize Instrument. Before you can collect data, the goniometers and detector must be initialized. If the instrument is already initialized (e.g., when you opened the sample) you can skip this step. See chapter 7 for more details.

Mount Crystal. This is the key step for automating data collection and processing and since it is very different for Auto mode than it is for other mode of CrystalClear, it is documented in this section.



Parameter	Description
Size (X, Y, Z)	If you already know the size of your crystal, you can just enter it here. If not, you may wish to run **RaxShape** through the **View Crystal** icon in the top toolbar.
Molecular formula	The molecular formula is vital if you intend to go on to structure solution. CrystalClear accepts the formulas such as C9H13O5N3.
Color	Color of the crystal. You may enter the color name or use a drop-down list which provides predefined colors. The predefined color choices are: • Blue • Colorless • Gray • Green • Orange • Red • White • Yellow
Morphology	Specify the Morphology of the crystal. You may enter the name in the

Parameter	Description
	field, or use a drop-down list which provides predefined morphology types. The predefined choices are: • Block • Chip • Chunk • Platelet • Prism
Mount	Type of Mount used for the crystal. You may enter the mount type in the field, or use a drop-down list which provides predefined mount types. The predefined mount choices are: • Fiber • Capillary • Loop
Initial screen images exposure time	The time you enter here is in seconds and specifies the exposure time for the screen images. If you know your crystal is week, you might want to make the initial time longer, if it is a very strong diffractor you may want to shorten the time.
Automatic behavior options	Once you click Start, the steps in the flow bar executes automatically. You may choose any of three different check points for CrystalClear to stop and allow you to examine preliminary results. • **Stop after screening to check indexing and unit cell information**. If you want to make sure this is a good crystal before you collect it, you may want to put a stop here. This also allows you to change the exposure time for **Collect**. • **Stop after Scale and Absorption to check processed information**. You should check this if you do not want to attempt to solve the structure automatically or if you would like to check the processing results (e.g., Rmerge) before you go on. • **Display AutoSolve dialog in CrystalStructure**. Normally, the automatic structure solution proceeds with the default parameters. By having it bring up this dialog this effectively stops the automatic execution at this point and also allows you to specify different parameters for the AutoSolve.
Start	This button starts the automatic execution of the steps in this flow bar. Depending what Stop boxes you checked, the data is collected and processed automatically unless in encounters and error.

Initial Images. It is usually useful to collect a set of screen images to assess the crystal quality so that you can make sure the crystal diffracts and is good enough to warrant data collection. You can also index the crystal and use the symmetry information to either manually select the appropriate collection schedule or run Strategy to calculate a collection schedule specifically for your sample. See Chapter 7 for a detailed description.

Find Spots. This step identifies spots to be used for indexing. In Auto mode the d*TREK version of this step is used. The parameters available are described in Chapter 8.

Index Spots. Indexing is also performed by d*TREK in Auto mode. The index parameters are described in Chapter 8. There is no explicit Refine step in Auto mode, but FS_PROCESS does an implicit refinement of the indexed cell during integration

Predict Spots. This step predicts and displays reflections. This gives you a visual check that the indexing is right, but does not change the state of the sample. Since the integration is done using FS_PROCESS, the FS_PROCESS version of **Predict Spots** is used.

Collect Images. The collection schedule defaults to three scans that give you a complete data set. If you have high symmetry you can remove one or two of these scans. See chapter 7 on Data Collection.

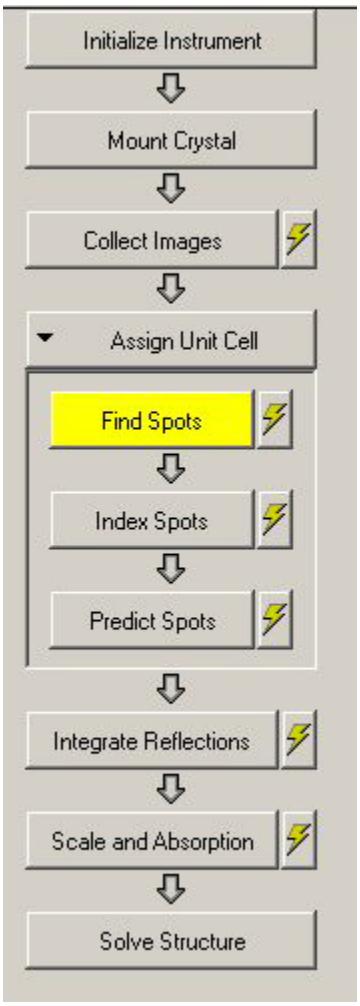
Integrate Reflections. In Auto mode, FS_PROCESS is used for the integration step. This step is described in more detail in Chapter 9.

Scale and Absorption. This is a FS_PROCESS step as described in Chapter 9. This step also does a Laue check, averaging, and a final cell refinement. Note that the default is Numerical absorption correction which requires the polygonal cell boundaries from RaxShape.

Solve Structure. The structure solutions step, which is new to version 2.0, requires that you have CrystalStructure 4.0 or above properly licensed and installed. Unless you have checked on the **Display AutoSolve Dialog in CrystalStructure** box, CrystalClear causes CrystalStructure to automatically execute the AutoSolve operation. CrystalStructure remains running so that you can examine and further process the solved structure. AutoSolve requires at least an approximately correct molecular formula.

Collect and Process

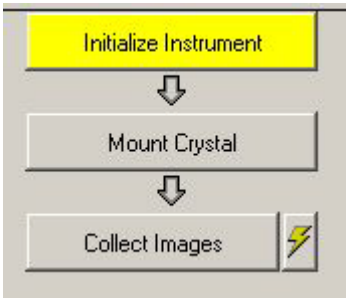
The flow bar for Auto mode **Collect and Process** is shown below



In the Auto mode Collect and Process task, there are no screen images and the Initial Images step is replaced by the Collect Images step. Thus Find and Index are performed using collected data images rather than the initial screen images. Otherwise the steps are identical to the Screen Collect and Process steps described in the last section.

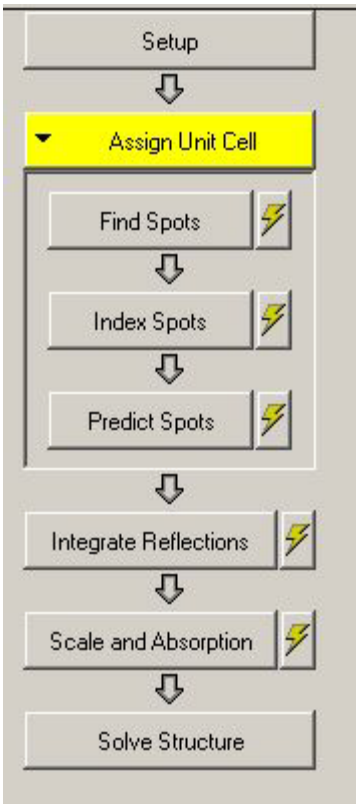
Collect

The Auto mode Collect task collects with the default schedule. The Auto mode version of the Mount dialog is displayed so that you can start this process (see description under Screen Collect and Process).

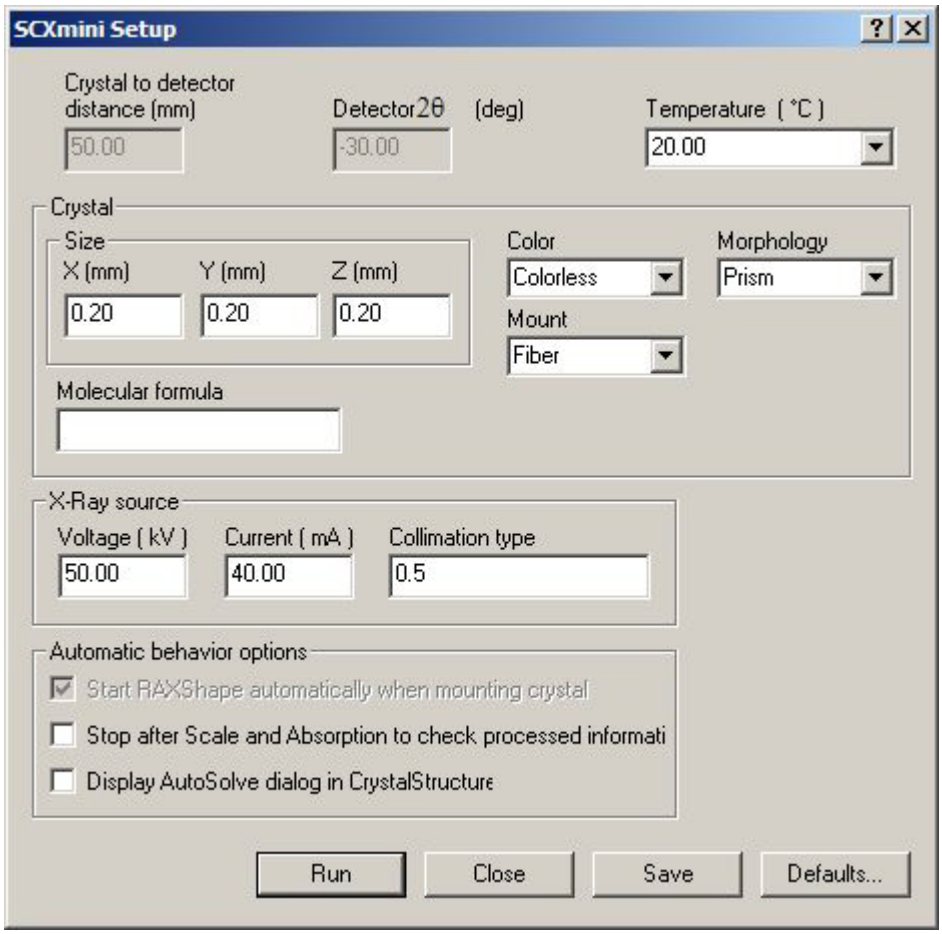


Process

The Process task in Auto mode is for automatically processing previously collected data. Since there is no data collection, it does not have the Auto mode Mount dialog to kick off the automation, but it does have its own version of Setup which is described in this section.



Setup. In the Auto mode Process task, the Setup step has a special dialog that replaces the normal Setup dialog.



Parameter	Description
Crystal to detector distance	The distance is taken from the image headers and is provided for informational purposes here. It is a read only control and the units are millimeters.
Detector 2θ	The 2θ value is taken from the image header also. It is in degrees.
Temperature	This is a Celsius value for the temperature at which the data was collected.
Size (X, Y, Z)	If you already know the size of your crystal, you can just enter it here. If not, you may wish to run **RaxShape** through the **View Crystal** icon in the top toolbar.
Molecular formula	The molecular formula is vital if you intend to go on to structure solution. CrystalClear accepts the formula in a variety of forms.
Color	Color of the crystal. You may enter the color name or use a drop-down list which provides predefined colors. The predefined color choices are: • Blue • Colorless • Gray • Green • Orange • Red

Parameter	Description
	• White • Yellow
Morphology	Specify the Morphology of the crystal. You may enter the name in the field, or use a drop-down list which provides predefined morphology types. The predefined choices are: • Block • Chip • Chunk • Platelet • Prism
Mount	Type of Mount used for the crystal. You may enter the mount type in the field, or use a drop-down list which provides predefined mount types. The predefined mount choices are: • Fiber • Capillary • Loop
X-Ray Source	These are the X-Ray Generator settings for this sample that may not have been entered when the data was collected. So you have another chance to enter them.
Automatic behavior options	Once you click Run, the steps in the flow bar executes automatically. You may choose any of two different check points for CrystalClear to stop and allow you to examine preliminary results. • **Stop after Scale and Absorption to check processed information**. You should check this if you do not want to attempt to solve the structure automatically or if you would like to check the processing results (e.g., Rmerge) before you go on. • **Display AutoSolve dialog in CrystalStructure**. Normally, the automatic structure solution proceeds with the default parameters. By having it bring up this dialog this effectively stops the automatic execution at this point and also allows you to specify different parameters for the AutoSolve.
Run	This button starts the automatic execution of the flow bar. Depending on the Stop boxes you checked, the data is collected and processed automatically unless an error is encountered.

Once you have click Run, the rest of the steps are executed automatically until the processing is completed unless an error occurs. The processing steps are the same as described in Screen Collect and Process for this section.

Auto Mode Quick Start Guide

Parameters

The values given for various parameters are suggestions, and may not be appropriate for all samples. Feel free to adjust certain parameters in order to evaluate their effect on the results. For parameters not listed here, start with the default values.

Getting Started

- Check the X-ray power settings with the JXG program.
- Check that both of the green LEDs next to the detector are on. If the power to the detector has been off, the second LED does not light until the CCD temperature has reached the set point (-40°C). The instrument cannot be used until this is reached.
- If a low-temperature device is attached, check the temperature and flow rates.
- Start CrystalClear, check the **New sample** box and enter a name for your sample. Select **Screen Collect and Process** as the Task, and press OK.
- Press **Yes** to initialize the instrument.
- ***WARNING:*** The **Door open** button must be pressed and the warning beeper heard before the door is opened. Failure to do so cuts the power to the X-ray generator. Close the door smoothly and firmly to avoid accidentally engaging this fail-safe feature.

Setup

Enter the molecular formula and check the temperature, color, etc. in the various fields in the Setup dialogue. (The “size” fields can be skipped for now.) The **Start RAXShap**e box should be checked. The Run button starts the video camera and crystal-centering routine.

Crystal Centering and Shape Measurement

Mount the crystal while watching the video screen to get the sample close to the center, then adjust the x, y, and z translations until the center of mass of the crystal no longer moves when rotated. The video output can display circles (CTRL-M) that are helpful. See the RAXShape Quick-start Guide for details.

Initial Images

Adjust the exposure time for the screening images: larger/better crystals require less time than smaller/poorer crystals. Exposure times can be as small as 2 seconds and as long as 300 seconds. The default screen scan creates only 6 images; for crystals that produce very few reflections per image, more images may be required. The **Step** is the number of degrees skipped between images, and if more images are required then the step can be reduced. (If the **Step** is not visible, right-click one of the column headings, and select **Step** so that the column is visible.) Images can be re-collected by changing the numbers in the **Scheduled** column.

The next few steps (under **Assign Unit Cell**) occur in succession without stopping. To stop at each step, click the small arrow between the steps in the flow bar; the arrow changes to a stop sign and the next step does not execute automatically. You can still run the next step just by clicking on the button.

Find Spots

The **Find Spots** step normally run automatically. By default the screen images are used and the Find Spots routine attempts to locate all reflections having I/σ(I) greater than 5.0. For very weak samples it may be necessary to reduce this value to 4 or even 3. For very strong samples, larger values (10 to 25) may be used to filter out spurious reflections.

Index Spots

This step is also automatic. The default parameters give good results for good crystals. If you suspect a very long axis, change the "Max cell length" (on the Advanced tab) to 100. **Beam check** and **Deice** should *not* be checked. For troublesome samples try using the **Diffs** option. A higher I/σ(I) value may be selected here as in the previous step (but a value lower than the one used in **Find Spots** is not useful).

Predict Spots

This step simply allows you to visually verify that the indexed unit cell accurately predicts the positions of all (or most) of the reflections. For poor/twinned/split crystals, there may be a number of spots that are not predicted, but in many cases the correct unit cell has actually been found and the reflections that are from satellite crystals or minor twin components can be safely ignored (although the structural results from such samples cannot be expected to be of high quality). In case most of the spots are *not* correctly predicted it is likely that the crystal is badly twinned, or split (or suffers from a serious problem such as modulation), and the results of the indexing are wrong. In these cases a search for a better sample is warranted.

Step through the images with the green arrows at the top of the image viewer window (not the ones above them in the main toolbar). By default, predictions are only produced for the first 5 images; you may select more images by opening the **Predict Spots** dialog. Use the zoom tool (or just right-click and drag on an area in the image) to get a closer look at the reflections. The contrast tool may also be useful for looking at higher resolution spots. Use the Reset Local Contrast button to refresh the contrast level.

Collect

The Collect dialog is similar to the Initial Images dialogue. Only the exposure time needs to be adjusted; other adjustments are optional. The default scans (three sets of 180 images) produce an almost complete sphere of data (i.e. a redundant triclinic data set). The safest strategy is to collect all three scans. However, monoclinic and orthorhombic data sets need no more than the first two scans, and tetragonal and higher-symmetry lattices need only one scan. As before, exposure time can be as low as 2 seconds per image and as high as necessary to produce observed data to a high resolution. (Note that exposure times longer than 300 seconds may not be useful.) To remove a scan, highlight the scan with a mouse click the row, then press the **Delete Scan** button.

Integrate Reflections

The Integrate Reflections and the Scale and Absorption steps run automatically when the image collection finishes unless the stop sign has been inserted after Collect. The default resolution limit for integration is 0.77Å (or $2\theta = 55°$ with Mo radiation). If a higher or lower resolution limit is desired this can be changed before the integration step starts, or the integration step can be run again with new parameters. Note that

resolution arcs can be displayed on any image by selecting **View → Resolution Arcs**. Possible changes to the default parameters are discussed in the **Common problems** section below.

Scale and Absorption

The **Scale and Absorption** step is actually a large number of steps combined into one menu. Several scale factor refinements, a Laue group check, an absorption correction, a final cell refinement and the output of intensity data are all included. Parameters for each step are set in sub-menus, but the default values are used in the automatic procedure. The user should look through the log file that is created, paying attention especially to the section headed by the title **Laue Class**. When this step finishes, a summary of the results appears; if the **Accept** button is pressed the automatic structure solution step starts. The **Common problems** section below suggests alternate parameter values for difficult cases.

Common Problems and Possible Solutions

During Integration:

- Note that all data sets are handled as Laue class **-1** (triclinic) during the integration step. Even when the symmetry seems obviously higher, the **-1** Laue class should be used at this point.
- If you notice that a lot of the integration boxes are overlapped by adjacent boxes during integration (common when the unit cell has one or more long axes), it is necessary to change the behavior of the automatic box size routine. This problem often leads to very low values of completeness since many reflections are rejected (the ideal value for completeness is 100%). On the Advanced tab of the Integrate Reflections dialogue, the integration box size can be changed from **Auto** to **Fixed** and the *x* and *y* dimensions specified explicitly. To determine an appropriate box size, run the Predict Spots step, then press the **Set Reflection Size** button on the toolbar. Different values can be tried and then set so the box size is slightly larger than the reflection size. You may want to use the measure pixels tool to estimate spot size.

During Scale and Absorption:

- If this step fails at the very beginning with a **Scale failed** message, bring up the dialogue and uncheck the first **Scale** check box (third check box from the top in the **Programs** section). This error occurs because the first scaling step uses only **-1** symmetry and shorter data sets (one scan) may not contain enough Friedel opposites for scaling.
- The Laue check commonly reports a Laue group that is of lower symmetry than expected. Carefully inspect the Laue Check section of the log file. The Laue class is determined by choosing the class that provides a low R_{merge} value. The default cutoff is 15%. (R_{merge} is the agreement index for symmetry-related reflections and is therefore different for each possible Laue group.) You may notice that a particular Laue group actually passes the R_{merge} test but is still rejected. This is usually because one of the symmetry checks fails the **Correlation** check. The default minimum correlation is 0.95; inspect the table in the log file to look for problems — especially an operation that passed the R_{merge} test but has a correlation of something just less than 0.95. You can change the minimum correlation value on the Advanced tab of the dialogue (in the **Laue** section) and run the step again.

Expert Mode using d*TREK

In Expert mode you have full control of the instrument and the processing procedure. In most cases when one step completes the next step's dialog is popped up automatically so that you can just fill in the desired parameters and run that step. If you want to skip that step or go back to a previous step, you just have to

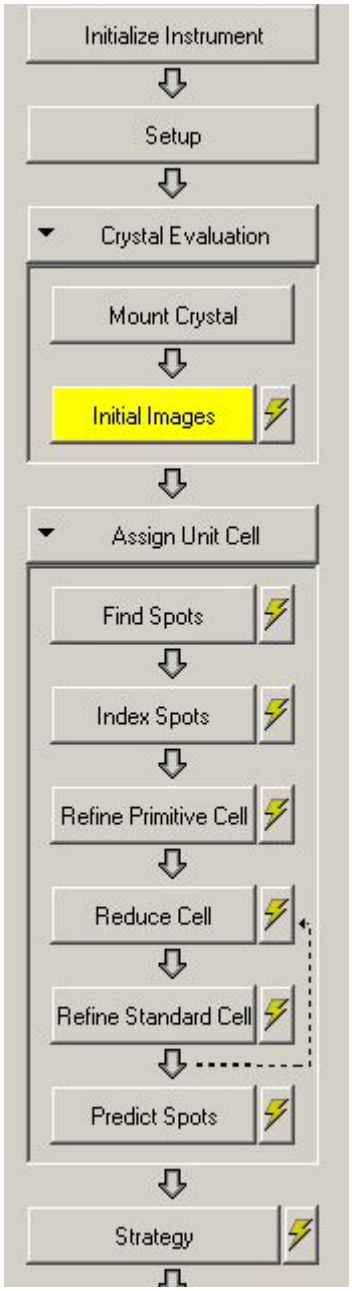
click the step you want to run and it automatically closes the open dialog and open the dialog for the new step.

The d*TREK processing suite is best suited to narrower images (e.g., under 2 degrees). It has a separate step for refining the orientation matrix values, unit cell parameters, and detector offsets. There is also a **Strategy** step which computes and optimizes collection schedule.

Screen Collect and Process

The Screen Collect and Process task is used when you want to collect and process a data set in one session. It is possible to integrate the data as it is being collected.

The flow bar for this task is fairly complex, so we break it down into two parts. The first part covers through Strategy and the second part covers from Collect Images on.



Initialize Instrument. Before you can collect data, the goniometers and detector must be initialized. This can also be done when the sample is opened or through the Manual Instrument Control. If the instrument is already initialized you can skip this step. See chapter 7 for more details.

Setup. This step allows you to specify information about the sample. If you are just starting to use this instrument, you should check the X-Ray Source tab to make sure that it is right. If it is not, you should set the values appropriately and then click **Save to Config**. See chapter 7 for more details. Setup is particularly important for small molecule work. Many of these values go into your CIF. But most importantly, you must have a valid molecular formula for structure solution.

Mount Crystal. This is really a pseudo step to remind you to mount the crystal and lock down phi. Once you have done this click OK and go on to Initial Images.

Initial Images. It is usually useful to collect a set of screen images so that you can make sure the crystal diffracts and is good enough to warrant collection. You can also index the crystal and use the symmetry information to either manually select the appropriate collection schedule or run **Strategy** to automatically determine an optimized collection schedule. You need to collect enough screen images to allow the processing software to index. For d*TREK this means that you need a few dozen spots. See Chapter 7 for a detailed description.

Find Spots. This step identifies spots to be used for indexing. The parameters available are described in Chapter 8.

Index Spots. Indexing determines the cell parameters and metric symmetry. If you know what the cell should be, this can be a good check that this is a good crystal. This also gives you a reasonability check. If the cell parameters do not make sense, it is an indication that something is wrong. The parameters for indexing are described in Chapter 8.

Refine Primitive Cell. For small molecule work, refinement proceeds in two stages. The first step refines the primitive triclinic cell. This uses the standard refinement step described in Chapter 8, but with parameters setup for refining the primitive triclinic cell.

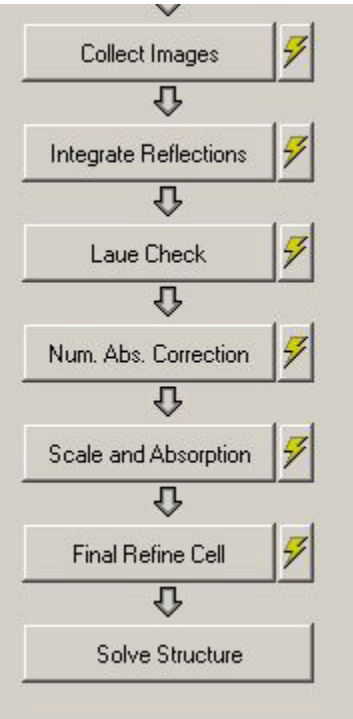
Reduce Cell. After the initial primitive cell refinement, the next step is to reduce the cell. The dialog for doing this is shown in Chapter 8.

Refine Standard Cell. Once the cell has been reduced, the second stage of refinement is performed. This is the same dialog as shown in Refine Primitive Cell, but the defaults are setup differently and it now works on the standard cell.

Predict Spots. This step predicts and displays reflections. This gives you a visual check that the indexing is right, but does not change the state of the sample.

Strategy. Strategy suggests a collection schedule based on the cell parameters and symmetry and completeness and redundancy requirements provided through the interface. If you prefer to specify the collection schedule manually, you can skip this step. Strategy is only available for the d*TREK suite and is documented in Chapter 8.

The second part of this flow bar is shown below.



Collect Images. This step performs the actual data collection. If you have run **Strategy**, the results of that step is a strategy collection schedule. Alternately, you can select and edit other collection schedules. If you are using the d*TREK processing suite, you can start the **Integrate Reflections** step as soon as the **Collect Images** step has started. The details for this step are in Chapter 7.

Integrate Reflections. This can either be done while the images are being collected or after collection has finished. Chapter 8 provides the details for the parameters that control integration.

These next three steps are optional steps to check the cell symmetry of the diffraction pattern and determine the space group using the integrated data.

Laue Check. This step checks the Laue group. See Chapter 8.

Centricity Check. See Chapter 8.

Space Group Check. See Chapter 8.

Num Abs. Correction. This step makes a numerical absorption correction and prepares the data for **Scale and Average**. This type of correction requires the shape of the crystal to have been saved through RAXShape. See Chapter 8.

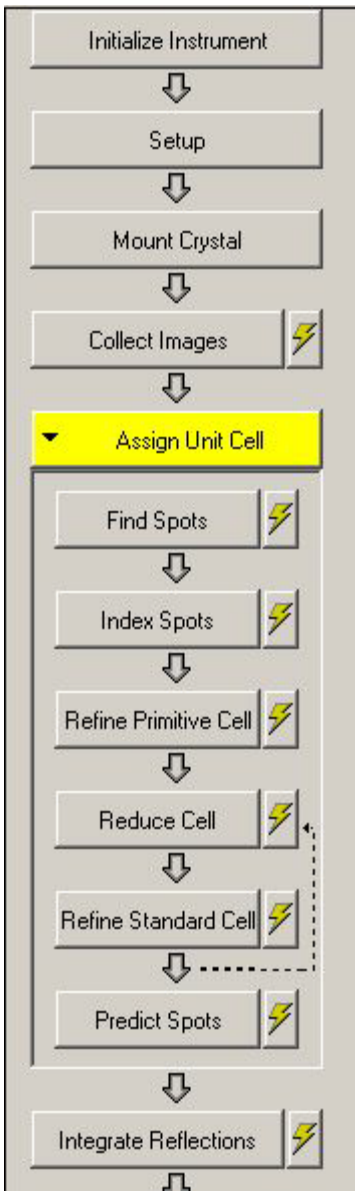
Scale and Average. The scale and average step scales and averages the data and may be used to carry out a multi-scan absorption correction. It creates a reflection file that can then be used in structure solution. See Chapter 8.

Final Refine Cell. In small molecule work it is customary to do a final cell refinement after the data has been scaled. This brings up the standard refinement dialog, but with the defaults set for a final refinement.

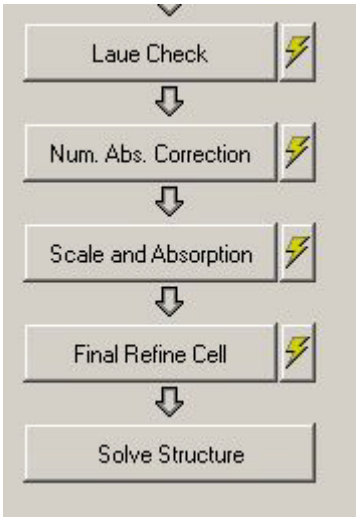
SolveStructure. Structure solution is performed through CrystalStructure and requires a CrystalStructure license. This step starts up CrystalStructure and brings up the AutoSolve Dialog. AutoSolve requires a molecular formula, so if you have not entered one, you are prompted to do so at this stage. See the CrystalStructure User Manual for information on running AutoSolve.

Collect and Process

This task replaces the **Initial Images** step with the **Collect Images** step and you then index and refine on some or all of the collected images.

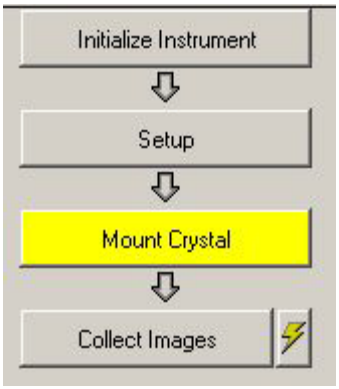


With the exception of the replacement of **Initial Images** with **Collect Images**, these steps are identical to the steps in **Screen Collect and Process**. The final steps in this process are also the same, so please refer to the previous section or directly to Chapter 8 for more details.



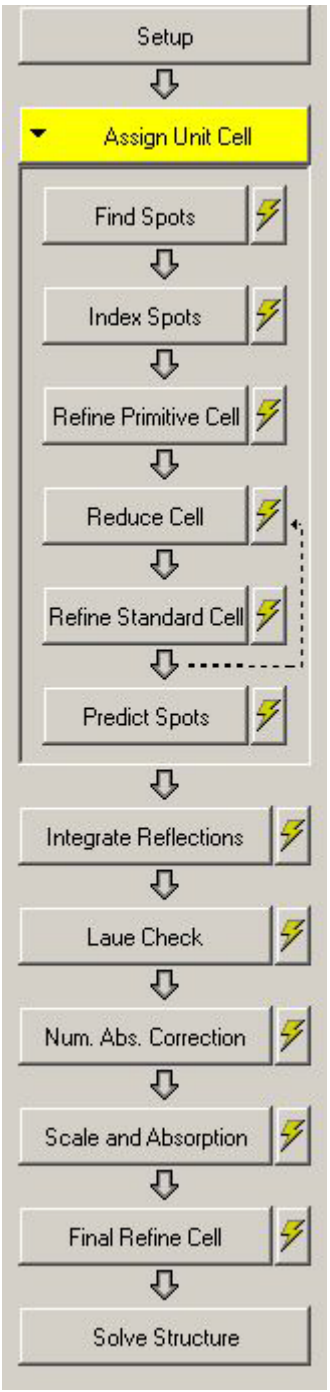
Collect

The collect task is the same regardless of the processing suite. This task does not offer the **Strategy** step since you have not yet indexed.



Process

Once you have collected your data, you can either go back into the sample and change it to a **Process** (sometimes referred to as process only) task, or you can create a new process only sample which refers to the images you collected in another sample (or on another instrument).



These processing steps are identical to the processing steps in **Screen Collect and Process**. You can refer to that section of this chapter or to Chapter 8 for more details.

d*TREK Quick Start Guide for CCD

Parameters

The values given for various parameters are simply suggestions, and may not be appropriate for all samples. Feel free to adjust certain parameters in order to evaluate their effect on the results. For parameters not listed here, start with the default values. Note that if a non-zero value is entered for either

min or max resolution, then the other value must also be non-zero. CrystalClear takes care of this automatically.

Setup

Main:
- check that crystal-to-detector and detector 2θ are correct.

Crystal1:
- Molecule type = Small

Detector:
- Check that Direct Beam is correct

X-ray Source:
- Check that the Element type (wavelength) is correct

Find Spots

Main:
- Use either the screening images or at least 20° of images from the first scan. A double-click in one of the **To Use** fields makes that field editable. A double-click in one of the **Available** fields selects that entire scan.
- I/Sigma = 5.0

Advanced:
- **Minimum pixel value** = 100.0
- **Find beam center** unchecked
- **Incl. saturated spots** checked
- **Det'n. strong peak info** unchecked

Index Spots

Main:
- **User chooses solution** checked

Advanced:
- **Deice** unchecked
- **Beam check** unchecked
- **Max. Residual** = 0.01. This should result in only one (primitive triclinic) solution.

Indexing notes: if the source is very strong and/or the crystal scatters extremely well, a few λ/2 spots may appear at low resolution. If these end up in the find-spots list, indexing may give the wrong cell. Try using only higher-resolution reflections for indexing if necessary.

Refine Primitive Cell

If the cell has been saved as triclinic, the angles are allowed to refine. By default, the refinement is done **On images**, using the same images on which indexing was done. This should work well, and the cell parameters should converge to values that reflect the true cell symmetry. The mosaicity often goes to some unreasonable value here, but it should not affect the results. If a **Warning** box appears, it is because

one or more values have shifted by more than some expected amount; this is usually OK - you can slide the warning message over and take a look at which values differ greatly from their starting values.

When refining **On images**, d*TREK uses the current orientation matrix to predict where spots should appear on each image. If a good reflection is found, its centroid is saved. You should press Run at least twice. Note the number of reflections **Accepted** (near the top right corner) after the first time. The number should increase the second time, as the "better" orientation matrix allows the program to find more good spots the second time. To selectively turn refinement parameters on or off, change Macro to **Single Step Refine**. For sealed tube sources all *source rotations* should be off. When convergence is reached, press the **Close** button.

Reduce Cell

This step searches for higher metric symmetry. If the cell is especially poor, you may need to raise the value of **Max. Residual** (try 1.0) and press the Reduce Cell button. *(Note: Be careful about choosing a cell of higher symmetry. If there is any uncertainty, it is better to choose a lower symmetry at this point and ensure that a complete dataset is collected. You will verify the Laue group after integration, at which time you will **know** the correct symmetry.)*

Refine Standard Cell

Run Refine once again; the cell dimensions are constrained by the saved crystal system. The final values in the Δ/σ row should be very low for each parameter. Note that you may also use the first few images from any scan for refinement.

Predict

Close the Refine dialogue and press Predict Spots. If the mosaicity shown is less than 1.0 or appreciably greater than 1.0, set it to 1. The first 10 images or so should be enough to check that the cell and detector corrections are appropriate. Step through the images with the green arrows. Use the zoom tool (or just right-click and drag on an area in the image) to get a closer look at the reflections. The contrast tool may also be useful for looking at higher resolution spots.

Create a Mask

Using any image that shows the beam stop shadow, reduce the High level of the contrast so that the shadow of the beam stop is obvious. Use the circle tool to draw a radius representing the beam stop (See Chapter 4). If you don't like it, use the **Undo last erasure** tool (two buttons to the right of the circle tool) to undo the erasure. Use the quadrangle eraser to remove the shadow of the beam stop arm; you get four clicks, each one defining a corner of a quadrilateral. Use the quadrangle eraser more than once to define oddly-shaped shadows. You can clear the erasures and start over by just clicking one of the green arrows to move to the next image. Click the Write Mask File button to save the mask file. If all images were collected at the same 2θ and distance, click Yes for **Use... for all scans?** If a set of images were collected at high 2θ swing (no beamstop shadow) these scans should have the mask file set to **FirstScanImage**.

Integrate Reflections

Main:

- All of the data collection images should be selected by default.
- Set min and max resolution if desired; otherwise leave at 0.0. Resolution arcs can be superimposed on the image by using **View → Resolution arcs**.
- Box width and height = 0 (determined automatically)
- Padding = 1

- Images per batch: Scaling = 1. For Refinement, choose a number of images that represents about 15 degrees of omega rotation (i.e. for 0.5deg images, use 30).

Advanced:
- Profile analysis ON, Num refl=20, Max images = 10
- Refinement macro = Most
- Find refls before... OFF
- Refine before integration: Macro = Most; Images = 3 refine batches (i.e. 3 batches of 15° to give about 45° of refinement before integration).
- Mosaicity model Off.

Note: During integration, the cell is refined and updated using the number of images determined by (Images per batch) times (no. refine batches). These should be adjusted, depending on the number of degrees per image, to represent 30 to 60° of data. These are used for local refinement of the cell during integration. These should be adjusted depending on the size of the cell. Integration of each scan starts with a prerefine using these parameters.

In order to monitor the log file as it is updated, you can turn off the image processing updates with the **View Image Processing Updates** button (or on the menu bar: **View → Image Processing Updates**).

Laue Check

- Set Maximum Residual to 1.
- If this fails miserably, select the Laue group that you "know" to be correct and continue.
- **IMPORTANT**: ***If*** the Laue check results in a cell transformation, the new reflection list is called 'dtcell.ref'. **Be SURE** to use this file for the next step.

Numerical Absorption Correction

If you have used the RAXShape utility to save a model of the shape of the crystal, you have a shape.dat file in the current sample directory. Enter the molecular formula for the compound; press the Tab key and the expected Z value is shown. Enter the correct (usually integer) Z value and press Run. The corrected file is called dtnumabs.ref. Be ***sure*** to use this file for the input to the next step if you want to use the absorption-corrected data.

Scale and Absorption

Main:
- Input reflection file: dtprofit.ref (unless as changed above).
- B Factor unchecked. Batch scale and absorption correction checked.
- Absorption correction method: Spherical 4,3 (may also try 'Fourier' method)
- Exclude sigma for refinement = 5 (adjust as necessary for strong or weak crystals)
- Scale anomalous I+ and I- separately unchecked
- Usually output is **Uncorrected, unaveraged reflections, with correction factors**, and the output file name is f2plus.dat
- Error model = (multiplier: Explicit, 3.0; addend: Explicit: 0.0)
    - Note: Try **Auto** error model to get an idea for a realistic value for the multiplier and/or addend values. Note how I/σI, Chi2 and Rmerge are affected by the variations in the error model.

Advanced:
- Rebatch unchecked.
- Restrain batches checked, set parameter to 0.005.

HINT: Click the arrow between the **Scale and Absorption** button and the **Final Refine Cell** button: it changes to a stop sign. Now the refine dialogue does not appear automatically after this step. This is useful since we often run the Scale and Absorption step multiple times. Just click the Final Refine Cell button when you are ready to run this step.

Final Refine Cell

- At this point, a final cell refinement is done using the integrated data from ONE of the scans.
- For **Reflection list**, data from the first scan (dtprofit1.ref) are used by default. Another scan may be chosen, i.e. dtprofit2.ref, etc.
- Press Run; this may take a while since a large number of data are being included

The final cell refinement uses (by default) the reflections from the entire first scan (dtprofit1.ref). If another scan is larger (contains more reflections) choose the appropriate dtprofit'n'.ref as the reflection file.

At the end of the Final Refine Cell CrystalClear writes the CrystalClear.cif file, which contains the cell and data collection parameters for transferring to the structure solution program.

When done, a lot of unnecessary files can be removed with the **File → Purge** files function.
Only two files are needed to use the CrystalStructure software: CrystalClear.cif and f2plus.dat. Starting in CC2.0, these files are automatically copied into a structure subdirectory.

Notes

- The size of the blue circles is arbitrary (does not signify integration area); the size can be changed to improve visibility with the Set Reflection Size button.
- If the Refine Cell dialogue is brought up during integration, the values are updated as refinement continues during integration.
- The **JDTPlot** utility is a very nice way to analyze the results of the integration and **Scale and Average** routines. In order to enable this utility, install the Java run-time environment available from http://java.sun.com/j2se/1.4.2/download.html **Use Tools → Preferences → ScaleAverage Plots** to select **JDTPlot**. Uncheck the **Prompt before** box to allow automatic display of the plots. The plots can be generated anytime after running Scale and Average from the menu **View → Scale/Average Plots**.

d*TREK Quick Start Guide for Rapid

This quick start guide runs through an example that was done with three scans, all scanning omega, 5° per image. For any parameters not listed, the default was used. Note that for wide sliced images FS_PROCESS to TwinSolve often product better results than d*TREK.

Scan 1: χ=0, φ=0, ω 20 to 200
Scan 2: χ=54, φ=0, ω 20 to 200
Scan 3: χ=54, φ=90, ω 20 to 200

Setup

Main:

- check that crystal-to-detector is shown as 127.4mm and detector $2\theta = 0$

Crystal1:
- Molecule type = Small

Detector:
- Check that Direct Beam is correct

X-ray Source:
- Check that the Element type (wavelength) is correct

Find Spots

Main:
- Use either the screening images or at least 30° of images from the first scan. A double-click in one of the **To Use** fields makes that field editable. A double-click in one of the **Available** fields selects that entire scan.
- I/Sigma = 5.0

Advanced:
- **Minimum pixel value** = 20.0
- **Find beam center** unchecked
- **Incl. saturated spots** checked
- **Det'n. strong peak info** unchecked

Index Spots

Main:
- **User chooses solution** checked

Advanced:
- **Deice** unchecked
- **Beam check** unchecked
- **Max. residual** = 0.01. This should result in only one (primitive triclinic) solution.

Indexing notes: if the source is very strong and/or the crystal scatters extremely well, a few λ/2 spots may appear at low resolution. If these end up in the find-spots list, indexing may give the wrong cell. Try using only higher-resolution reflections for indexing if necessary.

Refine Cell

If the cell has been saved as triclinic, the angles are allowed to refine. By default, the refinement is done **On images**, using the same images on which indexing was done. This should work well, and the cell parameters should converge to values that reflect the true cell symmetry. The mosaicity often goes to some unreasonable value here, but it should not affect the results. If a warning box appears, it is because one or more values have shifted by more than some expected amount; this is usually OK - you can slide the warning message over and take a look at which values differ greatly from their starting values. When refining **On images**, d*TREK uses the current orientation matrix to predict where spots should appear on each image. If a good reflection is found, its centroid is saved. You should press Run at least twice. Note the number of reflections "Accepted" (near the top right corner) after the first time. The number should increase the second time, as the "better" orientation matrix allows the program to find more good spots the second time. To selectively turn refinement parameters on or off, change Macro to

Single Step Refine. For sealed tube sources all *source rotations* should be off. When convergence is reached, press the **Close** button.

The **Reduce Cell** step searches for higher crystallographic symmetry. If the cell is especially poor, you may need to raise the value of **Max residual** (try 1.0) and press the **Reduce Cell** button. *(Note: Be careful about choosing a cell of higher symmetry. You will verify the Laue group after integration, at which time you will **know** the correct symmetry.)*

Run Refine once again; the cell dimensions are constrained by the saved crystal system. The final values in the Δ/σ row should be very low for each parameter. Note that you may also use the first few images from every scan for refinement.

Close the Refine dialogue and press Predict Spots. The first 5 images or so should be enough to check that the cell and detector corrections are appropriate. Step through the images with the green arrows. Use the zoom tool (or just right-click and drag on an area in the image) to get a closer look at the reflections. The contrast tool may also be useful for looking at higher resolution spots. If the mosaicity is much different from 1.0, you may get better results if you set it to 1.0.

Create a Mask Image

Using any image, reduce the High level of the contrast so that the shadow of the beam stop is obvious. Use the circle tool to draw a radius representing the beam stop. If you don't like it, use the **Undo last erasure** tool (two buttons to the right of the circle tool) to undo the erasure. Use the quadrangle eraser to remove the shadow of the beam stop arm; you get four clicks, each one defining a corner of a quadrilateral. Click the Write Mask File button to save the mask file. Click Yes for **Use... for all scans?**

Integrate Reflections

Main:

- All of the data collection images should be selected by default.
- Set min and max resolution if desired; otherwise leave at 0.0 (Note: if a non-zero value is entered for either min or max, then the other value must also be non-zero.) Resolution arcs can be superimposed on the image by using **View -> Resolution** arcs.
- Box width and height = 0 (determined automatically)
- Images per batch: Scaling = 1, For Refinement, choose a number of images that represents about 15° of omega rotation (i.e. for 5° images, use 3).
- Padding = 0

Advanced:

- Profile analysis ON, Num refl=20, Max images = 7
- Refine before integration: Macro = Most; Images = 3 refine batches (i.e. 3 batches of 15° to give about 45° of refinement before integration).
- Mosaicity model: Mult=1, Add=1. This parameter setting depends somewhat on the image width. If image width is more than 5°, then use Add=2. For images of 1° or less, turn off mosaicity model.

Note: During integration, the cell is refined and updated using the number of images determined by (Images per batch) times (no. refine batches). In the above paragraph, this is 3x3, so 9 images, representing 45° of data, are used for local refinement of the cell before integration. These should be adjusted depending on the size of the cell. Integration starts with a prerefine using these parameters. In order to monitor the log file as it is updated, you can turn off the image processing updates with the **View Image Processing Updates** button (or on the menu bar: **View → Image Processing Updates**).

When integration is finished, the actual integration ellipses are superimposed on the images of the final scan. These can be useful for evaluation of the quality of the integration.

Laue Check

- Maximum Residual = 1.
- If this fails miserably, select the Laue group that you "know" to be correct and continue.
- NOTE: If the Laue check results in a cell transformation, the new reflection list is called 'dtcell.ref'. Be ***sure*** to use this file for the next step.

Numerical Absorption

If you have used the RAXShape utility to save a model of the shape of the crystal, you have a shape.dat file. Enter the formula for the compound; press the Tab key and the expected Z value is shown. Enter the correct (usually integer) Z value and press Run. The corrected file is called dtnumabs.ref. Be ***sure*** to use this file for the input to the next step if you want to use the absorption-corrected data.

Scale and Absorption

Main:

- Input reflection file: dtprofit.ref (unless as changed above).
- Batch Scale checked. B Factor unchecked. Absorption correction Checked.
- Absorption correction method: Spherical 4,3
- Exclude sigma for refinement = 5
- Error model = Auto, also try explicit values (start with multiplier = 3 and addend = 0)
- Scale anomalous I+ and I- separately unchecked
- Output: Uncorrected, unaveraged reflections with correction factors
- Output name: f2plus.dat (Alternatively, output SHELX data directly)

Advanced:

- Rebatch unchecked
- Restrain batches checked, use a value of 0.005.

Also may want to try without batch scaling. Compare the results and decide which is better. *(If you did a numerical correction, change the selection to dtnumabs.ref as the input file.)* See the notes below regarding the plots that are generated.

HINT: Click the arrow between the 'Scale and Absorption' button and the 'Final Refine Cell' button: it changes to a stop sign. Now the refine dialogue does not appear automatically after this step. This is useful since we often run the Scale and Absorption step multiple times.

Final Cell Refinement

- At this point, a final cell refinement is done using the integrated data from *one* of the scans.
- For **Reflection list**, data from the first scan (dtprofit1.ref) are used by default. Another scan may be chosen, i.e. dtprofit2.ref, etc.
- Press Run; this may take a while since a large number of data are being included

At the end of the Final Cell Refinement a CIF file is automatically created.

A lot of unnecessary files can be removed with the **File → Purge** files function.
Only two files are needed to use the CrystalStructure software: texray.inf and f2plus.dat. These files are automatically put into the Structure directory.

NOTES:

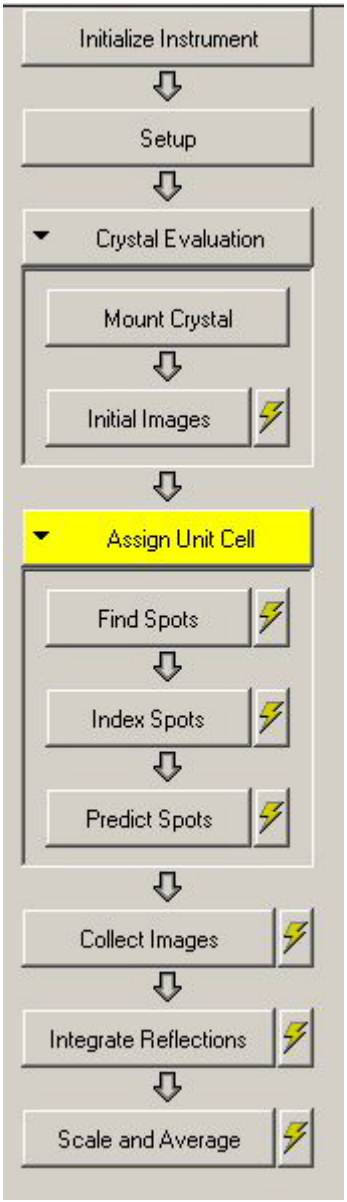
- o The size of the blue circles is arbitrary (does not signify integration area); the size can be changed to improve visibility with the Set Reflection Size button.
- o If the Refine Cell dialogue is brought up during integration, the values are updated as refinement continues during integration.
- o The JDTPlot utility is a very nice way to analyze the results of the integration and 'Scale and Average' routines. In order to enable this utility, install the Java run-time environment available from http://java.sun.com/j2se/1.4.2/download.html Choose the Java Runtime Environment (JRE) component - "allows end-users to run Java applications". Download and install this package. Back in CrystalClear, use Tools → Preferences → ScaleAverage Plots to select JDTPlot. Uncheck the "Prompt before..." box to allow automatic display of the plots.

Expert Mode using FS_PROCESS

The FS_PROCESS (for Fine Slice PROCESS) is suitable both for narrow and wider images. There is no separate Refine Cell step. There is no strategy available and the Laue check and other cell operations are options within the **Scale and Average** step.

Screen Collect and Process

The collection steps are the same regardless of the processing suite, but the processing steps are different.



Intialize, **Setup**, **Mount Crystal**, **Initial Images,** and **Collect Images** are all the same as for d*TREK and are described in Chapter 7. Note that there is no **Strategy** step available in FS_PROCESS so the collection schedule must be selected and edited manually.

Find Spots. FS_PROCESS has a separate **Find Spots** step which is described in Chapter 9. The dialog and parameters are different than for d*TREK.

Index Spots. For FS_PROCESS Index Spots also includes refinement, so there is no separate refinement step. The details are in Chapter 9.

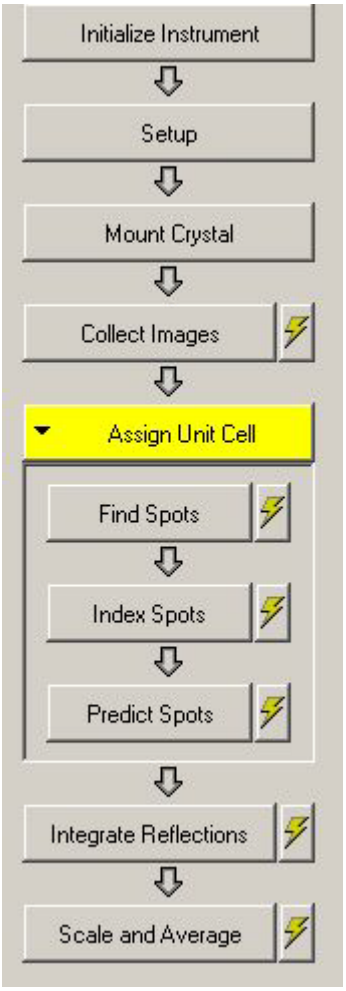
Predict Spots. FS_PROCESS also has its own predict step. See Chapter 9 for the FS_PROCESS predict details.

Integrate Reflections. FS_PROCESS integration cannot be run until the **Collect Images** step has completed. See Chapter 9 for details on the FS_PROCESS integration dialog.

Scale and Average. The **Scale and Average** step is also different in FS_PROCESS. It includes Laue check and other cell operations that are separate steps in d*TREK. See Chapter 9.

Collect and Process

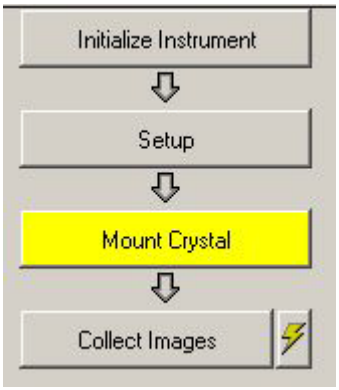
In this task the **Initial Images** step is replaced with the **Collect Images** step and since there are no screen images, you index and refine on some or all of the collected images.



These steps are described in the previous section.

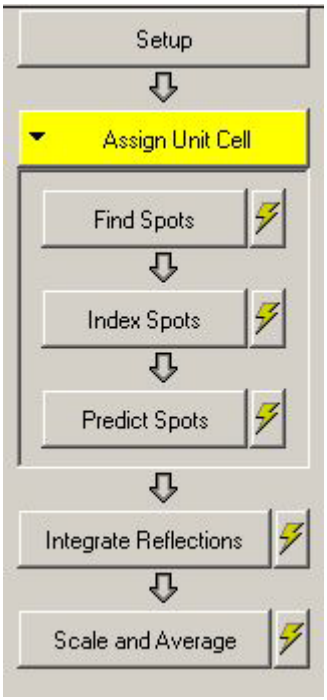
Collect

The collect task is the same regardless of the processing suite. This task does not offer the **Strategy** step since you have not yet indexed.



Process

Once you have collected your data, you can either go back into the sample and change it to a **Process** (sometimes referred to as process only) task, or you can create a new process only sample which refers to the images you collected in another sample (or on another instrument).



Expert Mode using TwinSolve

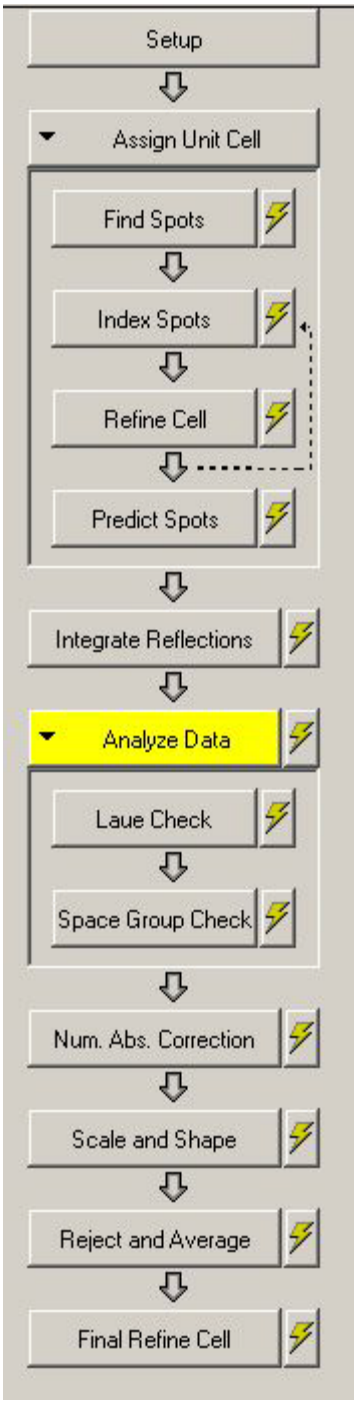
TwinSolve is available under a separate license for processing twinned crystals. Most installations of CrystalClear for customers doing small molecule work include a TwinSolve license. If you don't see a Swedish flag in the toolbar between the American and Japanese flags, you should contact your Rigaku support person to see about getting a TwinSolve license or getting your existing license activated. There must be a TwinsolveLicense.lic with the proper key in your installation directory.

TwinSolve is most often run as a process only sample once you suspect that your crystal is twinned. The other tasks are available in CrystalClear for completeness, but since they are seldom used, the focus is on the **Process** task.

If you do want to collect in TwinSolve mode, you can refer to the collection steps in the d*TREK mode for additional description of these tasks.

Process

Once you suspect your crystal is twinned, you may want to open a new process only sample using the TwinSolve processing suite. This automatically puts you in the **Process** task where you see the following flow bar. The details for each TwinSolve step and general information on TwinSolve are presented in Chapter 10.



Setup. TwinSolve uses the standard **Setup** step described in Chapter 7.

Find Spots. In TwinSolve you should usually select a large number of images (often the whole data set) for the find.

Index Spots. This step search indexes the major twin component and then removes those reflection from the found spots. You should repeat the **Index** and **Refine** steps for every twin component of your crystal.

Refine Cell. TwinSolve has a separate refine step that should be repeated for every indexed twin component.

Predict. Once you have indexed and refined the twin components, you use the Predict step to see if the predicted reflections match up with the spots.

Integrate Reflections. TwinSolve integrates each twin component in a single step.

Laue Check. In TwinSolve this is a separate step

Space Group Check. The TwinSolve space group check along with these other steps is document in Chapter 10.

Num. Abs. Correction. TwinSolve does numerical absorption correction in this step.

Scale and Shape. TwinSolve adds shape analysis to the scaling procedure.

Reject and Average. TwinSolve separates rejection of bad reflections and averaging into a separate step.

Final Refine Cell. At the end of the process, TwinSolve does a final cell refinement.

TwinSolve Quick Start Guide

This is a short overview with useful information to allow you to get started. However, to get the full benefit of TwinSolve, you really should take the time to read the details in Chapter 10 along with the TwinSolve interactive manual.

1. Start with a *complete* set of images before starting TwinSolve (TS). It is best if multiple scans have been collected, each involving rotation about a different direction through the crystal (for example, one scan with χ=54°, φ=0° and a second scan with χ=54°, φ=90°).
2. For the **Find Spots** step, use the *entire* set of data collection images (except screen images). It may be useful to examine a few images very closely before starting here in order to set the **Peak threshold minimum counts**. Use a **Sigma** (this is I/sigma(I)) of 5 or 6 here. Zoom in on a few spots and look at the max. counts/pixel to determine whether the “minimum” value in the **Peak threshold** box is appropriate. After the **Find Spots** step is finished, the first image should be displayed with the found spots circled (SCXmini: see Note 1), if processing updates are enabled (**View → Image Processing Updates** checked, or associated button on the toolbar enabled). Step through the first few images to see whether **Find Spots** has detected a reasonable number of reflections. If powder rings exist, the program may detect hundreds or thousands of reflections within the rings; in this case re-run with the Sigma values increased and/or a higher **Peak threshold minimum counts**, or set the 2θ limits (**Sample → Set Resolution**… or **Set Resolution Limits** icon on toolbar) to use only those reflections above the 2θ of the powder rings.
3. Index - normally just use all defaults here. Use **Quick search** with 90%. If you know that there are spots which are very close together, you may need to decrease the 'hkl deviation' value to something less than the default 0.2 (like 0.1). [**NOTE** - even if the first indexing results look wrong, go ahead and refine the cell to improve the detector corrections. This wrong twin component can be deleted later.] Input: oriref.xor.
4. Refine - Normally should **not** check the **Constrain unit cell according to symmetry** box until the crystal system is definite. After refinement the program asks **Transform to the conventional cell?** If you say yes, the program looks for a higher symmetry cell and offers to change it for you.

In the current version you can only accept or reject the suggestion in this version of the program. This is a problem if you have something that is close to higher symmetry but not quite. There is an interactive mode that includes much more flexibility in what you can do with the cell, etc. Eventually you may want to learn some of the extended commands. Input: oriref.xor

5. Once the refinement is done, look at the last line printed in the refinement log printed to the screen. It shows the number of reflections assigned to the current twin component and the total number of reflections available. If the number of reflections assigned to the current twin component is very much less than the total, then you may have a twin and need to do another round of indexing to find another component. Just click **Index Cell** again. Only the reflections which are not yet assigned to a twin component are used. Refine each component until you are satisfied with convergence of the least-squares results.
6. If you have two or three (or more) twin components that all have the same cell dimensions, you should finally refine them all together. In the **Refine** menu, make the **Active component** the one that has the most reflections (usually component 1), then check the box **Restrain using other components** box. Click the **Reset all twins** button if the list of twins is not correct.
7. If you have more than one component, and you haven't done so already, use the **Get twin relations** button to get the twin law(s), and use the **Predict Spots** function to get a visual representation of how the twin components overlap on your images.
8. For integration, the default parameter settings are usually best. Check the **Use Twinsolve proposed values** for **Box Size** and **FWHM** unless you have reasons to set them manually. Output: datmi?.raw, ?=1,2,… and datmg.raw.
9. A numerical absorption correction (shape-based) is NOT recommended for a twin. Unless you are handling a single crystal, skip to the next step. For single crystals, input is datmg.raw. If you have measured the shape, use Gaussian correction. For a spherical crystal, just specify the radius. Output: datab.raw
10. **Scale and Shape**: The default values are recommended. You may need to increase the number of cycles if the calculation does not converge. For problem data, try different **Shape factor model** settings. The recommended ones are the first, third, and fifth ones in the list. The one that you use last is the one kept. Input: datmi%.raw (% = 1,2,3..) unless Numerical Abs.Corr. has been run, then use datab.raw. Output: dataa.raw.
11. **Reject and Average**: Input: dataa.raw if **Scale and Shape** was run, otherwise datmg.raw.
    a. For single crystals, **Twin output** doesn't matter. Set averaging to **None**, for rejecting outliers start with **Reject limit** = 3.0.
    b. For twins, this step may be run more than once. Run once with **Twin component** set to 1 and **No twin overlaps** checked. This creates a file named hklf_1.hkl, containing only reflections from component 1 that are not overlapped with other components. To create a twin reflection file (in SHELX 'HKLF 5' format), use Twin components = 0, **Calculate for all twin combinations**, and *turn on Averaging* (unit weights OK). Reflections in a HKLF 5 format file *must be averaged!*
12. Do a final cell refinement with constraints. All of the operations are summarized in the CIF (tsarchive.cif). The reflection data are in hklf4.hkl or hklf4_1.hkl and/or hklf5.hkl.

CrystalStructure

To use TwinSolve data in the Rigaku CrystalStructure program, you need the data file and the CIF created by TwinSolve. The CIF is named **tsarchive.cif**, and normally can be used as-is. Alternately you can use the texray.inf file that is created in the Structure directory.

The data file that you use depends on how you want to handle the data. If you are using only a single component or if you created a non-overlapped file of one component, you should have a file named **hklf4.hkl** or **hklf4_1.hkl**. This file is edited to add a format line and then renamed to **f2.dat** and stored in the Structure directory.

If you have created a twin data file, it is named **hklf5.hkl** and contains data from multiple twin components. This file can be copied into the Structure directory, and will be copied to **shelxl.hkl** when refinement with SHELX is begun.

Once the project has been opened, click **Tools → Refine mode → Twin**. You are asked to enter the twin law. *If you are refining with SHELXL, this matrix can be left as the identity matrix, otherwise enter the twin law for refinement with Crystals*

Tips and Tricks

In C:\Rigaku\CrystalClear20\Twinsolve folder, find a file named *tshelp.xhe*. This is a text file that contains all of the author's help information for every command, and is especially useful for using TwinSolve in interactive mode (**Processing→Twinsolve interactive***)*.

Every operation in TwinSolve is written to one long log file called *logger.xlg*. This is a text file that you can go through to verify everything that happened during the processing.

Notes:

1. For SCXmini, the circles displayed after **Find Spots** do **not** correspond to the reflections on the image. Continue with indexing and refinement, then "Predict" correctly indicates the reflection positions.

2. Be careful when using index more than once. If you have already indexed a cell, and use **Index Cell** again, the program only uses the reflections that were not assigned to the first cell, and a second cell is added to the twin list. There are times when you may want to index several times, then go back and delete the unwanted cells: in the **Processing State Display**, choose twin number (**Crystal component**) from the drop-down list, press **Delete**. When a component is deleted, reflections assigned to that twin component are released and then assigned to another component (if they fit) during cell refinement.

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6. Macromolecule Tasks

There are two available processing suites each with four different tasks for protein and other macromolecular crystallography. You normally select the processing suite when you create a sample, but you can change it though the icons in the main toolbar. For each of these combinations there is a flow bar provided which guides you through the steps for that task. This chapter concentrates on the work flow. The details on the steps themselves are provided in chapters 7-10.

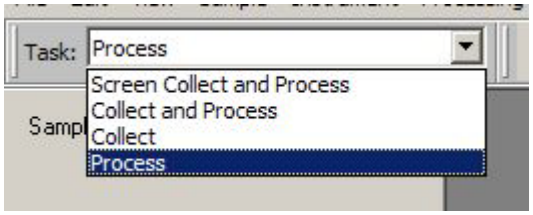
d*TREK Tasks

Selecting d*TREK

 To select d*TREK as your processing suite click the American flag icon in the top toolbar. This is the default for CrystalClear. You also have the chance to select the processing suite for each sample as it is created (see Chapter 3).

Selecting the Task

Whenever you create a sample, you must select the initial task for that sample. However, it is possible to change that task either when you re-open the sample or using the task drop down list.

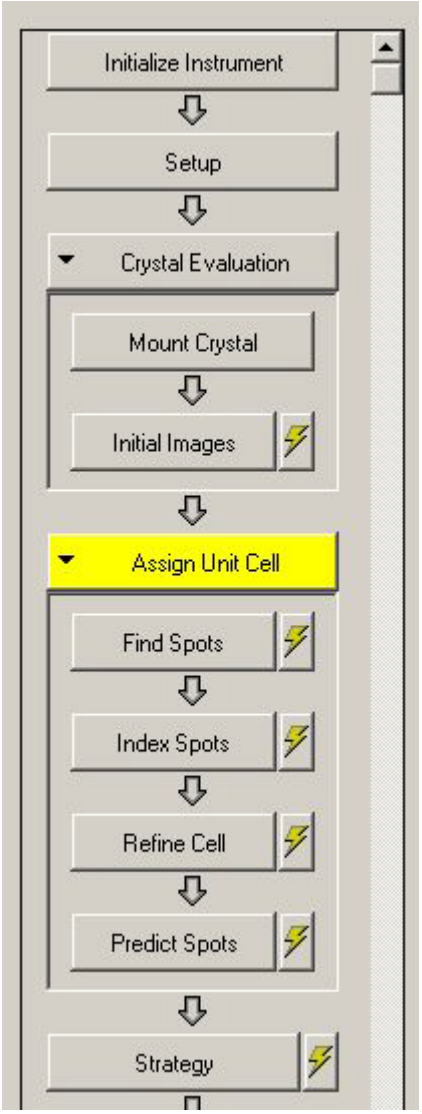


Simply select the task you want and the flow bar is updated with the new task flow.

Screen Collect and Process

Select this task when you plan to screen samples and then collect and process a full data set for the best one. Only one instance of CrystalClear that involves any image collection can be open at a time. However, you can run additional instances of CrystalClear with Process only samples.

The first part of the flow bar is shown below.



Initialize Instrument. Before you can collect data, the goniometers and detector must be initialized. This can also be done when the sample is opened or through the Manual Instrument Control. If the instrument is already initialized you can skip this step. See chapter 7 for more details.

Setup. This step allows you to specify information about the sample. If this is a new instrument, you should check the X-Ray Source tab to make sure that the X-Ray source information right. If it is not, you should set the values appropriately and then click **Save to Config**. See chapter 7 for more details. Once you system has been configured properly, this step is often skipped for macro work.

Mount Crystal. This is really a pseudo step to remind you to mount the crystal and lock down the rotation axis. Once you have done this click OK and then click Initial Images, which unlike some other steps does not open automatically.

Initial Images. This step allows you to collect a few screen images for evaluation and possible indexing.. See Chapter 7 for a detailed description.

Find Spots. This step identifies spots to be used for indexing. The available parameters are described in Chapter 8.

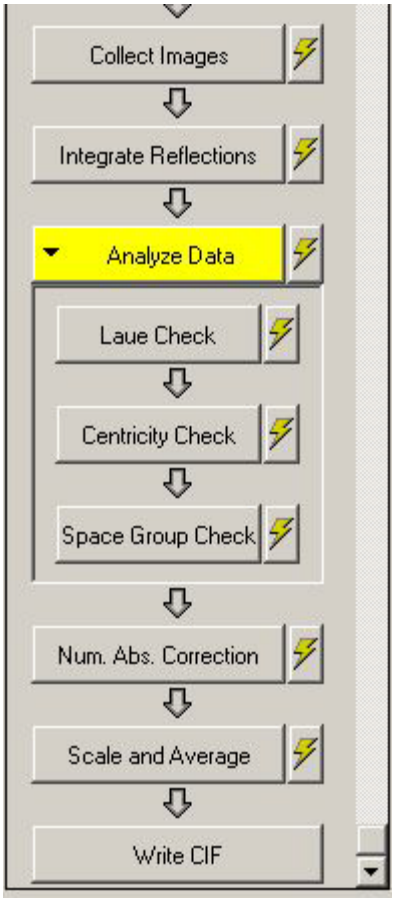
Index Spots. Indexing determines the cell parameters and symmetry. The parameters for indexing are described in Chapter 8.

Refine Cell. For d*TREK indexing and cell refinement are two separate steps. CrystalClear provides a comprehensive interface into refinement. You can refine either on the reflection list from Find Spots or on the screen images themselves.

Predict Spots. The purpose of this step is to allow you to compare the position of the predicted and experimental spots and thus check the validity of the refined unit cell parameters. You can also use this step to experiment with various mosaicity values.

Strategy. Strategy suggests a collection schedule based on the cell parameters and crystal symmetry determined in previous steps. The user sets the completeness and redundancy requirements. If you prefer to specify the collection schedule manually, you can skip this step. Strategy is only available for the d*TREK suite and is documented in Chapter 8.

The steps from **Collect Images** on are shown in the next screen shot.



Collect Images. The collection schedule for this step can be entered by the user or can come from **Strategy**. You can start the **Integrate Reflections** step before Collect has completed so that the images can be integrated as they are being collected. See Chapter 7 for details

Integrate Reflections. This can either be done while the images are being collected or after collection has finished. Chapter 8 provides the details for the parameters that control integration.

Laue Check. These next three steps are optional steps to check the cell parameters after integration of the entire data set. This step checks the Laue group. See Chapter 8.

Centricity Check. See Chapter 8.

Space Group Check. See Chapter 8.

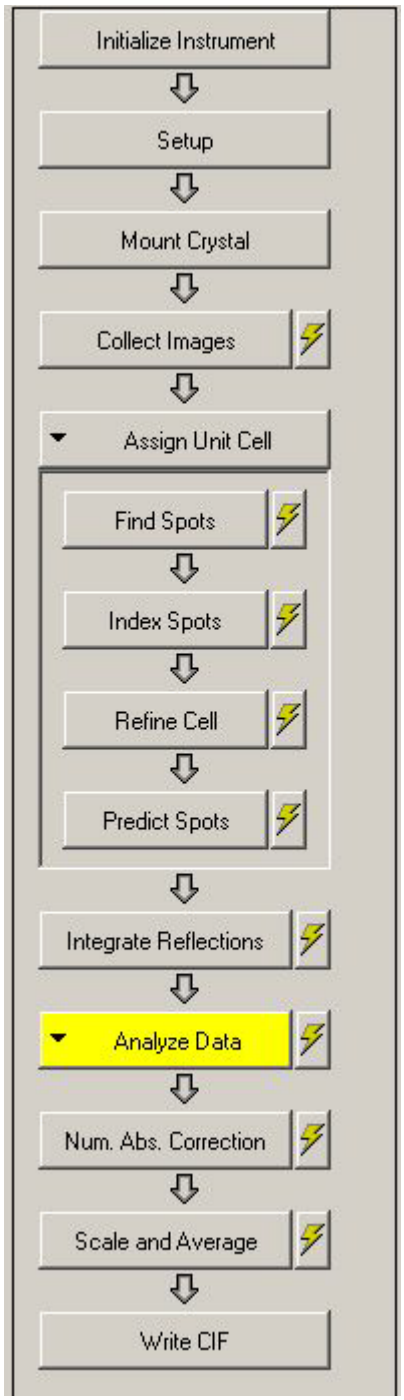
Num Abs. Correction. This step does numerical absorption correction to prepare the data for **Scale and Average**. This is option since other mathematical absorption models are available in the **Scale and Average** step. See Chapter 8.

Scale and Average. The scale and average step creates a file of merged, averaged and scaled reflections that can then be used in structure solution. See Chapter 8.

Write CIF. The sample and cell information can be written to a CIF file if desired. See Chapter 8.

Collect and Process

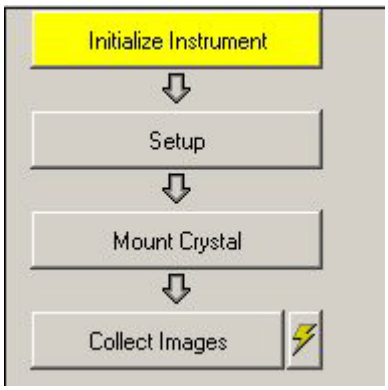
The Collect and Process task implements the so called “Wild West” method. Shoot first and ask questions later. You can use this task when you want to collect a full data set without previously screening the crystal. The flow bar is show below.



All these steps are described above in Screen Collect and Process. The difference is that the Initial Images step is omitted and the Assign Unit Cell steps (Find, Index, etc) are moved after Collect and before integrate. Note that it is not possible to integrate during data collection in this task because you have not yet indexed the cell and you need to be able to predict to integrate.

Collect

The simplest task is just a simple data collection. This assumes processing is done later or even with a 3rd party program.

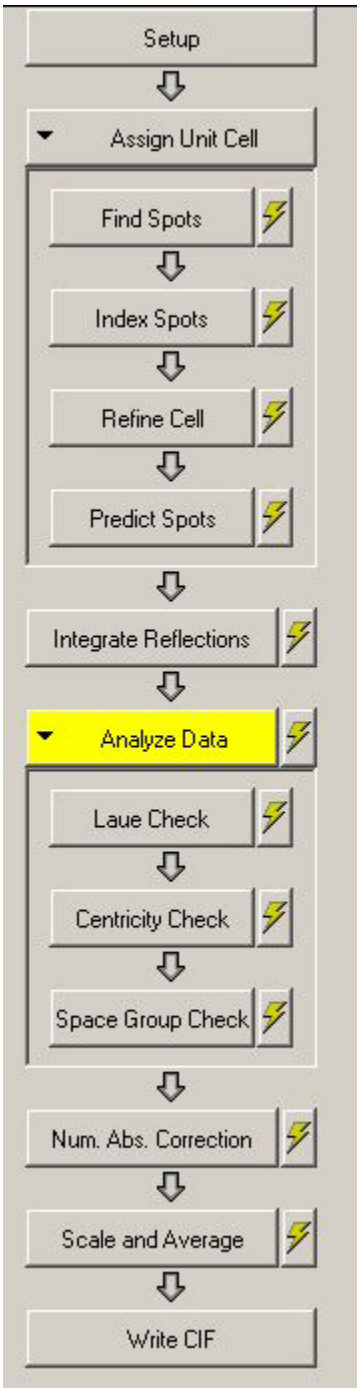


Process

The **Process** task (sometimes referred to as **Process Only**) is commonly used for processing a data set that has been collected previously. It can run on a different computer from the control PC on which the data was collected. The only requirement is that the PC which is running the process only task must be able to access the image directory.

It is also sometimes useful to create a Process only task to if you want to change the processing suite or process the data with different parameters. This can keep the cell parameters, log files, etc separate and gives you a “clean start”.

The flow bar for **Process** is show below.



These steps are identical to the **Screen Collect and Process** steps except that the **Initialize**, **Initial Images**, and **Collect Images** steps are removed.

Fine Slice Process

Selecting FS_Process

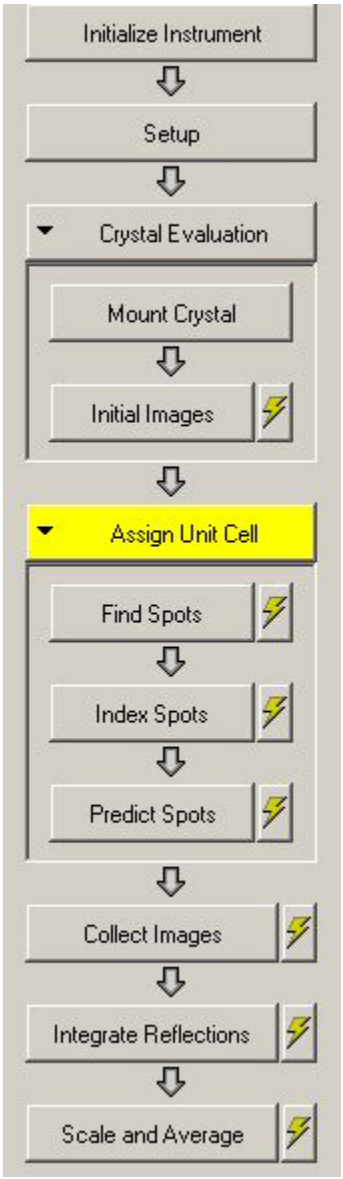
 To select Fine Slice Process (FS_PROCESS) as your processing suite click the Japanese flag icon in the top toolbar. You also have the chance to select the processing suite for each sample as it is created (see Chapter 3).

Selecting the Task

FS_PROCESS has the same set of tasks as d*TREK.

Screen Collect and Process

The collect steps are the same in FS_PROCESS as in d*TREK, but the processing steps are different. The flow bar is:



Intialize, **Setup**, **Mount Crystal**, **Initial Images,** and **Collect Images** are all the same as for d*TREK and are described in Chapter 7. Note that there is no **Strategy** step available in FS_PROCESS so the collection must be set up manually.

Find Spots. FS_PROCESS has a separate **Find Spots** step which is described in Chapter 9. The dialog and parameters are different than for d*TREK.

Index Spots. For FS_PROCESS there is no separate refinement step. The details are in Chapter 9.

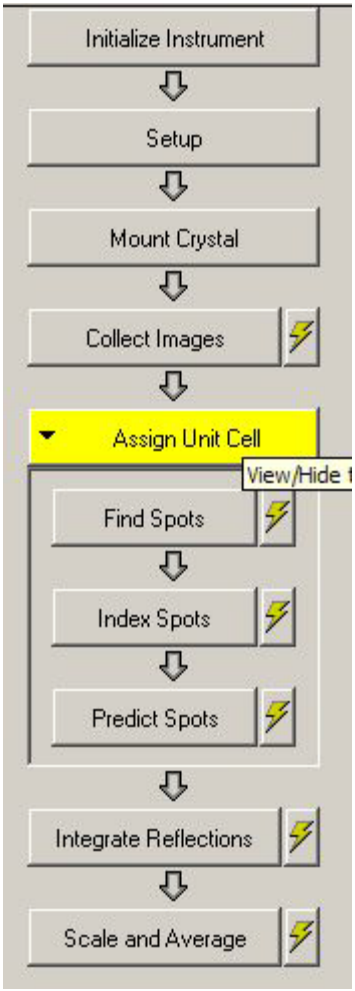
Predict Spots. FS_PROCESS also has its own predict step. See Chapter 9 for the FS_PROCESS predict details.

Integrate Reflections. FS_PROCESS integration cannot be run until the **Collect Images** step has completed. See Chapter 8 for details on the FS_PROCESS integration dialog.

Scale and Average. The **Scale and Average** step is also different in FS_PROCESS. See Chapter 9.

Collect and Process

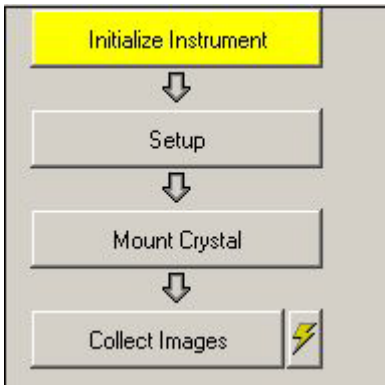
The **Collect and Process** task in FS_PROCESS simply replaces the **Initial Images** step with the **Collect Images** step. This is useful if you want to collect a data set without first collecting screen images.



The collection steps (**Initialize Instrument, Setup, Mount Crystal**, and **Collect Images** are described in Chapter 7. The FS_PROCESS processing steps are described in Chapter 9.

Collect

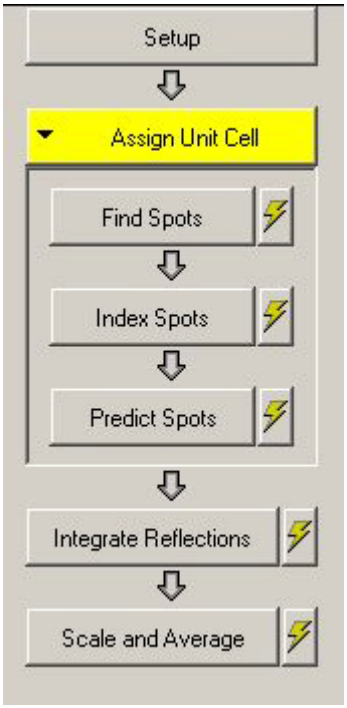
The collect task is identical to d*TREK..



Process

The **Process** task (sometimes referred to as **Process Only**) for FS_PROCESS has the same role as it does for d*TREK. FS_PROCESS does not allow you to integrate data until the data collection step has completed. You cannot create the sample until the data collection has completed.

The flow bar for **Process** is show below.



These processing steps are identical to the processing steps in FS_PROCESS **Screen Collect and Process** and are document in Chapter 9.

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7. Data Collection

This chapter describes the steps that are related directly to setting up a sample and collecting data. These steps are normally activated through one of the task flow bars, but can also be activated through the Instrument menu. The steps in this chapter apply to all processing suites. They have different defaults for macromolecular and small molecule modes, but have the same functionality.

Initialize

Initialization

Before you can perform any data collection steps, you must initialize the instrument. The instrument must be initialized at the following times:

1. Whenever you start or re-start CrystalClear. CrystalClear restarts the instrument server, so the instrument server must initialize so that it knows the exact status of the instrument.
2. If there is an error in the data collection or goniometers movements. When these errors occur they often leave the instrument in an unknown state, so it must be re-initialized.
3. If you do an **Abort**, either through the software abort button or any of the hardware aborts. Again this leaves the instrument in an unknown state so it is necessary to re-initialize.

In some cases CrystalClear realizes that it needs to re-initialize and do this automatically, but when you start or re-start CrystalClear you must do it explicitly.

There are several ways in which you can initialize in CrystalClear

1. When you open a sample with a task that involves Collect, CrystalClear asks you if you want to initialize.
2. You can open the **Manual Instrument Display** (see Chapter 4 Views) and click **Initialize**. For an R-AXIS and other image plate systems this can give you more control over the initialization process
3. You can click the first step in the **Screen Collect and Process**, **Collect and Process**, or **Collect** flow bars.

Initialize Instrument Step

The **Initialize Instrument** step in the flow bar issues an initialize command to the Instrument Server and brings up the **Instrument Sate Display** so that you can monitor the progress of the initialization.

Setup

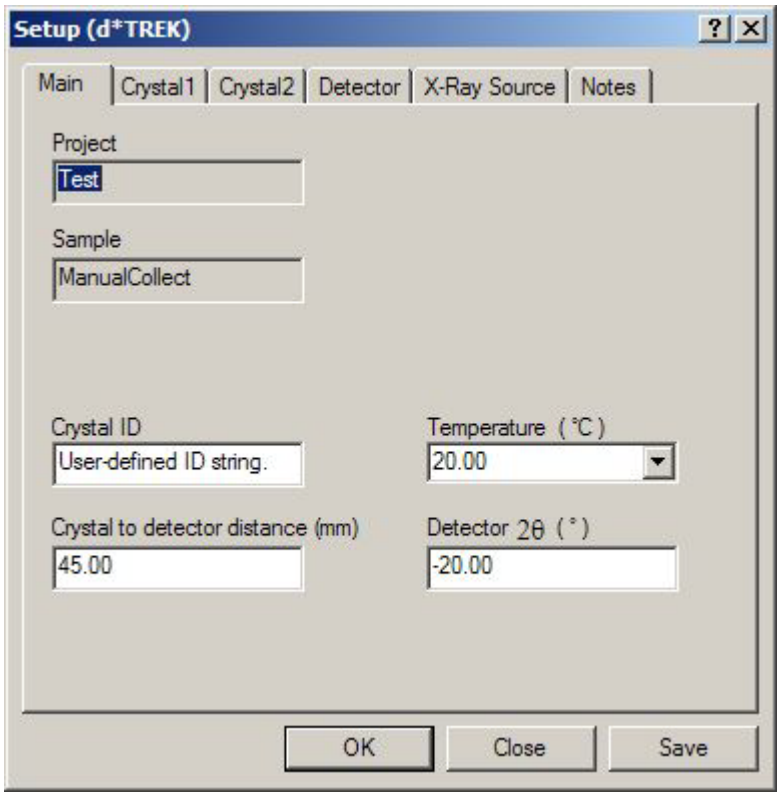
Overview

The **Setup** step is used for both tasks which collect data and process only tasks. Its primary purpose is to collect information that is useful in processing the sample. It also gives you a chance to enter information that is useful in solving the structure. Much of this information is for record keeping only and can be skipped if you do not need this information or have it available from another source.

Setup is also used to define X-Ray generator configuration settings. For samples you are collecting on your home lab instrument, updating the X-Ray configuration only needs to be done rarely. To do this enter the information for your site and then save this to the configuration file. If you are processing data from a beam line or elsewhere you may need to change the X-Ray information if it is not right in the image headers. But in this case you should not save the updates to the configuration since they do not apply to all data sets. You should pay particular attention to the section about the X-Ray Source tab.

Setup Dialog MainTab

When you click the Setup Step, the following dialog appears:

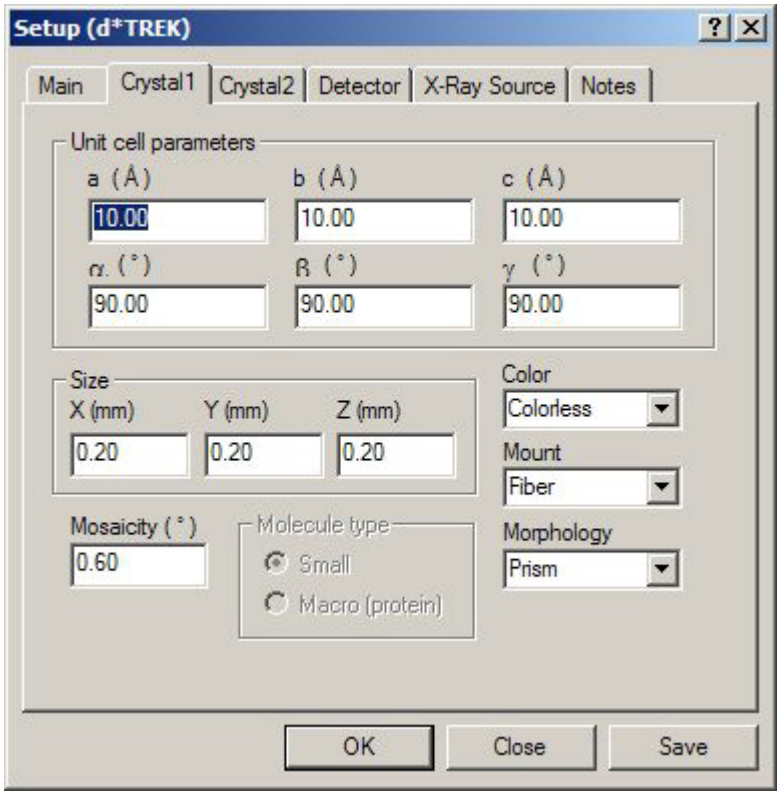


Parameter	Description
Project	Current Project (Read only – to make sure this is the sample you think it is)
Sample	Current Sample. (Read only)
Crystal ID	Crystal ID is a name or number you can give to your crystal for your personal reference. This value is not used in collection or processing.
Crystal-To- Detector Distance (mm)	The distance from the crystal to the detector when collecting images. If you are processing existing images, the value specified when the images were collected is displayed. NOTE: Crystal-To-Detector Distance (mm) field shows the hardware or unrefined values. NOTE: This information is *descriptive* and not *proscriptive*. If you are collecting new data for this scan, you should enter the Distance in the scan table. If "per scan" is displayed in the Crystal-To-Detector Distance (mm) field, different scans have different values. In this case, refer to the Scan State

Parameter	Description
	Display window to view these values for each scanned image.
Temperature (°C)	The Temperature of the crystal sample at the time of collection, in degrees Celsius. The options are: • 20.0 • 4.0 • -160.0 • custom value
Detector 2θ (°)	2θ angle of the detector goniometer. Detector 2θ (°) field shows the hardware or unrefined values. NOTE: If existing images are being processed, the value specified when the images were collected is displayed. NOTE: This information is *descriptive* and not *proscriptive*. If you are collecting new data for this scan, you should enter the 2θ in the scan table.

Setup Dialog Crystal1Tab

There are two tabs for entering crystal information. Much of this information is used for record keeping (e.g., a CIF file), but is not used in processing. The exceptions are the Unit Cell Parameters which are used if you choose Known Space Group and the Molecular Formula which can be used for numerical absorption correction and small molecule structure solution. The first Crystal tab is:

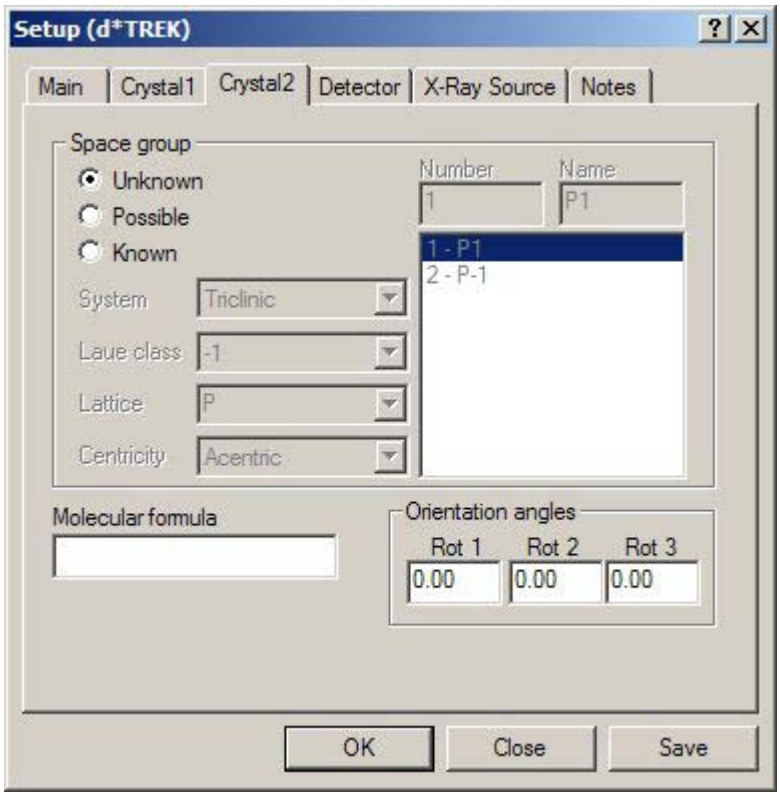


Parameter	Description

Parameter	Description
Unit Cell Parameters	The Unit Cell Parameters for the sample. a (Å) default = 10 b (Å) default = 10 c (Å) default = 10 α (°) default = 90 β (°) default = 90 γ (°) default = 90
Size	The Size of the crystal in millimeters. X (mm) default = 0.2 Y (mm) default = 0.2 Z (mm) default = 0.2
Mosaicity	Starting value to use for crystal Mosaicity. The default starting value is 0.6.
Molecule Type	Specify the Molecule Type: • small • macro (protein) NOTE: Setting this does **not** change the mode of CrystalClear itself.
Color	Color of the crystal. You may enter the color name or use a drop-down list which provides predefined colors. The predefined color choices are: • Blue • Colorless • Gray • Green • Orange • Red • White • Yellow
Mount	Type of Mount used for the crystal. You may enter the mount type in the field, or use a drop-down list which provides predefined mount types. The predefined mount choices are: • Fiber • Capillary • Loop
Morphology	Specify the Morphology of the crystal. You may enter the name in the field, or use a drop-down list which provides predefined morphology types. The predefined choices are: • Block • Chip • Chunk • Platelet • Prism

Setup Dialog Crystal2 Tab

The second Crystal tab is:

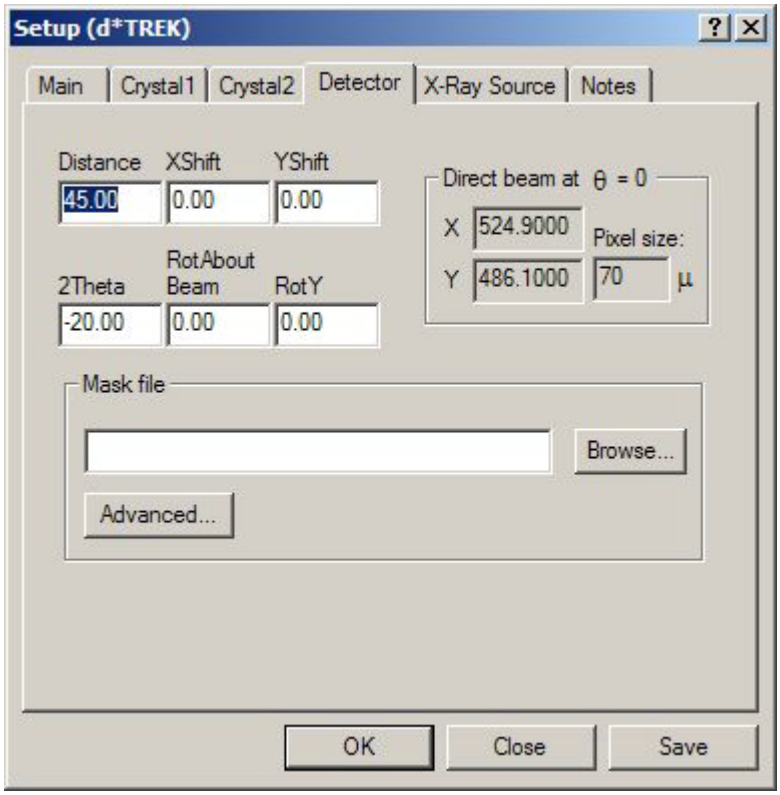


Parameter		Description
Space group		Specify the Space group of the crystal: • Unknown • Possible • Known To enter a Known Space group, type either the number or the name in the appropriate field. Alternately, if you know the Crystal System and need to be reminded of the possible Space groups, select the Crystal System, then scroll and choose the Space group from the list under Number and Name.
	System	If you selected Known Space group, this option becomes enabled. You can select from the drop down list of predefined Crystal Systems. Known Crystal System options are: • Triclinic • Monoclinic • Orthorhombic • Tetragonal • Trigonal • Hexagonal • Cubic

Parameter		Description
	Laue class	Select the Laue class from the drop-down list. Options are: • -1 • 2/m • mmm • 4/m • 4/mmm • -3 • -3m1 • -3/m • 6/m • 6/mmm • m-3 • m-3m
	Centricity	Select the Centricity from the drop-down list. Options are: • Centric • Acentric
	Number **Name**	Scroll through the available space groups from the list under Number and Name, or alternately, type either the number or name in the appropriate Number or Name field. The other field updates automatically to match.
Molecular Formula		The Molecular Formula for the crystal. You may enter the formula as it appears in your compound. CrystalClear determines the empirical formula for you. This is, of course, for small molecule users.
Orientation angles		Rot1, Rot2, Rot3 values are reported here for a sample that has been indexed. For a sample yet to be collected they are calculated later on by CrystalClear so you should leave them as 0.0 when starting a new sample.

Setup Dialog Detector Tab

This tab describes information about the detector.

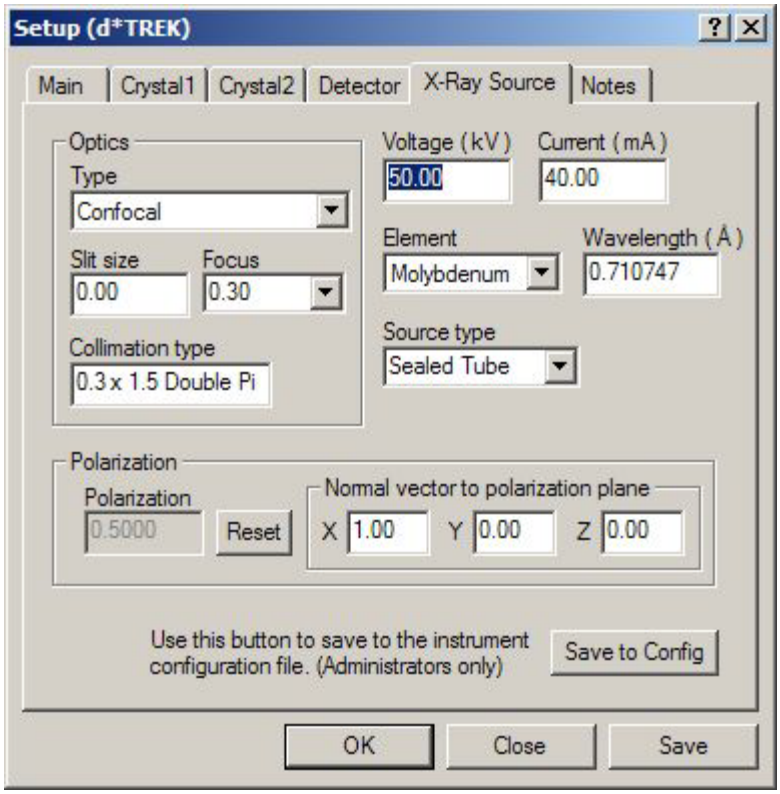


Parameter	Description
Distance	Crystal-to-Detector distance. NOTE: The Crystal-to-Detector Distance may be changed on the Main tab of the Setup dialog, as well as on the Detector tab. The 2θ (°) can also be set on the Main tab. The other Direct Beam settings on this tab are generally not changed.
X Shift	Detector shift along the X-axis (usually zero).
Y Shift	Detector shift along the Y-axis (usually zero).
Rotation About 2 Theta	Amount the detector is rotated about 2θ (swing angle).
Rotation About Beam	Amount the detector is rotated about the beam axis (usually zero).
Rotation About Rot Y	Amount the detector is rotated about the Y-axis (usually zero).
Direct Beam at θ = 0	The Direct Beam settings are based on the type of X-ray detector. Normally, the values are set as site defaults by your CrystalClear administrator. You may set the X, Y values and Pixel Size (μ).
Mask File	The Mask File group box displays information about the mask file. Using a Mask File may be useful when running Integrate Reflections to define the shadow of the beam stop or other shadowed regions. Reflections overlapping the masked region is excluded during data processing. It is not necessary to use a mask file during processing, however, it is advisable. If you are collecting a new data set, there is another opportunity to specify a mask file at the Collect step. Click the Browse... button to access the Open Image File dialog. Click the Advanced... Button to access the Non-Uniformity dialog. The

Parameter	Description
	recommendation is that you use the non-uniformity information that comes automatically in the image headers since this reflects what is actually collected, so you rarely need to use this button.

Setup Dialog X-Ray Source Tab

Much of the setup information is for book keeping or informational purposes only and does not affect data collection and processing. However, this tab contains information that does affect data processing (e.g., the Wavelength) and so it is very important that this is right. Some of the X-Ray Source information can also be used to update a system configuration file (if you are a user with administration privileges). In fact, if you are the system administrator, you should open a dummy Screen Collect and Process sample and then go into the Setup dialog, select the X-Ray Source tab and make sure this information is correct. If not, you should update it and then click **Save to Config**.



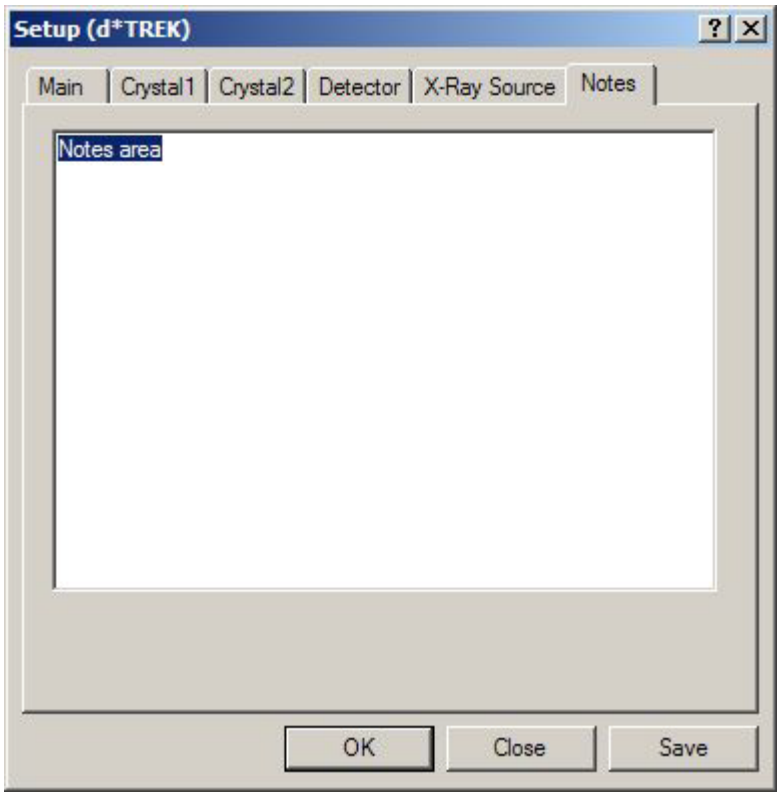
Parameter	Description
Optics	The following attributes pertain to your detector:

Parameter		Description
	Type	Select from the drop-down list. The options are: • Confocal • Graphite Monochromator • Mirrors • Multilayer • Synchroton • Other
	Slit Size	Specify the value.
	Focus	Select from a drop-down list: The options are: • 0.1 • 0.15 • 0.2 • 0.3 • 0.5
	Collimation Type	Specify the value.
Voltage (kV)		The Voltage (kV) of your X-ray source.
Current (mA)		The Current (mA) of your X-ray source.
Element		The Element type of your X-ray source from the predefined drop down list. If the Source Type is Synchrotron then this field is disabled. The predefined choices are: • Chromium • Copper • Gold • Iron • Molybdenum • Silver • Tungsten
Wavelength (Å)		The Wavelength of your X-ray source. The wavelength is based on the Element type. When you select an Element, a valid wavelength is automatically selected for you. If the Source Type is Synchrotron, then you need to enter this value explicitly.
Source Type		The Source Type of the X-ray source from the predefined drop down list. The predefined choices are: • Rotating Anode • Sealed Tube • Synchrotron
Polarization		Polarization (per scan) displayed. Use the Reset button if necessary. If there is no polarization set this value to 0.5. Synchrotron beams may be highly polarized, so it is important to specify a value and the normal vector.
	Normal vector...	Normal vector to polarization plane: • x • y

Parameter	Description
	• z
Save to Config	The values in the X-Ray Source Tab are saved in a configuration file rather than the default database. Use the **Save to Config** button to save the X-Ray source information to the configuration file. This makes these settings the default for all new samples. NOTE: This operation is available for CrystalClear Administrators only.

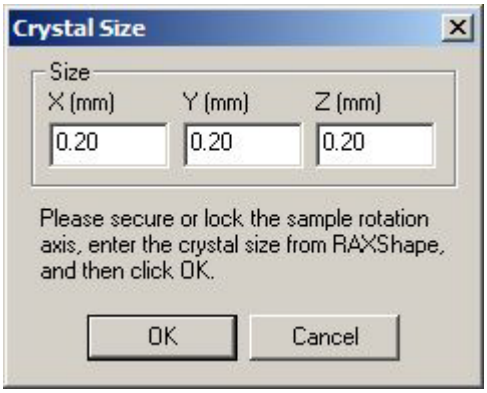
Setup Dialog Notes Tab

The final tab allows you to enter notes for future reference.



Mount Crystal Step

The **Mount Crystal** step is just a reminder that you need to mount the crystal and lock down the rotation axis at this point in the procedure. It also gives you another chance to enter the size of the crystal. This is handy if you have run RAXShape to determine the size of the crystal (small molecule modes) since it means you do not need to go back into the setup dialog.

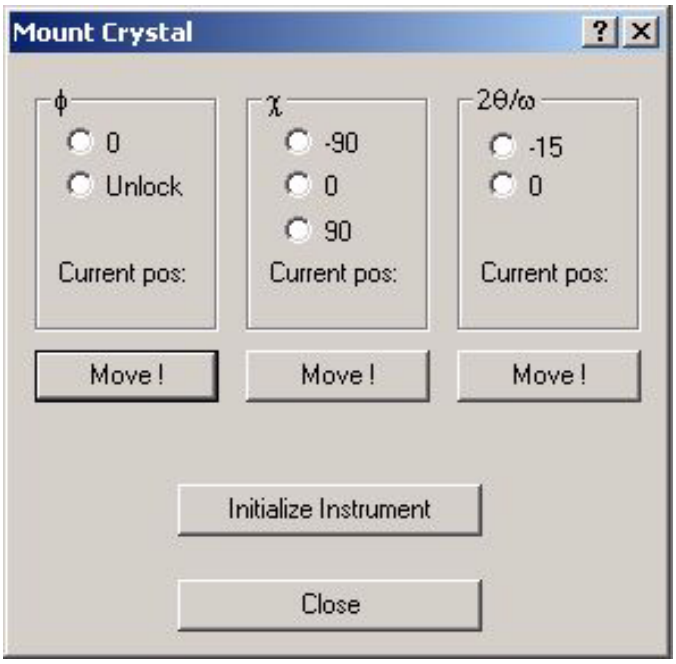


When you have mounted the crystal and locked the rotation axis, then you should click OK and go on to the **Initial Images** step.

Mount Crystal dialog using AFC7

You may skip this section if you do not have an AFC7.

The AFC7 has a special dialog for mounting.



Parameters	Description
Move! ϕ	The **Current Position** of the ϕ axis is displayed. To move the ϕ axis to the position, select **Unlock**, then click **Move!** You can then manually unlock ϕ. Once you are finished mounting, you should move the ϕ axis position back to **0,** after locking the ϕ axis, by selecting **0** and then clicking **Move!** **NOTE**: When positioned at 0, the CCD detector blocks access to the locking screw.
Move! χ	The **Current Position** of the χ axis is displayed. To position the axis, select **-90**, **0** or **90**, then click **Move!** **NOTE**: χ allows adjustment of the sample height. Since the microscope is

Parameters	Description
	mounted at an angle on the χ circle, the χ positions of -90 and 90 are generally used to check the height of the sample. Once the sample has been positioned at the appropriate height with χ at 90, the sample can then be moved to χ at -90 to verify that the height is correctly adjusted.
Move! 2θ/ω	The **Current Position** of the **2θ/ω** axis is displayed. To change position, select **-15** or **0**, then click **Move!** **NOTE:** **2θ/ω** allows the detector to be moved so the χ circle may be conveniently positioned for mounting the crystal. When positioned at **-15** degrees, the detector is towards the back of the generator and the χ circle may be rotated for convenient crystal access.
Initialize Instrument	Click **Initialize Instrument** to initiate the procedure.
Close	Click **Close** to exit the dialog.

Initial Images

Overview

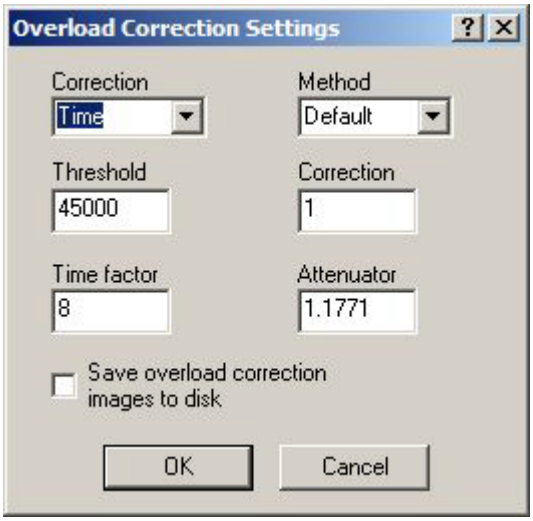
It is often useful to collect screen images before doing a full data collection. This allows you to screen crystal to find one that diffracts well and then index the sample and determine symmetry so that you can either use Strategy to calculate a collection schedule or manually choose a schedule that is appropriate for this sample. For protein work, it is often sufficient to only collect one or two screen images, but for small molecule work you usually need to collect more (e.g., 10 to 20) to get enough spots to index.

Initial Images Dialog

This version of the dialog is for a CCD. If you have an R-AXIS or Rapid, the controls may be slightly different.

The central control in this dialog is the scan table. The scan table is described in detail as part of the Scan State Display in Chapter 4. If you are not familiar with the scan table, it is highly recommended that you read that section.

In addition to the scan table, there are the following parameters and controls available.

Parameter	Description
Dezinger exposure	For CCD cameras, long exposures may record larger number of random zinger events (cosmic or optic taper related). Collecting the image a second time allows for analysis of intensities with respect to zingers. Note: this doubles the number of images and radiation decay effects, and thus the length of time for collection.
Bin mode	2x2 binning is the most common for CCDs. But you may also select 1x1 and on certain detectors 4x4. 2x2 binning means that 4 camera pixels are summed into a single image pixel.
Overload correction	There are various options for handling overloads (i.e. when the intensity for a given pixel exceeds the dynamic range of the CCD; i.e. over 16 bit or 18 bits depending on your detector). When you click Settings… the following dialog opens:  Your options are • Time • Attenuator • None You also have the option of saving overload correction images to disk. This is usually only necessary if you are having problems with the overload correction not being right. But it can be extremely valuable in that case.
X-ray Generator Scripts	If you have JXG installed and active, you have the option of running scripts before and after the data collection. This can be useful to power up before you start data collection and power back down after the data collection is finished, but this is a general mechanism and there are varous scripting options available. These are shown as disabled in this example because it was configured without JXG.

Parameter	Description
Screen Schedules	This is a drop down list with all of the predefined screen schedules. When you select a schedule, the scans in that schedule display in the scan table. Adding new schedules is easier through the **Scan State Display**, but some functions can be done from this dialog by click **Import…** (see below)
Add Scan	When you need to add an additional scan to this schedule, click this button. The values in the scan are copied from the currently selected scan.
Delete Scan	Deletes the currently selected scan
Estimated time to collect	This gives the estimated collection time for both the selected scan and the entire schedule.
Required disk space	Since images are quite large, it is good to check the disk space requirements before you start the scan. When you run out of disk space you may have unpredictable results in CrystalClear and the instrument servers.
Total disk space	This shows you how much disk space there is left on the drive where your User Data Directory is stored. **Available** means what there is now, and **Remaining** is what there is estimated to be after this schedule is collected. If insufficient disk space is available to complete the scheduled image collection request, a CrystalClear warning dialog opens. The collection request may be cancelled to allow the user to free more disk space on the system before continuing.
Image Dir…	When you created the sample, you specified where images were to be collected. It is possible to override the specification at this point.
Preferences…	Preferences are settings for each user about how you want to see information presented and how certain things behave. There is a link to this dialog from here since some of the preferences have to do with how images are calculated (e.g., what the independent variable are for start, stop, and number of images).
Import…	This brings up the **Schedule Manager Dialog** described in the section on the **Scan State Display** in chapter 4. This allows you to easily import schedules from other samples, projects or users.
Errors	If there are errors in your schedule (e.g., it would collide somewhere) an error message with a yellow background with a More… button that tells you more about the error.
Completeness	This is not currently functional.
Save	Most dialogs have the ability to save their settings to the defaults. Clicking on this button brings up the **Default Manager** (see Chapter 3 Getting Started). If you want to change the default settings for the schedules, you can do it here. If you have created or modified a schedule that you want to be available to other samples in this project or for all your projects, you need to Save.
Run	You are asked to verify that you really want to collect this data and then the

Parameter	Description
	collection starts.

Once the **Initial Images** step is running, it brings up **the Instrument State Display** so that you can observe the images. If you want to view these images as they are collected you should click the  icon in the top tool bar. By default this is enabled, but it can be turned off since it adds a little time to the collection.

During the time that the Initial Images step is running, the user interface is “live” so that you can look at already collected images and use other viewers to examine the state of the system. However, you should not try to execute any other steps since it could result in an unintended sequence of events.

Collect Images

Overview

The **Collect Images** step brings up a dialog which is almost identical to the Initial Images dialog. The only difference is that the schedule list is labeled Collect Schedules rather than Screen schedules. For a detailed description of this dialog, see the previous Initial Images section. For a description of the scan table itself, see the Scan State Display in chapter 4.

Strategy

The collection schedule can be created manually or it can be generated by the **Strategy** step. This step is dependent on indexing and previous processing steps and is only available if d*TREK is the processing suite. Strategy is documented in Chapter 8 d*TREK Steps.

Integration

Most steps are mutually exclusive and you should not attempt to run them in parallel. The one exception to this rule is the **Integrate Reflections** step which can run in parallel with data collection. This option is only available when d*TREK is the processing suite. To use it just go on to the **Integrate Reflections** step once data collection has started. With a modern PC it is usually possible to keep up with the data collection so that your integrated data is available shortly after the data collection is completed.

The one risk you run if you do this is that any severe problems in integration could halt your data collection. This is a rare occurrence, but the risk is not zero. If you have an older control PC, there is also a small risk that the timing of data collection or the readout of the image (on R-AXIS systems) could be disrupted by processing while you are collecting. Again the risk is small but not zero.

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8. d*TREK Steps

The d*TREK processing suite includes a set of crystallographic data processing programs originally developed by Dr. James W. Pflugrath and enhanced by Dr. Pflugrath and various members of the scientific and programming staff at Rigaku Americas Corporation. The command line version of d*TREK is available under separate license for Windows, Linux, and Mac. CrystalClear has a version of d*TREK which is packaged into a single server program. The license for this packaged version is included with your CrystalClear license.

The d*TREK processing steps in CrystalClear generally correlate to the separate programs in d*TREK (e.g., Index Spots uses dtindex, etc). However this is not always exactly 1:1 and the parameters may have different names and defaults in CrystalClear and the command line version of d*TREK.

Working with d*TREK

Log Files

Each d*TREK function creates a log file which contains information that is often useful to determine if the function was successful as well as information about the data, crystal, etc. These log files are displayed automatically in the Log File Viewer when a d*TREK function is run. For example, Find Spots runs dtfind which produces dtfind.log. Log files are automatically versioned up to 5 levels so that the second time you run Find Spots, the dtfind.log is for the latest run and dtfind.log.1 is for the run just before the latest run.

Command Line

CrystalClear sends the d*TREK function a command line that corresponds exactly with the command line version of d*TREK. This command line is printed at the top of the log file for the d*TREK function. To see the command sent to dtindex, look in dtindex.log. Looking at the command line in the log file also shows you how the CrystalClear parameters are mapped into the d*TREK command line.

Header Files

Some of the inputs for d*TREK functions come through the command line, but there is also accumulated state information (e.g., unit cell parameters etc) that is passed through header files. Each function reads input.head when it runs and produces an output.head when it completes. These header files have the same format as the header section of an image file. CrystalClear reads the output.head file to update its internal state history and writes input.head before running a d*TREK function. Looking at these header files can help track down a problem, but since they are written by CrystalClear at each step, attempting to edit them to change the behavior of d*TREK won't work.

Environment Variables

CrystalClear does not normally use Environment Variables to control the operation of d*TREK, but since the CrystalClear version of d*TREK is built with the same source code as the command line version, they are still active. Normally you don't need to worry about these and should not set them.

DOS Window

When CrystalClear starts up the d*TREK server, a DOS window opens in your tray. There is usually nothing in this DOS window. This is normal and you should not close this window. If you accidentally close it between d*TREK operations, it is restarted automatically by CrystalClear. It also closes automatically when CrystalClear exits. However if there is an abnormal exit of CrystalClear that leaves this DOS window open, you should close it manually by activating the DOS window and clicking on the x in the upper right corner.

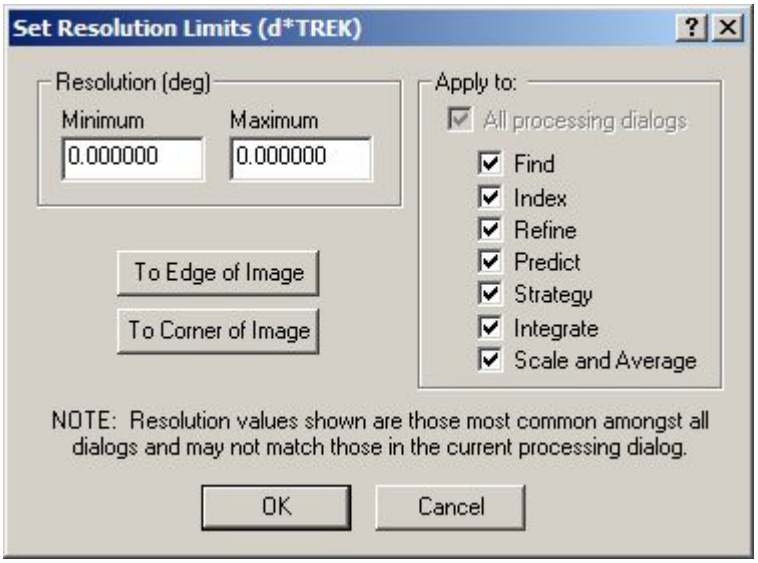
Defaults as 0

The d*TREK convention is that a parameter which is 0 (where 0 does not make sense – e.g., as a resolution cutoff) means “use the internal d*TREK default” or sometimes “calculate a reasonable value”. CrystalClear adopts this convention also. So an input of 0 or 0.0 usually means, “Do the sensible thing”.

Resolution Limits

Since it is often convenient to use the same resolution for multiple steps, there is a dialog set the resolution limits for multiple steps. This dialog can be activated by clicking on the toolbar icon or by clicking on the Set… button provided in the dialogs that contain resolution limit parameters.

 Set Resolution Limits icon



Parameter	Description
Resolution	0.0 means use the default. Otherwise these are the resolution limits to be used in all the checked d*TREK steps. These limits are in degrees for small molecule mode and in Angstroms for macromolecule.
To Edge of Image	Set the resolution limits so that they go to the edge of the image. This is of course dependent on the crystal to detector distsance.
To Corner of Image	Set the resolution limits so that they go to the corner of the image.
Apply to	Check All processing dialogs or select the specific dialogs you want these limits to apply to.
OK, Cancel	Make sure you click OK in order to set the values. Cancel quits without

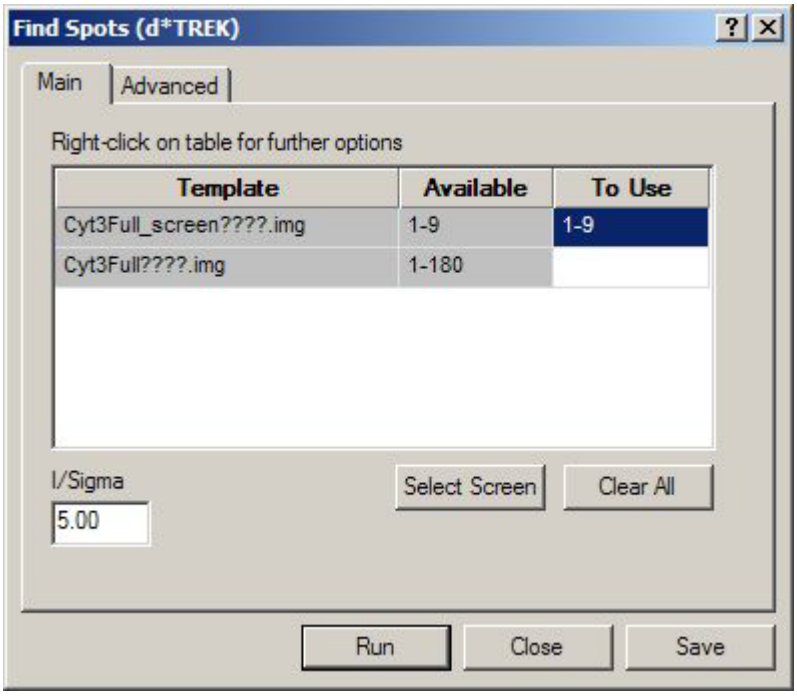
Parameter	Description
	updating the values.

Pre Collection Steps

Find Spots

The first step in processing your data is to find spots to be used in indexing. The **Find Spots** step locates reflection centroids in a single image or a collection of images. Finding reflection centroids requires knowledge of the image properties. For subsequent steps, for example, Index and Refine, to function correctly, you need at least 50 to 70 reflection centroids in the Find spots step. The number can vary, depending on the centroid accuracy and location in reciprocal space.

Find Spots Main Tab

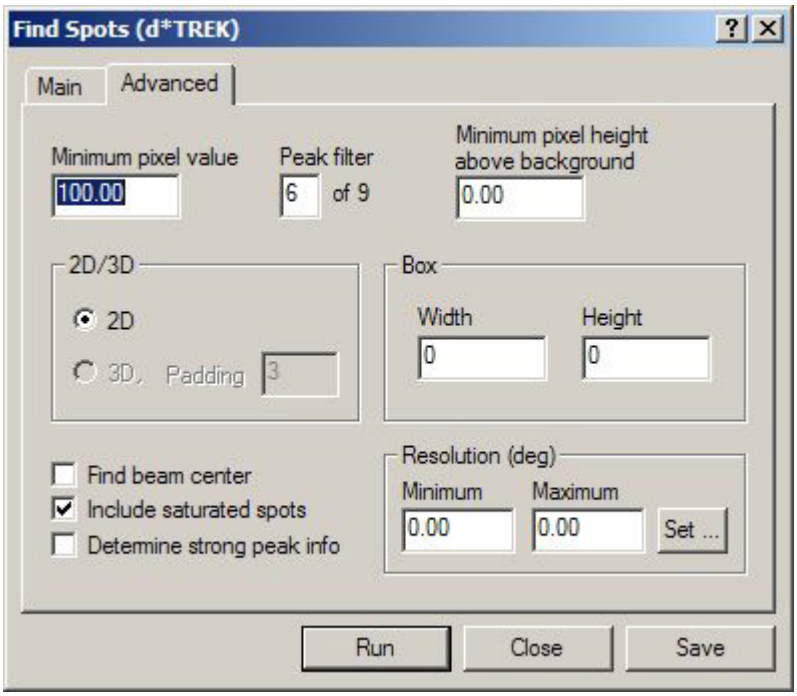


Parameter	Description
Scan Selection Table	All of the screen and collect scans that been completed are listed here. You can fill in the images you want to use in the **To Use** section. See chapter 4 for a description of the Scan Table.
Select Screen	This is a shortcut to select all of the _screen images for Find.
Clear All	This is a shortcut to clear all image selection to let you start over.
I/Sigma	Specifies the minimum I/Sigma that a peak must have to be considered a spot. If you have weak data you may have to lower this number to get enough spots to index. Standard deviation (default 5.0) above background is tested after any non-uniformity correction is applied.

Parameter	Description
	If I/Sigma is 0, **Find Spots** processes faster because it does not calculate average background and its standard deviation. However, a high **Minimum Pixel Value** (in the **Advanced** tab) should be chosen to avoid finding too many spots that are not Bragg reflections.

Find Spots Advanced Tab

The advanced tab has additional parameters. These values seldom need to be changed.



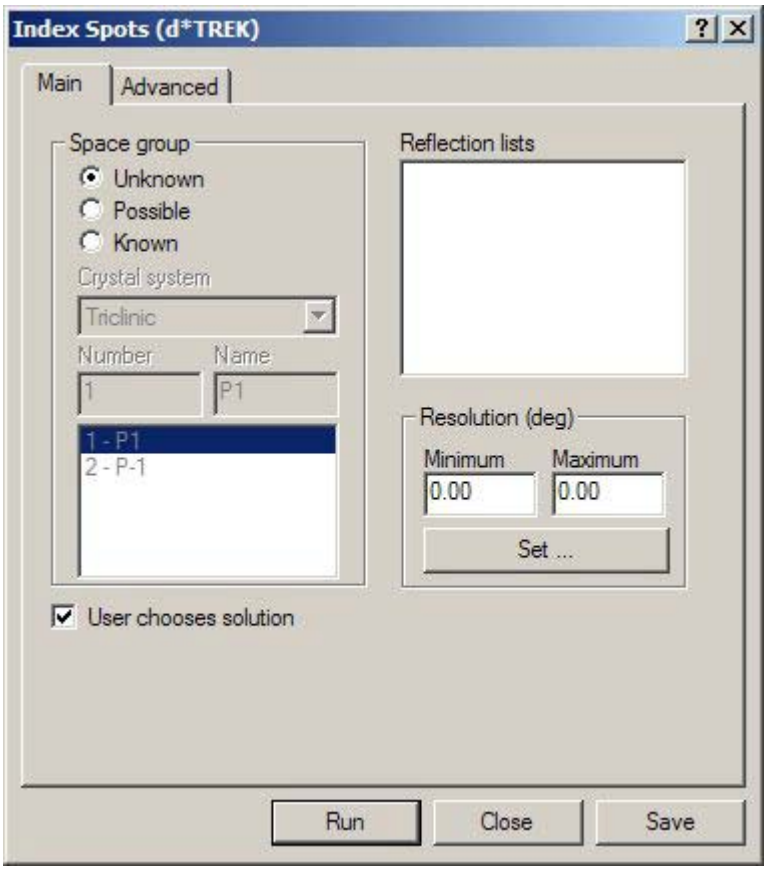
Parameter	Description
Minimum pixel value	Specify a **Minimum Pixel Value** for a pixel to be considered a peak. The Minimum Pixel Value is compared to non-uniformity corrected pixel values. The actual threshold used is the maximum of Minimum and Sigma above the average background. If you are picking up too many spots that are not actually Bragg reflections, you should increase this value.
Peak Filter	Specify the **Peak filter** to help distinguish between true spots and noise in the images. The **Peak filter** is specified as the number of pixels in a 3x3 area that must be above the threshold (Sigma, Minimum Pixel) value for a peak to be considered a reflection. If spots are large, you might use value 9. If the spots are small, you might use value 4.
Minimum pixel height above background	Specify a value for **Minimum pixel height above background** for a pixel to be considered. The actual minimum value for a pixel to be considered part of a peak is the largest of 1. I/Sigma 2. Minimum pixel value 3. Minimum difference between the maximum pixel value found in the peak and the average background pixel value near the peak.

Parameter	Description
2D/3D	Specify a **2D/3D** spot search method. • **2D** search is performed by default. • **3D** search is used to determine peak widths for mosaicity refinement. **3D** search uses a two-pass algorithm to add images to either rotation side. If you are going to examine the peaks with Reciprocal Lattice Viewer, it is best to use 3D.
Padding	The number of padding in images for 3D shoeboxes (in 3D search mode only) at the start and end in the rotation angle direction. In order to completely contain a 3D peak with a 3D shoebox or volume, pad by 2-4 images and have a **Box** size large enough. If any significant peak intensity is on the edge of a shoebox, **Find Spots** rejects the centroid determined for that peak, thus **Padding** is important to ensure that the peaks are recognized properly.
Box	Specify the Box **Width** and **Height**, in pixels, for the box appearing around each spot in the display. A good **Box** size is 3 to 4 times larger than the biggest spot, but without intruding more than half way into neighboring spots. If both **Width** and **Height** are set to 0, CrystalClear determines the box size for each spot automatically which takes extra computational power and time. Default **Box** settings include the entire image less a 1% border.
Find beam center	This attempts to find the direct beam position. It is usually better to do this in **Index Spots**.
Include saturated spots	Saturated spots are rejected for integration since you don't really know their value, but they can still be used in indexing, so the default for this is checked.
Determine strong peak info	This adds information about strong peaks into the log file.
Resolution	These are the standard resolution controls for d*TREK.

Index Spots

The **Index Spots** step indexes reflection centroids in the selected reflection list to elucidate the crystal unit cell dimensions, crystal orientation, and Bravais lattice type. Indexing normally starts with the dtfind.ref file from **Find Spots** and produces a set of cell parameters. In d*TREK the cell is not refined in this step, but rather in the **Refine Cell** step. The indexing in d*TREK is generally very robust, so you don't normally need to use the Advanced tab.

Index Spots Main Tab

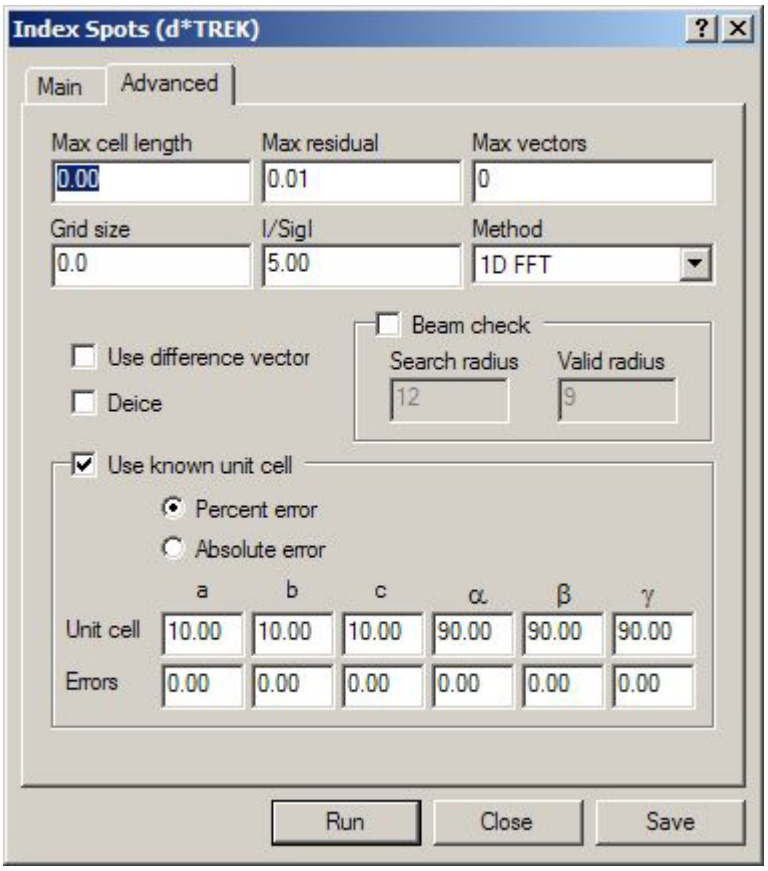


Parameter	Description
Space Group	**Unknown** – You don't know the space group or want to let index work without bias. This is the recommended setting unless you want to force the space group. **Possible** – You have an idea of what the space group should be and would like indexing to bias towards your guess. **Known** – You know what the space group should be and want indexing to attempt to fit that space group.
Crystal System	For **Possible** or **Known** space groups, you should select the relevant crystal system. This then filters the list of possible space groups so that you can select the space group you want. This is just a short cut to help you find the space group and does not actually affect the calculation.
Number – Name	For **Possible** or **Known** space groups you can specify either the space group number or the name. The parameter you are not editing is updated automatically to correspond to the value you entered. Once you choose a crystal system, the list of possible space groups in that system fills in the selection box below **Number** and **Name** and you can simply select the one you want and **Number** and **Name** is updated automatically.
Reflection Lists	This is a list of the .ref files in your sample. The default is dtfind.ref which is usually what you want to use.
Resolution	You may specify resolution limits for indexing. These are in either degrees in 2θ or Ångströms for small molecule depending on your preferences. For

Parameter	Description
	macromolecule they are in Ångströms.
User chooses solution	Indexing produces an ordered list of possible cells with the most likely at the top of the list. If this box is not checked, the top (most likely) solution is used, but if the box is checked then you get the opportunity to see this list and select the solution you think is best.

Index Spots Advanced Tab

The advanced tab has additional parameters that you can specify if the standard indexing is not producing the results you want.

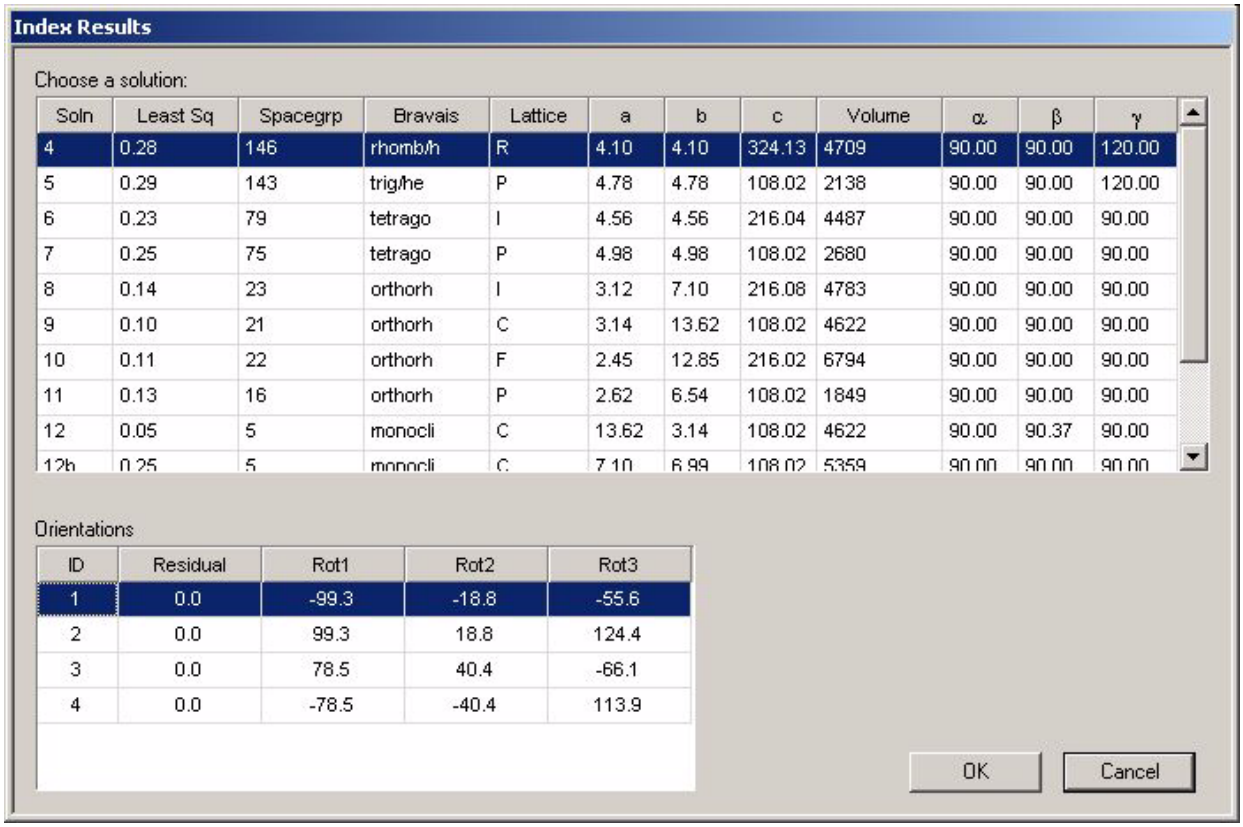


Parameter	Description
Max cell length	This is the maximum length of any axis in the cell. It can be used to force indexing to avoid very large cells even if they have a low residual.
Max residual	**Max residual** represent the residual between the current cell and the triclinic cell (lower is better). Depending upon the degree of accuracy of your detector geometric parameters, the **Max residual** varies (very small is a good fit.) CrystalClear fits the reduced primitive cell to 44 lattice groups. Only those solutions with a residual less than or equal to **Max Residual** are displayed. Typically, residuals greater than 2.5 are not valid solutions. The default of 2.5 reduces output and helps make it easier to choose the correct solution. If using the 1D FFT method, a residual of 1 or higher indicates either the wrong solution

Parameter	Description
	or misaligned hardware.
Max vectors	Maximum number of difference vectors to use in the calculations. With more difference vectors the calculations take more time and the residuals are larger. With fewer differences, the calculations take less time and solutions may be missed. If 0, **Index Spots** picks an appropriate number.
Grid size	Specify the **Grid Size** in Ångstroms for the direct space Fourier map. This parameter applies only if the **Fourier Method** is used. If 0, then **Find Spots** selects an appropriate grid size.
I/SigI	For historical reasons relating to d*TREK, this parameter has two interpretations depending on I/SigI. 1. If **I/SigI**>=1.0 then it specifies the minimum **I/SigI** required for indexing contributors. 2. If 0.0< **I/SigI** <1.0 then it specifies the fraction of the input list reflections to use.
Method	There are several indexing methods available. • **1D FFT** (default and recommended for most uses) The DPS indexing algorithm of Steller, Bolotovsky, & Rossmann is used. About 7300 different directions are examined using a 1D FFT for whether a principal cell axis is parallel to that vector. Three of the vectors with the most order are selected for cell reduction. • **3D Fourier** A 3D cosine Fourier transform is calculated over a real unit cell volume with grid points x. $F = \sum_{i=1}^{n} \cos[2\pi(\vec{d}_i^{\,*} \cdot \vec{x})]$ where $\vec{d}_i^{\,*}$ is the reciprocal lattice vector for reflection *i*. Peaks appear in the Fourier function at the end points of the real cell axes a, b, c and linear combinations of these axes. • **Reciprocal Space**. The reciprocal space method of Sparks.
Deice	Deletes spots (thought to be in **Ice Rings**) found in resolution rings close to the resolution of the powder rings of hexagonal ice.
Use difference vector	**Index Spots** can use the vectors calculated from the reflection centroids (e.g., d(i)), or it can use difference vectors calculated from these vectors: d(i)-d(j).
Beam Check	Select the **Beam Check** option if you think that the direct beam location may not be correct in the image header. If this option is selected, you need to enter a **Search radius** (in pixels) to define a search area and a **Valid radius** to define a valid area, from the input header's direct beam position. The beam check is performed after a pre-reduced cell solution is found by attempting to re-index the data with different beam centers within the search radius pixel value of the entered beam center. If a local maximum is not found within the valid radius pixel value of the original center, the original beam center is used.
User known unit cell	When you know the cell parameters you expect to find, you can check this box and force indexing to attempt to match this cell. You can specify the allowable errors as either a **Percent error** or an **Absolute error**. You need to then fill in the cell parameters and the allowable error. As in many cases 0.0 means use the internal default in d*TREK and not that the error has to be 0.0.

Choosing a Indexing Solution

If you have selected the User chooses solution option, you need to choose an indexing solution before you can continue. An additional pop up dialog allows you to select both the desired indexing solution and the orientation. This table is sorted so that the most probable solution and orientation is at the top of the list. The number of Bravais Lattices in the list depends on the value set for Max residual. The Least Sq column represents the residual between the current cell and the triclinic cell. Lower is better. If User chooses solution is not checked, then the top solution and orientation are used.



Soln	Least Sq	Spacegrp	Bravais	Lattice	a	b	c	Volume	α	β	γ
4	0.28	146	rhomb/h	R	4.10	4.10	324.13	4709	90.00	90.00	120.00
5	0.29	143	trig/he	P	4.78	4.78	108.02	2138	90.00	90.00	120.00
6	0.23	79	tetrago	I	4.56	4.56	216.04	4487	90.00	90.00	90.00
7	0.25	75	tetrago	P	4.98	4.98	108.02	2680	90.00	90.00	90.00
8	0.14	23	orthorh	I	3.12	7.10	216.08	4783	90.00	90.00	90.00
9	0.10	21	orthorh	C	3.14	13.62	108.02	4622	90.00	90.00	90.00
10	0.11	22	orthorh	F	2.45	12.85	216.02	6794	90.00	90.00	90.00
11	0.13	16	orthorh	P	2.62	6.54	108.02	1849	90.00	90.00	90.00
12	0.05	5	monocli	C	13.62	3.14	108.02	4622	90.00	90.37	90.00
12b	0.25	5	monocli	C	7.10	6.99	108.02	5359	90.00	90.00	90.00

ID	Residual	Rot1	Rot2	Rot3
1	0.0	-99.3	-18.8	-55.6
2	0.0	99.3	18.8	124.4
3	0.0	78.5	40.4	-66.1
4	0.0	-78.5	-40.4	113.9

Improving Indexing Results

If difficulties arise in indexing spots, consider the following solutions.

- Ensure that the detector, source, and scan properties in the dialog boxes are accurate. Ensure that the detector translations (direct beam position on detector and crystal-to-detector distance) and scan rotation axis are correct. Ensure that the calculated or entered maximum cell length is large enough.
- Ensure that the crystal is not split, cracked, or twinned. Expose images 90° and 45° away from the original image to verify the crystal integrity.
- Use a different image or set of images in finding spots to get reflection centroids.
- Use reflections from more than one image for indexing. The images should be separated 10° or more in rotation angle. But you generally do not need to use too many images to index with d*TREK. A rotation axis specified incorrectly with a slight tilt not specified in the input header may create difficulties. Refine small tilts in the rotation axis.

- Use three-dimensional reflection centroids from a single scan (contiguous set of images) or from more than one scan.
- Enter a higher sigma or minimum fraction cutoff.
- Accept a triclinic cell, then refine the detector position, and re-index.

Refine Cell

The Refine step refines the crystal, crystal goniometer, detector goniometer, and source properties to reduce the differences between observed and calculated reflection centroids. You can refine these parameters using a reflection file or with images.

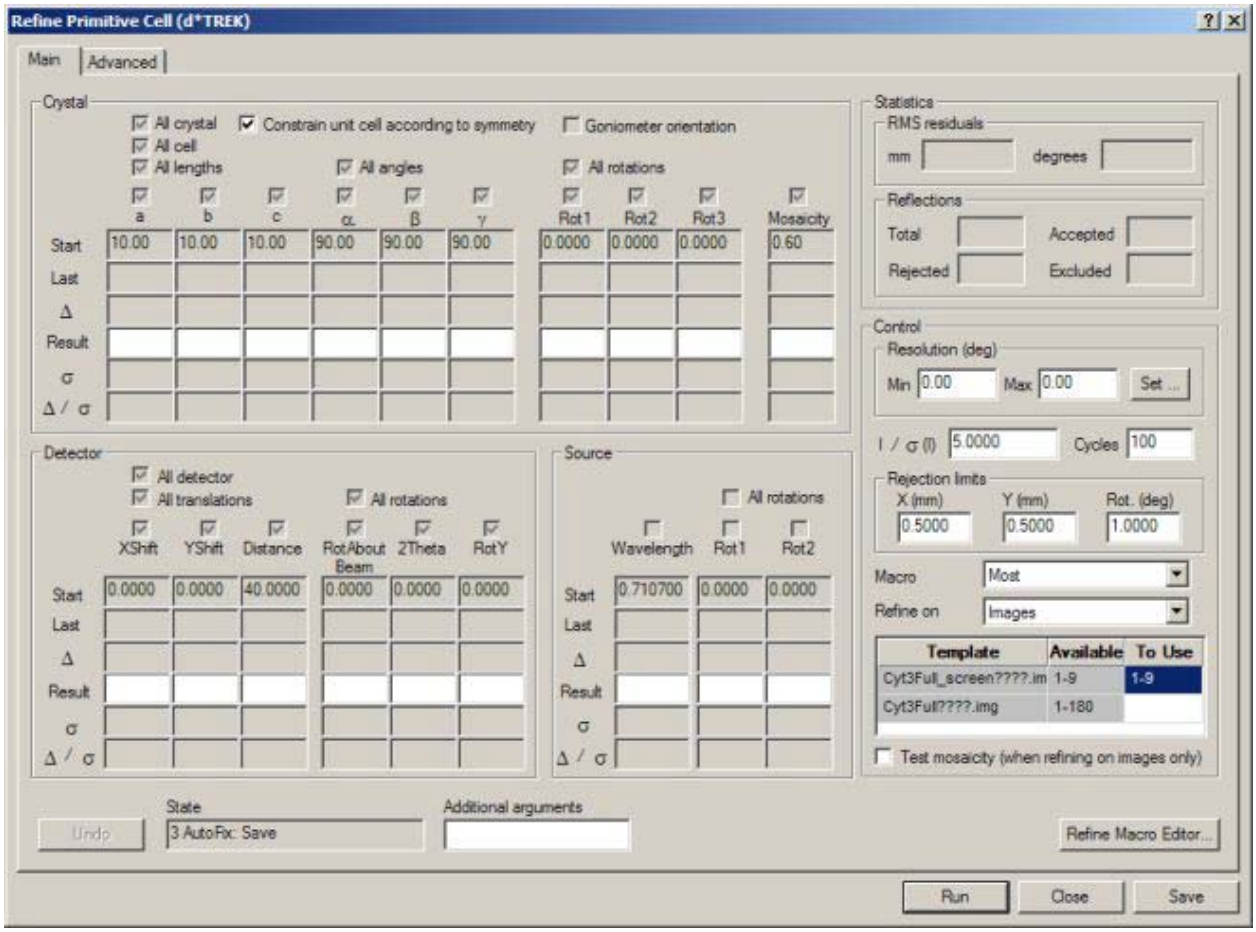
The Refine Cell dialog can appear in a variety of steps for small molecule work. The Process task, for example has a **Refine Primitive Cell**, **Refine Standard Cell**, and **Final Refine Cell**. These all pop up the same dialog and run the same d*TREK refine function, but just have different default parameters.

The Refine Cell dialog is quite imposing but gives you fine control over the refinement and also allows you to monitor the progress of the refinement. But in practice, you do not need to use most of these controls and it is quite easy to run successfully. Refine is normally run simply by selecting the appropriate **Macro**. These macros are predefined and installed with CrystalClear. There are macros for most common refinement procedures, but you can add new macros or edit existing macros for your installation.

In Small Molecule mode, the pre-collection refinement is done twice in the following sequence (see Chapter 5)

- Refine Primitive Cell
- Reduce Cell
- Refine Standard Cell

Refine Cell Main Tab



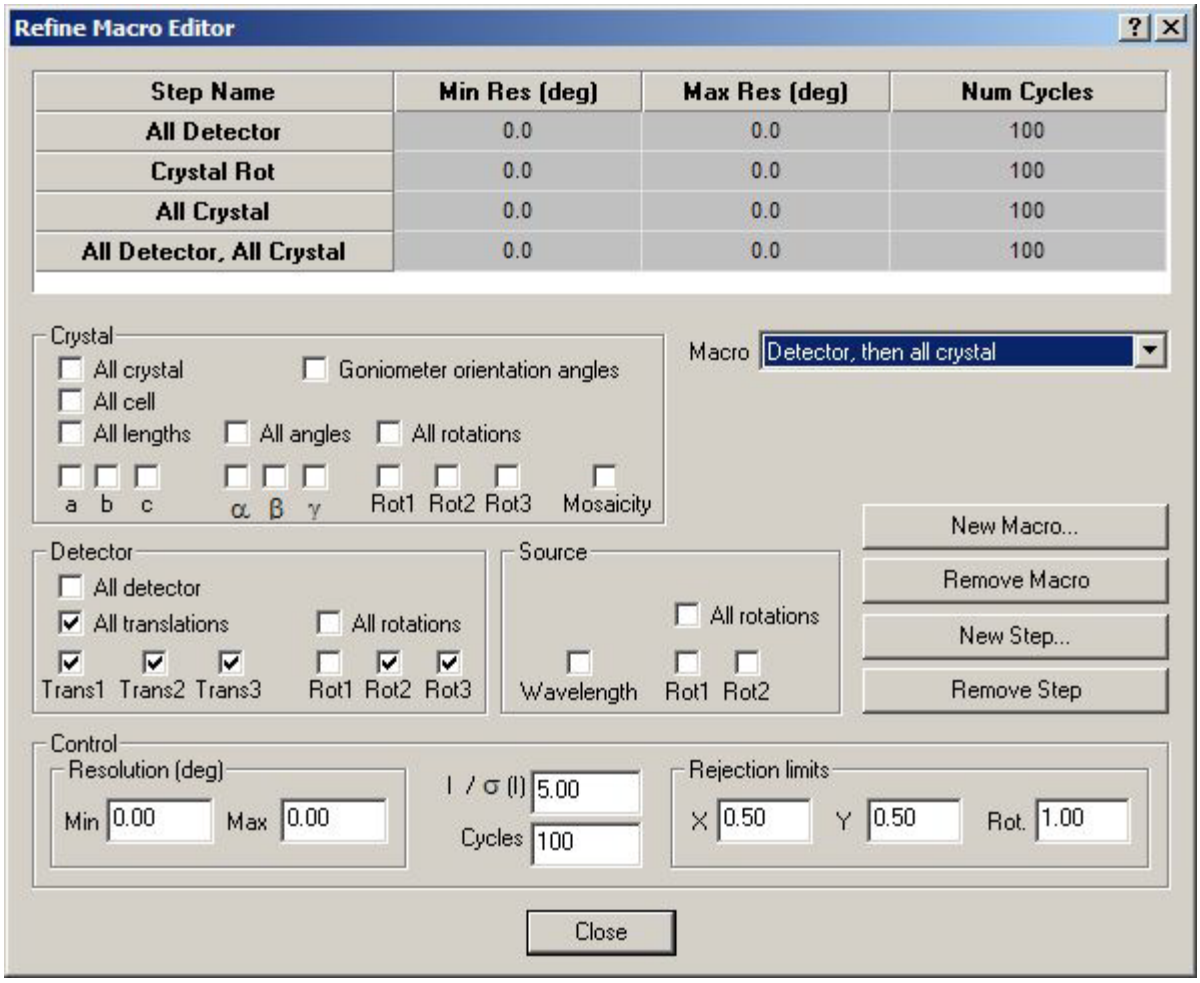
Parameter	Description
Crystal	This section contains a series of check boxes most of which set by the macro you are running. For example, for the **Most** macro which is recommended, the only one you can change is: • **Constrain unit cell according to symmetry** – does not allow refinement to change the current symmetry of the cell. This section also shows the cell information, the orientation angles and the mosaicity before and after refinement. • **Start** – the starting values for the cell parameters before the first refinement • **Last** – the values of the cell parameters after the last refinement. • **Delta** – the differences after refinement between Result and Last. • **Result** – the values of the cell parameters after refinement. These fields are editable, but that is not normally necessary or recommended. • **Sigma –** the error in the cell parameter after the last refinement. • **Delta/Sigma** – the refinement difference divided by the error. This can be used to determine if the refinement has converged (the value is 0 or very small

Parameter	Description
Detector	In the **Detector** section, you can specify which detector parameters to refine. The **Trans1** and **Trans2** options refer to detector translations along the X and Y axes and **Trans3** refers to the detector distance. The **Rot1**and **Rot3** refer to rotations of the detector about the X-ray beam and Y axes, and **Rot2** refers to the detector 2θ position.
Source	In the **Source** section, you can specify which source parameters to refine. **Rot1** and **Rot2** refer to rotations of the source around the X and Y axes. Source parameters are typically not selected for refinement.
Statistics	This section is also read only and reports the refinement statistics • **RMS residuals** (in mm and degrees) • **Reflections.** These are reported counts for **Total**, **Rejected**, **Accepted**, and **Excluded** (masked out) reflections.
I/SigI	This specifies the I/Sigma(I) level below which spots are rejected and not used for refinement.
Cycles	The maximum number of refinement cycles to run. The refinement stops whenever it meets the convergence criteria or when it reaches this number of cycles.
Rejection Limits	These control when a reflection is rejected (i.e. not included in the refinement) if it has to be moved more than the specified amount • **X** max translational deviation in millimeters • **Y** max translational deviation in millimeters • **Rot.** Max rotational deviation in degrees.
Macro	The refinement procedure is usually driven by a multiple step macro. The recommended macro is **Most**. It is also possible to define a new macro or edit the existing macros, but this is not normally recommended.
Refine On	Refinement can work on either a selected **Reflection List** or **Images** (the default).
Reflection List	This is a drop down list that you only see if you have selected **Reflection List** as the **Refine On** selection. This lists all the *.ref files in the sample directory. It is normal to refine on dtfind.ref
Image Selection Table	If you have selected **Images** for the **Refine On** selection, you see the standard image selection controls. The default is to refine on the same images that were used in indexing.
Test Mosaicity	No longer used.
Undo	If you have done several steps of refinement, you can undo the last step by clicking on this button.
State	This is a brief form of the instrument state control (See Instrument State Display in chapter 4). It allows you to select a previous state. This allows you to back up more than one step.
Additional Arguments	If you are an expert in d*TREK, it is possible to enter additional d*TREK arguments that are passed to dtrefine. This is not normally recommended and may compromise the state information.

Parameter	Description
Refine Macro Editor	It is possible to edit existing macros or define your own new macros. This is not normally needed or recommended. If you do feel the need to make a change, it is recommended that you create a new macro rather than editing the existing macros.

Refine Cell Macro Editor

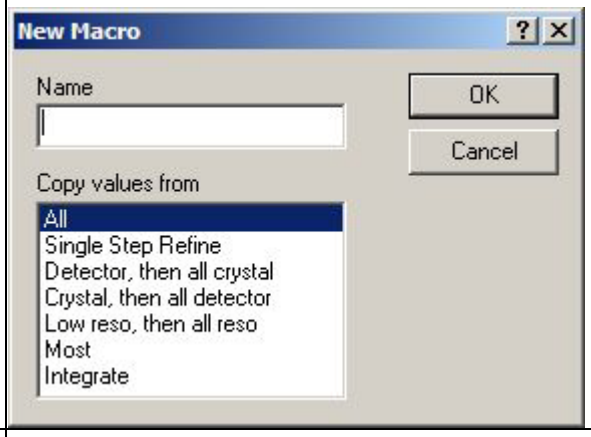
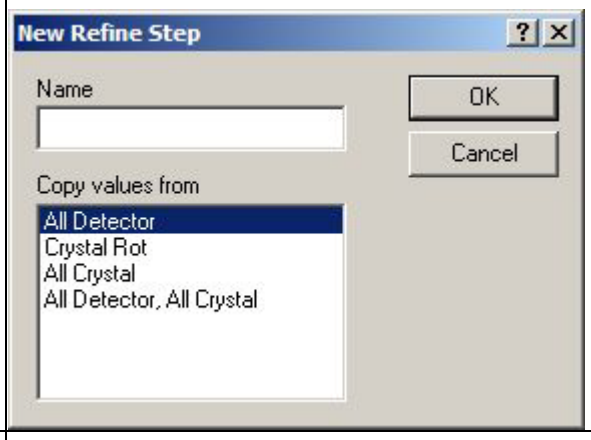
The Refine Macro Editor pops up the following dialog:



Note that the macro is updated immediately for local use when you make a change rather than with an explicit save or write button. However, to save the new macro or edits for later use or for use in other samples you must use the **Save** button on the **Main** dialog.

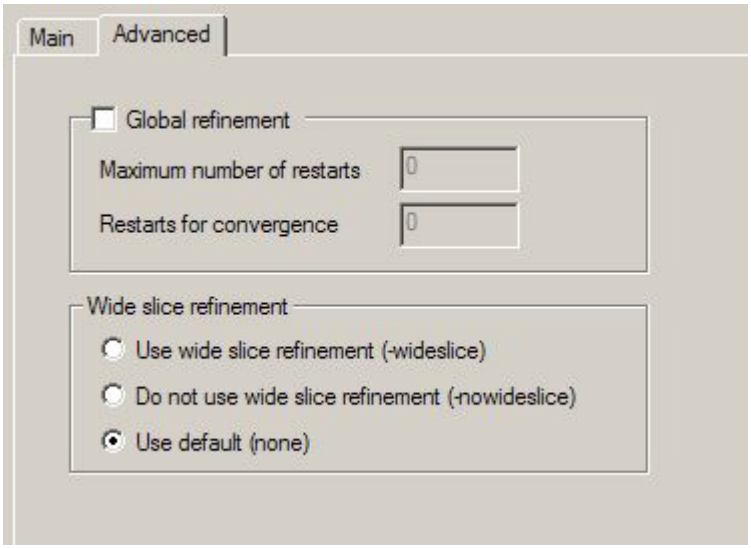
Most of these controls replicate the corresponding controls in the main Macro window, so the list below only shows the edit controls themselves.

Parameter	Description
Macro	This is a drop down list of existing macros. Whatever macro you select is the currently active macro and edits are recorded directly into that macro for this invocation of the refine dialog. For this reason, it is suggested that you create a new macro before you edit any of the controls.
New Macro…	This pops up a dialog which allows you to enter a new name and to copy the

Parameter	Description
	exiting values from another macro. 
Remove Macro	Delete the current macro definition. Once it is gone, there is no way to restore this automatically, so you should be careful doing this. You are asked to confirm the delete.
New Step	Add a new step to the current macro (at the end of the step list). You are given the option to copy the step values from a step of the same name in another macro. 
Remove Step	Removes the first step in the macro. This does not ask for confirmation, so be careful.

Refine Cell Advanced Tab

The Advanced tab is actually much simpler than the main tab for **Refine**.



Parameter	Description
Global refinement	No longer used.
Wide slice refinement	This gives you the option to invoke a special refinement methods for wide sliced images (e.g., more than 3 degrees). However, for wide slice images (e.g on a RAPID or SPIDER) it is often better to use FS_PROCESS. You can open a d*TREK sample and index with d*TREK and then change the mode to FS_PROCESS to integrate. You need not refine with d*TREK at all in this mixed mode.

Viewing refinement results

If you have refined on images, when the step has finished running, the results are displayed graphically in the **Image Display**. This displays the image used by the step, and also shows the reflections used by the step. The reflections are color-coded according to their usage:

- Blue circles – reflections used in the refinement
- Red circles – reflections rejected from the refinement
- Green circles – reflections ignored in the refinement.

The displayed reflections also show, as short red line, the difference vector between the observed and calculated reflection positions. If your refinement results show more red circled reflections than blue, or have large difference vectors displayed, then you should consider using some of the solutions in the next section to help improve your refinement results.

Improving refinement results

If difficulties occur during refinement, use the following solutions.

- If the starting crystal and detector properties are not close to the true values, you might increase the rejection limits so more reflections are included in the refinement. However, there is a danger they are increased too much and refinement diverges away from the true answer. It is sometimes better to use only low resolution reflections at first because the radius of convergence with them is larger. Then use high resolution reflections to get the more accurate results. With good reflection centroids, it is best to use all reflections.
- Sometimes it is useful to fix or not refine a property. With input reflections found in a single image, the crystal rotation around the X-ray source and the detector rotation around the X-ray beam are correlated, so one of them should be fixed. The same is true for the detector translation

along Y and the detector 2θ rotation. It is a good idea to fix one of them. With input reflections from widely spaced rotation values, these correlations should not be a problem. The detector distance can be refined even with reflections from a single image, especially if you have a tetragonal or higher space group.

- Sometimes refinement works better if you fix most properties and refine only a few at a time. Common sense should dictate the order of properties that are fixed and refined. For example, if you know the unit cell parameters from previous work, a, b, c and α, β, γ can be initially fixed. If the detector has just been moved, refine the detector translations first, then the crystal orientation angles, then the unit cell parameters, then everything. The detector distance should be fixed if there are reflections from a narrow rotation range and a crystal principal axis is nearly parallel to the source.

Reduce Cell

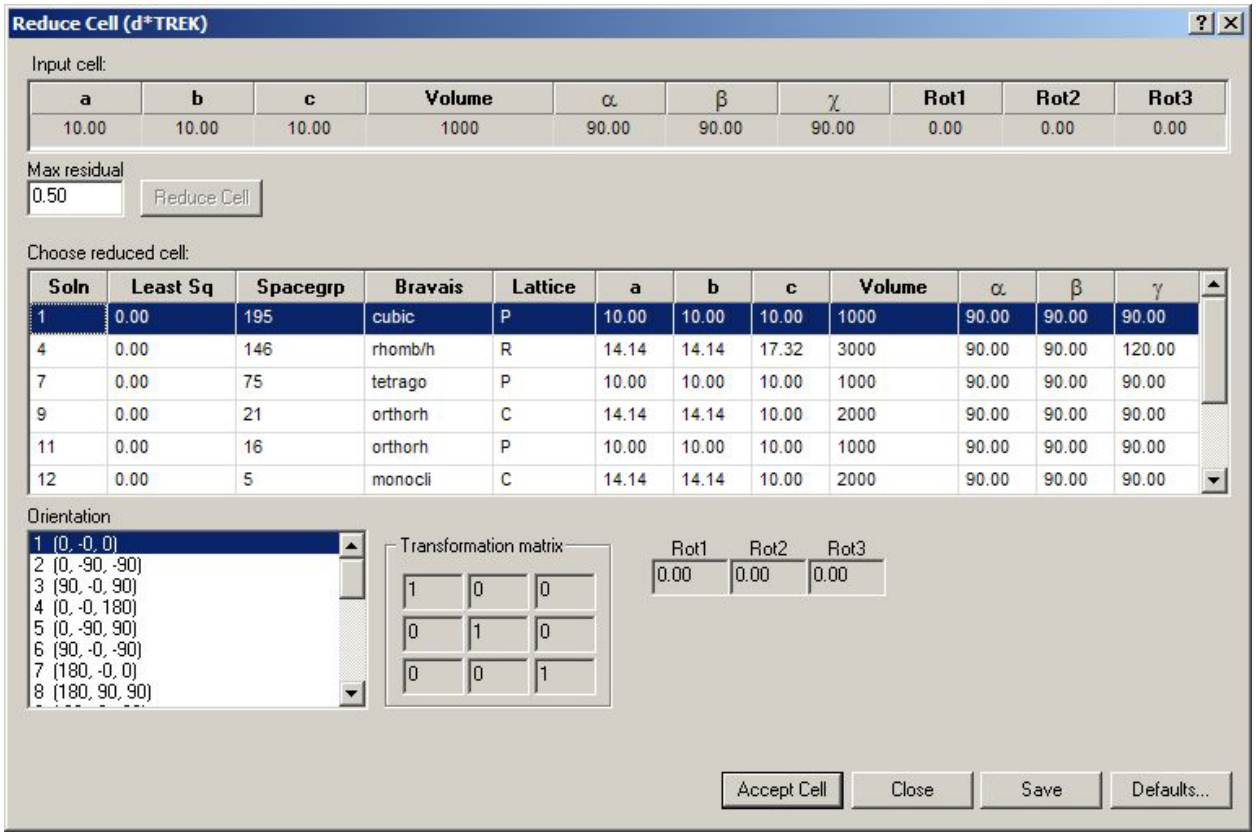
Cell Reduction may be required if:

1. The wrong Bravais lattice was chosen during indexing. After integration, the cell parameters are known with more accuracy, enabling the user to select the correct lattice.
2. The wrong cell **Orientation** was selected. Since the choice of cell orientation is arbitrary, it may be necessary to transform the data to match a cell orientation obtained from another program, or a separate CrystalClear sample.

If all goes well, indexing a sample should be easy and automatic. However, in some difficult cases, determining the correct lattice might require some additional work. For example, after indexing a set of screening images, it might be unclear which lattice parameters should be used. Sometimes, this information is not readily available until the entire data set has been integrated. For this reason, cell reduction (i.e. determination of the correct Bravais lattice) is provided as a separate tool which can be executed before *or after* integration to alter the cell parameters.

There is no dialog that pops up when you click **Reduce Cell**. It runs immediately and then presents you with the opportunity to choose the solution or change the residual and try re-run it.

Reduce Cell Results Table



Parameter	Description
Input cell	These are the current cell parameters and rotation angles when you click **Reduce Cell**.
Max residual	You can change the residual and re-run Reduce cell. For example, if there are no solutions proposed, you might want to double the Max residual and try again.
Choose reduced cell	The options that satisfy the residual specified are presented in a table. You should select the one that you think is right and want to use for the rest of your processing.
Orientation	You also have the option of changing the orientation. The results of this transformation is shown in both the **Transformation matrix** and **Rot1-Rot3** rotation angles.
Accept Cell	Once the parameters are set to your liking, you should click this button to accept the cell. If none of them work and you want to re-index or re-refine, then click **Close**.

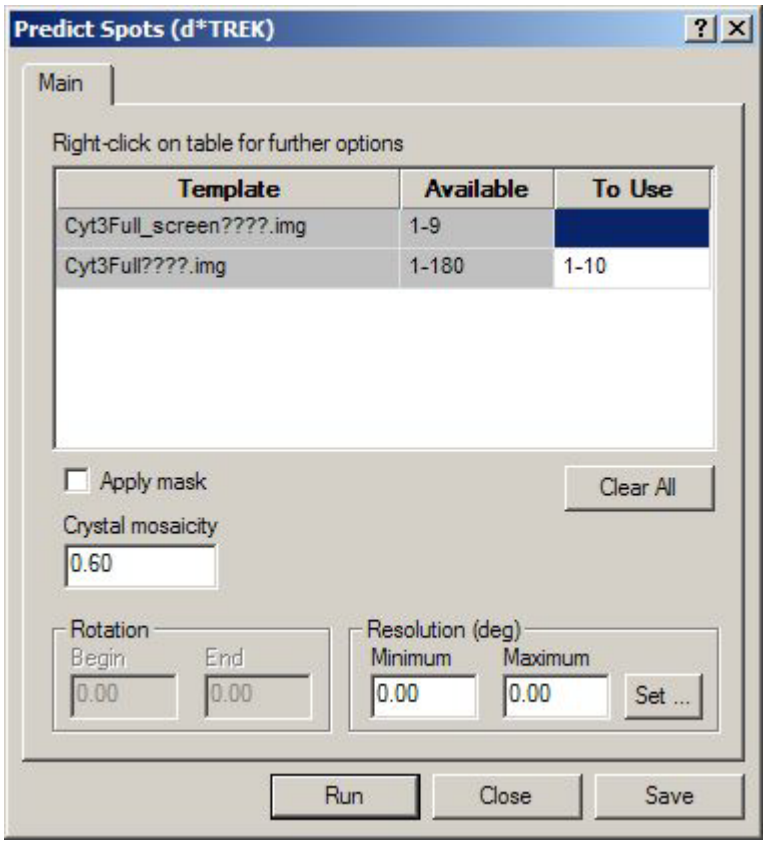
Predict Spots

The **Predict Spots** step creates a reflection list with calculated reflection centroids that can be displayed on more or more images. It is typically used to confirm the results of the refinement step and to better estimate the crystal mosaicity if it was not refined previously. The step predicts the reflections to appear

on a range of images. When the step has finished, CrystalClear can then display the calculated reflection positions, based on the orientation matrix and unit cell, on the corresponding images.

Predict can also be useful when configuring the system to make sure the goniometers vectors in the header are right. If the spots match for one scan, but not for another scan with different goniometer angle settings, then the goniometers vector is probably wrong and you should contact your Rigaku support person.

Predict Spots Dialog



Parameter	Description
Images Scan Table	This is the standard image selection control for selecting the images to use. It is usually only necessary to predict on a few images in each scan.
Clear All	This is a short cut for clearing all the current image selection.
Apply mask	Selecting **Apply Mask** causes prediction to use the mask file specified in the **Setup** dialog (Detector tab) and ignore any reflections masked out by that file. If no mask file has been specified in the **Setup** dialog, it is as if the **Apply Mask** checkbox is unchecked
Crystal mosaicity	If not 0, the specified crystal mosaicity overrides the value found in the input header. **Predict Spots** uses this new value for subsequent calculations.
Rotation	Specifies a different rotation angle **Begin** and **End** in degrees. This overrides the values found in the input image header or in a previous option.

Parameter	Description
Resolution	This is the standard resolution control which limits the resolution range for predicting reflections.

Viewing the results of Predict Spots

When Predict Spots has finished running, the results are displayed graphically in the **Image Display** view. This view displays the first image used by the step, and also shows the positions of the predicted reflections. The reflections are color-coded according to their type:

- Blue circles – normal reflections
- Red circles – reflections that were predicted, but are considered unusable (*i.e.*, lie in a region that has been masked out, *etc.*).

Estimating Mosaicity with Predict Spots

Examining the predicted reflections displayed on an image can help determine if the mosaicity value used is correct or not. Although the refinement does a good job of estimating the mosaicity for sharp reflections, for weakly diffracting crystals or spots that have weak tails, it may be best to run the Predict step multiple times, each with a different mosaicity, in order to determine the best effective mosaicity.

If there is a good match between visually observed reflections and predicted reflections, then the mosaicity is set correctly. If there are many predicted reflections that don't match up with observed spots, then the mosaicity is probably set too large and the prediction should be run again using a smaller mosaicity. If there are many observed spots that are not predicted, then the mosaicity is set too small, and the prediction should be run again using a larger mosaicity.

Using the results of a poor refinement can also artificially inflate the value of the mosaicity. If the crystal orientation refinement is poor, then a larger mosaicity is required to predict the observed reflections on an image.

Troubleshooting Prediction Problems

If you have predicted on multiple images, and the predictions look great on the first image, but are off on the second (and later) image, there are several possible causes for the problem.

- It may be that the rotation range for the second image is not contiguous with the first image. Two images are contiguous if the starting angle for the second image is the same as the ending angle for the first image. If you are predicting on images that are not contiguous, or using a scan that has an image step that is different from the image width, make sure that they are specified as separate images in the **To use** field, rather than a sequence of images (*i.e.*, 1,2,3 instead of 1-3).
- It may be that the rotation axis direction is wrong.
- If you predict on multiple scans and the first scans looks OK, but other scans with different goniometer axis settings do not match up well, you may have a problem with the goniometer vector information in your image headers. If you are processing data from a synchrotron beamline or a newly installed system, it is possible that the goniometer vectors in the header are wrong. Contact your beamline support people or Rigaku support staff if you suspect this is true.

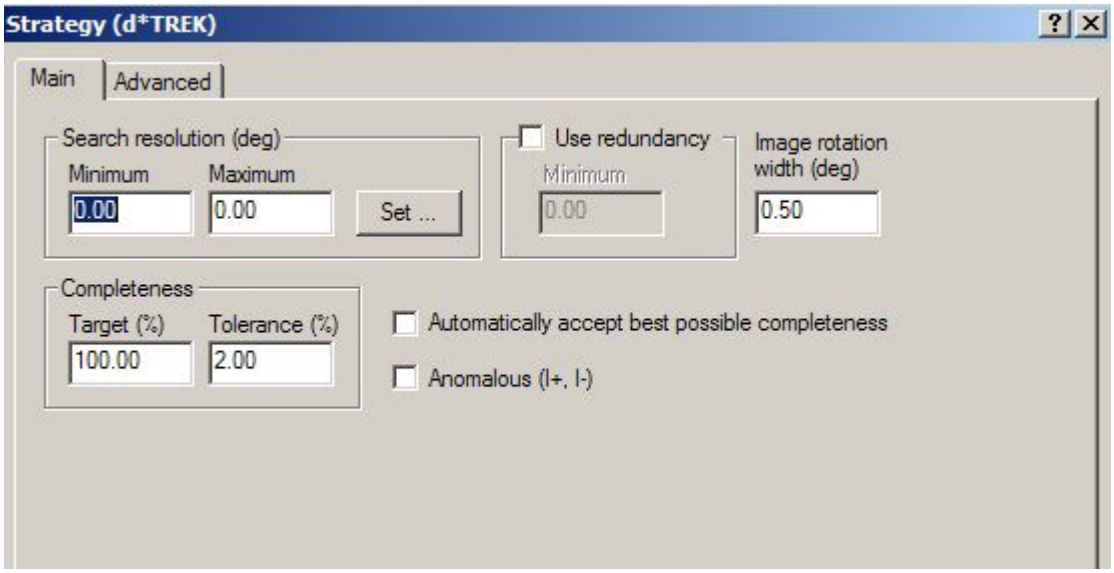
Strategy

The strategy step uses the cell and orientation information to compute a data collection strategy that meets the completeness and redundancy conditions that you specify. You can only run strategy after you have successfully indexed your sample. By using strategy you can often greatly reduce the number of images that you need to collect and still have a complete data set. The output of strategy is a collection schedule which can then be selected in the **Collect Images** step described in Chapter 7.

The strategy algorithm predicts reflections for various settings of detector and goniometer axes and then selects ranges that meet the completeness, redundancy and other criteria and finally chooses the best combination as the suggested collection schedule. So this is essentially a search process and the parameters you enter control the settings that are searched.

Strategy Main Tab

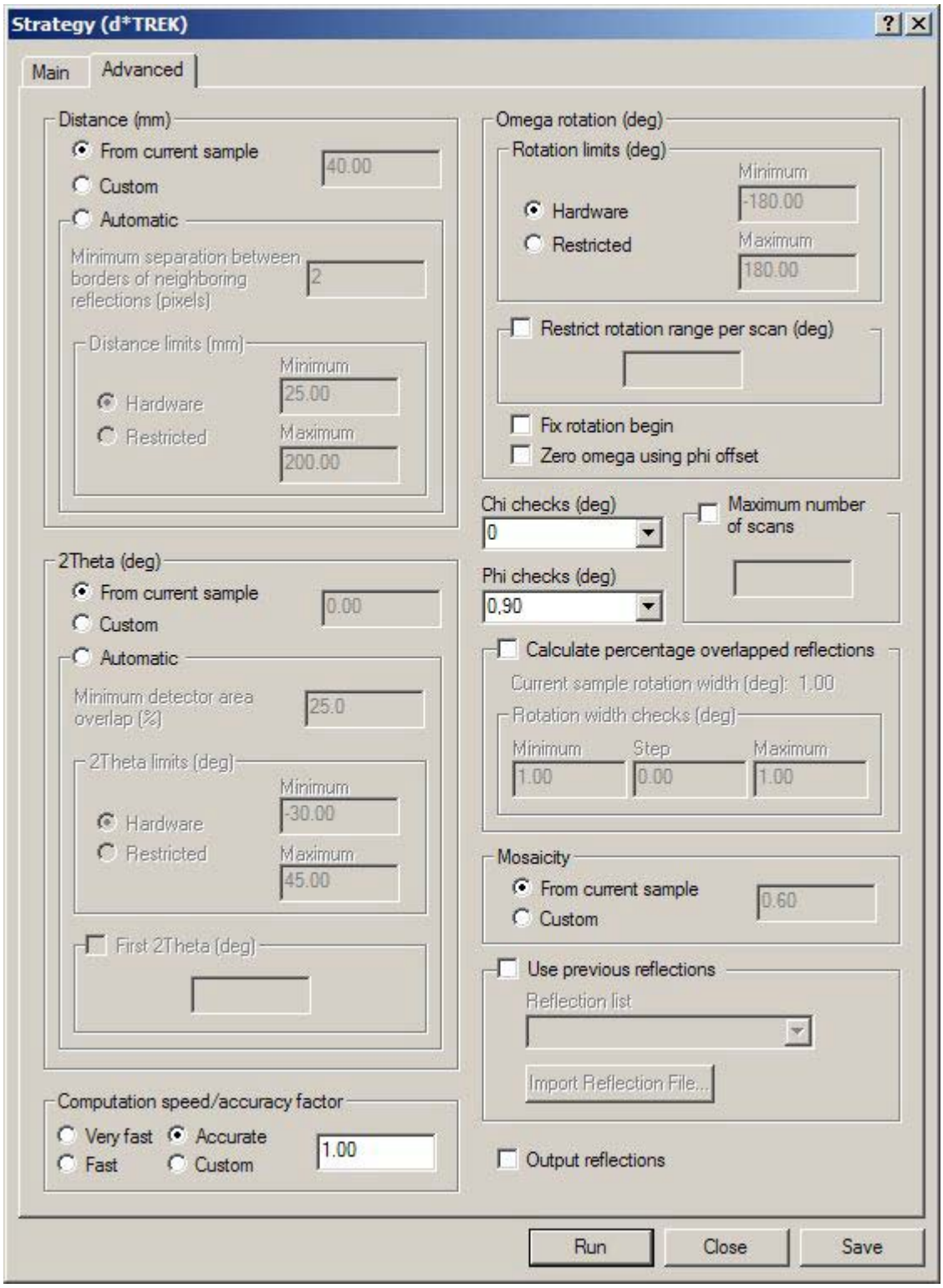
It is usually possible to run strategy with just a few input parameters. Most of the complexity is hidden in the advanced tab and not normally needed. Unless you override these values in the advanced tab, the detector and goniometer settings for the data collection (distance, 2 Theta, etc) are taken from the screen images you collected.



Parameter	Description
Search Resolution	These are the standard resolution controls.
Use redundancy	If the **Use redundancy** box is checked, you need to specify the **Minimum** redundancy that is acceptable.
Image rotation width (deg)	This defaults to the image width that you used to collect the screen images, but if you want to collect with a different image width, you can specify it here.
Completeness	**Strategy** tries to reach the **Target** completeness you specify. The **Tolerance** is how close you must get to this target before **Strategy** is considered successful. So if your Target is 100% and the Tolerance is 2% and **Strategy** can only get to 98.6%, this is still considered a success. However, 95.4% would be considered a failure.
Automatically accept best possible completeness	If you check this box then the best solution that **Strategy** can come up with is accepted.
Anomalous	If this box is checked then Strategy computes a strategy that optimizes the collection on Bijvoet pairs in order to maximize the anomalous signal.

Strategy Advanced Tab

The advanced tab provides many more options to control **Strategy**.



Parameter	Description
Distance	The options for distance are:

Parameter	Description
	• **From current Sample.** This is the default. The distance is taken from the distance used to collect screen images • **Custom.** If you select his, you can enter a different distances for the data collection • **Automatic.** Strategy calculates a suggested distance based on the **Minimum Separation between neighboring reflections** specified in pixels. This maximizes the resolution while maintaining the spot separation that you specify. You can restrict this distance either to the **Hardware** limits for your instrument or further **Restricted** limits that you specify by giving a **Minimum** and **Maximum** distance.
2Theta	The options for 2Theta are similar: • **From current Sample.** This is the default. The 2 Theta is taken from the distance used to collect screen images • **Custom.** If you select his, you can enter a different 2 Theta for the data collection • **Automatic.** For the first two options, Strategy produces a schedule at a single 2 Theta angle. Strategy calculates a set of scans which may have multiple 2 Theta settings. The **Minimum detector overlap** percentage that you specify controls this. You can select either **Hardware** or **Restricted** limits for the 2 Theta values that are produced. You can also check **First 2Theta** to specify the first 2 Theta setting to use.
Computation speed/accuracy factor	This factor ranges from 0.1 to 1.0 and controls how fast and how accurate the Strategy results are. The options are: • **Very fast** (0.3) • **Fast** (0.6) • **Accurate** (1.0) • **Custom** (enter a number between 0.1 and 1.0)
Omega rotation	For the scan axis (e.g., Omega), you have the choice of selecting either **Hardware** or **Restricted** Resolution limits. You can also check the **Restrict rotation range per scan** box which sets the maximum rotation that can be used for a single scan. If you set **Fix scan rotation start**, then all scans have a starting rotation angle value equal to the start of the allowed range specified. You can also check **Zero omega using phi offset** which counter rotates phi so that each scan starts at omega=0.
Chi checks	There is a drop down list with the most common chi (or kappa) angles to use in trying to generate a strategy. Each angle is separated by a comma, so 0,30 means check at chi=0 and at chi=30. You can also enter your own values into this drop down list.
Phi checks	The most common phi angles are listed in the drop down, but you can also enter your own list.
Calculate percentage overlapped reflections	If this box is checked, the **Current sample rotation width** reports the rotation width from the main tab. In the **Rotation width checks** area you can specify a **Minimum** and **Maximum** rotation width to test. The **Step** parameter specifies the step size to use in testing. For example if you specify Minimum=0.5, Step =0.25 and Maximum=1.0, Strategy tests at 0.5, 0.75, and 1.0.
Mosaicity	Since the mosaicity plays a major role in spot size and overlaps, you have the choice of either using the mosaicity **From current sample** or specifying a **Custom** value.
Use previous reflections	If you have already collected data for this sample but would like to collect more data to increase the completeness and/or redundancy, you have the option of

Parameter	Description
	using an existing reflection list by checking **Use previous reflections**. The reflection lists in the current sample appears in the drop down list. If the reflection list you need is in another sample or somewhere else on disk, you can click the **Import Reflection File…** button and import it to the sample and then select it.
Output reflections	If you check this box, Strategy creates a dtstrategy.ref file with the predicted reflections from the last run of Strategy.

Strategy Results

You can look at the results of the strategy calculation in dtstrategy.log, or you can go to the scan table in the **Scan State Display** or the **Collect Images** dialog. The latest strategy run should automatically be selected and you can review the settings for each scan. If you want to rerun strategy with a different set of parameters, both strategy collection schedules appear in the selection list, so that you can select each one in turn to compare them.

Post Collection Steps

Integrate Reflections

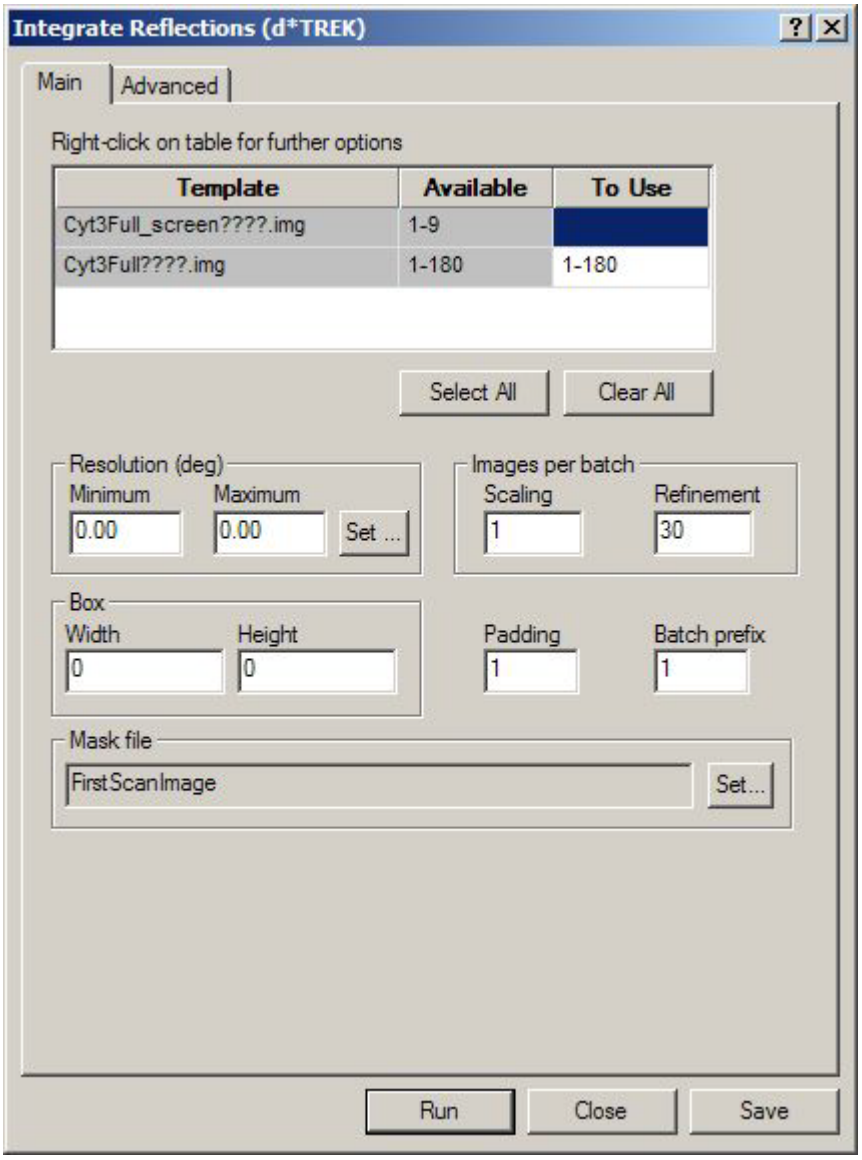
The **Integrate Reflections** step integrates the reflections that appear on a range of images, producing intensity data. The step can be performed after the images have been accurately predicted. The operation may produce several reflection files that are merged as part of the integration. Although d*TREK can only integrate a single scan at a time, CrystalClear can integrate and merge data from scans with multiple goniometer angles in a single step by looping over each scan and performing integration on that scan and then merging the results.

Before integrating reflections, you should create a mask file to define the shadow of the beam stop on the images (see the Image Display section in Chapter 4). Reflections intersecting with the shadow are excluded during data processing. Currently, CrystalClear can only use a mask file for images with a single 2θ orientation. If you have multiple scans with different 2θ values and want to use a mask file, you need to run the Integrate step multiple times, integrating each scan separately, and then merge the results using the **Processing>Reflection Lists>|Merge Reflections...** menu to bring up the Merge Reflection Lists dialog.

The amount of time required to integrate a data set depends on the number of images to be integrated, number of reflections per image, and computer memory and processing speed. With modern computers integration can normally keep up with image collection and so in d*TREK **Integrate Reflections** can be started before the **Collect Images** step has completed. The d*TREK code waits for each image file to show up in the image directory and then it integrates it as soon as the image file appears. This way the data integrated data is available very soon after the data has been collected.

The integration in d*TREK is normally done with a 3D box that spans several images to include the whole reflection.

Integrate Reflections Main Dialog

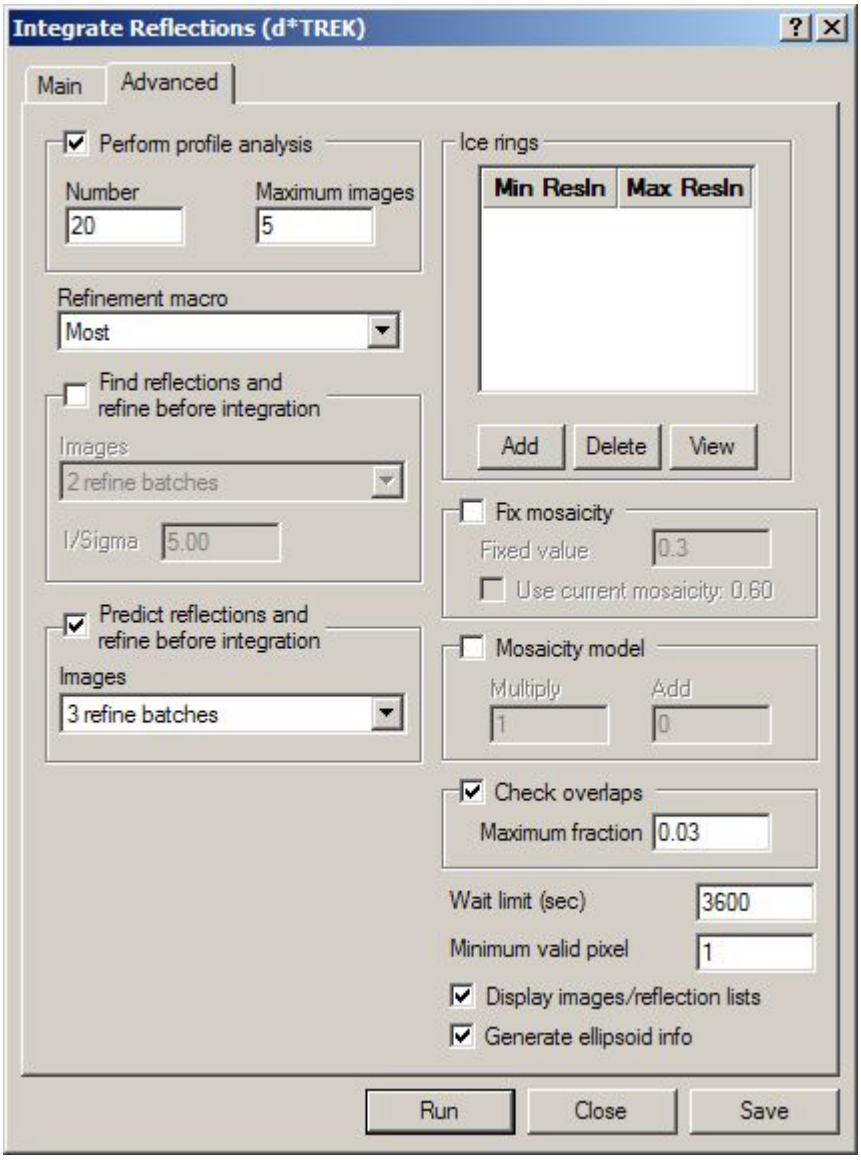


Parameter	Description
Scan Selection Table	This is the standard image selection control for selecting the images to use. Usually you select all images. At the CrystalClear level you can integrate multiple scans at once. Right-clicking a field accesses the pop-up menu to select images using the **Select Scan Images** dialog.
Select All	This is a short cut for setting all the non screen images. This is what you normally use for integration.
Clear All	This is a short cut for clearing all image selection.
Resolution	These are the standard controls for setting limits on the resolution of the reflections which are to be integrated. Values of 0 for Min and Max are interpreted as meaning the whole screen. An option is provided in User

Parameter	Description
	Preferences for switching the units from degrees to Angstroms.
Images per batch	Scaling and refinement are done in batches. • **Scaling**. In the **Scaling:** field, enter the number of images per batch to be used during scaling. 1 is a good number for macromolecular crystals, but if scaling and averaging fails you might want to increase this to something in the range of 2-5. 20 is a good number for small molecule crystals. • **Refinement**. In the **Refinement:** field, enter the number of images per refinement batch. 2 is a good value for viruses, 4 for most proteins and 15 for small molecule crystals. After each refinement batch, integration does a refinement, re-prediction and integration. You may need to use a larger number of images if o you are doing thin or fine slice data collection o your refinements are not stable o you have too few reflections per refinement
Box	A 3D box is used for integration. The **Width** and **Height** of this box are in millimeters. 0 means the d*TREK determines the box size automatically. If you do not set this to 0, you must specify the maximum width and height of the box in the **Width** and **Height** fields, respectively. A good estimate of the values to use can be obtained by examining the spots in the **Image Viewer** view to determine the size of the spots, and then multiplying that value by 3. When specifying an explicit box size, try not to choose a size that includes more than half of a neighboring spot. Also do not make the spot size too small; leave a border around the spot for background determination. Note that when you explicitly set a box size, you are setting a *maximum* box size, and that integration may adjust the box size used to be smaller.
Padding	In the **Padding** field, set the number of images to use for padding. Padding is the number of images added to the integration volume both before and after the predicted rotation width of a reflection. Padding allows for a shift of the predicted peak position or change in crystal mosaicity. For wide-sliced images, 0 or 1 is a good value, and for fine-sliced images, use 2 or 3. If the crystal mosaicity is significantly smaller than the image width, you may want to set the padding to 0. In this case, 0 does **not** mean use the default since it is a valid value.
Batch prefix	In the **Batch prefix** field, set the prefix that you want to use for each scan. You only need to set this if you have multiple scans and are integrating each scan separately. If you are integrating multiple scans in a single run of the **Integrate** step, the prefix is automatically changed for each scan.
Mask file	This gives you the final chance to specify a Mask file for integration. Integration results are normally more accurate if you use a mask file so it is normally recommended.

Integrate Reflections Advanced Dialog

The Advanced tab for Integrate offers a lot more control, but is recommended only for expert users.



Parameter	Description
Perform profile analysis	When this box is checked (the default), then profile analysis is performed during integration. • **Number**. In the **Number** field, enter the number of reflections to use when creating reflection profiles. 50 is a good number for most macromolecular crystals, and 20 or 10 for small molecule crystals. • **Maximum Images**. In the **Maximum images** field, enter the maximum number of images to use when creating reflection profiles. 7 is a good number for most macromolecular crystals, and 20 or 10 for small molecule crystals.
Refinement macro	Refinement is performed during integration. The refinement macro used for this refinement is in a drop down list. The default is **Most**. You should only change

Parameter	Description
	this is there are special reasons why the **Most** refinement is not working.
Find reflections and refine before integration	Integration needs to know where to expect reflections. This is usually done by predicting the reflections based on the current cell parameters and orientation matrix. However, if you check this box it is possible to do **Find** and **Refine** steps on the images. • **Images.** This drop down has various options for how many images to use in the Find process. Note: The number of pre-refinement images is calculated as the product of number of images per batch (for refinement, main tab) and number of batches. At the start of integration, if the software determines this is too few images, it increases the number automatically • **I/Sigma.** Specifies the minimum I/Sigma need for a pixel to be considered part of a spot.
Predict reflections and refine before integration	This is normally checked on. It uses the **Predict** function to predict the reflections that are on the image you are integrating. • **Images.** This is the number of batches that you want to use for prediction (i.e. Refinement batches).
Ice rings	This table allows you to define ice rings or other powder rings. If a reflection profile falls within these rings, it is not integrated. For each ring you define • **Min Resln**. The inner resolution boundary of the ring. • **Max Resln**. The outer resolution boundary of the ring. • **Add**. Add another resolution ring • **Delete**. Delete the current selected resolution ring • **View**. Display the resolution rings on the currently open image in the **Image Display**.
Fixed Mosaicity	The integration process normally refines the mosaicity as it is progressing. You can lock the mosaicity to a fixed value by checking this box. • **Fixed value**. If you know the mosaicity, you can fix it a specific value. • **Use current value**. This uses whatever the current value of the mosaicity is before you start integration.
Mosaicity model	In the **Multiply** and **Add** fields in the **Mosaicity model** section, enter the mosaicity model multiplier (Mos_{Mul}) and addend (Mos_{Add}). The mosaicity used during integration can be modified using the equation: $$Mos_{Used} = Mos_{Refined} \times Mos_{Mul} + Mos_{Add}$$ where $Mos_{Refined}$ is the mosaicity from refinement. When $Mos_{Mul} = 1$ and $Mos_{Add} = 0$, the refined mosaicity is used during integration. When $Mos_{Mul} = 0$, the value of Mos_{Add} is used as the mosaicity during refinement and results in a fixed mosaicity.
Check Overlaps	Select the **Check Overlaps** option if you want integration to check spots for overlap with adjacent spots. When enabled, spots that significantly overlap neighboring spots are excluded from integration. • **Maximum fraction.** A spot is flagged as overlapped with the fraction of intensity shared with neighboring spots is greater than or equal to the

Parameter	Description
	value specified.
Wait limit	d*TREK waits for images to show up during integration. This allows you to integrate as you are collecting data. However, there needs to be timeout in case collection stops prematurely for some reason. This specifies the maximum number of seconds to wait for the image to show up.
Minimum valid pixel	In the **Minimum pixel value:** field, enter the minimum pixel value that can appear in a spot. Spots that contain pixel values less than the value specified (even in their background) are excluded from integration.
Display images/reflection lists	When this is checked the images and the reflection lists are displayed as they are integrated. This gives a good visual feedback to insure that integration is proceeding as expected, but does slow down the process.
Generate ellipsoid info	Select the **Generate ellipsoid info** option if you want to turn on generating profiling ellipsoid information. Turning this one increases the processing time. The results are stored in a file ellipsoids.ref.

Viewing the Results of Integration

While images are being integrated, the results are displayed graphically in the **Image Display** if the View Image Processing Updates icon in the main toolbar is enabled. This slows down integration somewhat, but can be useful especially if you suspect that there might be problems. This view displays the current image being used by the step, and also shows the positions of the reflections being integrated. The reflections are color-coded according to their type:

- Blue– normal reflections
- Red– reflections that were predicted, but are considered unusable (*i.e.*, lie in a region that has been masked out, *etc.*).
- Green – excluded reflections (high Lorentz and polarization corrections, *etc*).

If you have disabled filtering of reflections based on rotation range in the image viewer  , the reflections are displayed as a mixture of circles and squares. Circles are reflections that should appear on the image based on the mosaicity (ignoring systematic absences), and squares are reflections that should not appear on the image. You should see spots in the circles, and no spots in the squares.

Once integration has finished, you should examine the Summary table to determine if any problems occurred. If the parameters drift too much, you may need to fix them and run the Integrate step again.

Scientific Description

This section describes how integration works in d*TREK and discusses issues that help you understand the process so that you can set the parameters in the **Integrate** step to get the best results. The **Integrate** step process a collection of single-crystal, X-ray diffraction images, generating a list of intensities describing crystal diffraction data found on the images.

More formally, a list of estimated intensities and standard deviations is calculated for each Bragg reflection appearing on the data images. The resulting data are stored as a list of reflections which can later be scaled, averaged, or manipulated using other CrystalClear tools, such as **Scale and Average**. After integration, the resulting diffraction data are encapsulated completely in a single reflection list file and may be processed without further reference to the original data images.

Basics

The single most important algorithm in the entire d*TREK suite is integration. An understanding of its basic operation is essential for all users. Before attempting to run the program, become familiar with its overall operation (described below). The basic operation of **d*TREK integration** is performed by **dtintegrate** and **dtprofit** and is conceptually simple:

1. **dtintegrate** uses the crystal lattice parameters (obtained from **Index Spots**) to predict the location of Bragg reflections on the images.

2. **dtingegrate** then, grabs the reflections off the images by drawing a box around each predicted diffraction peak on each image.

Depending upon the intensity of the diffraction spot found inside each box, a special algorithm is used to localize further the spots inside the small, two-dimensional ellipsoidal areas. Each ellipsoidal area is chosen to be the smallest possible region that still covers all statistically significant diffraction information. At the same time, diffuse X-ray background and/or detector-dependent background is subtracted from the summed intensity of each spot, and an estimate is made of the statistical variance.

This process is repeated for all the images provided to **dtintegrate**. Because diffraction spots often straddle two or more images, **dtintegrate** merges the information from spots occurring on multiple images. The resulting data are collected in a reflection list that is indexed according to the Miller Index (hkl value).

The **dtprofit** program is also used (optionally) to obtain estimates of the reflection profile shapes of spots collected with **dtintegrate**. A profile is a mathematical model of what a spot looks like on the detector plate and is a tool used to obtain better intensity estimates for weakly diffracting data. **dtprofit** calculates empirical profiles and uses them to adjust the intensities of weak data by fitting each spot to a profile shape. More generally, **dtprofit** fits weakly diffracting data to two-dimensional profiles generated from strongly diffracting data and uses the fitting parameters to estimate reflection intensities.

The purpose of the following sections is to address the issues involved with integration and to familiarize the user with problems that may be encountered during an integration run. The documentation in this section has been arranged topically, so as to allow the reader the freedom to target specific integration issues. For each issue, helpful hints are provided.

Refinement

To integrate the intensities of Bragg reflections in a single crystal diffraction experiment, their positions in images must be predicted accurately. This may be done provided that information about the source, the crystal, the crystal goniometer, the detector, the detector goniometer, and the sample rotation is known. For the predictions to be accurate, the crystal, source, and detector properties must be refined by minimizing the differences between the observed and the predicted reflection positions.

To predict accurately the reflections for images collected after the start of a scan, the properties of the crystal, the detector, and the source should be refined periodically throughout integration. Experience shows that the values for the detector and source may change during the course of an experiment, therefore it is best to refine these values as one would refine the crystal properties. Moreover, crystal properties may change as the result of radiation damage and of incorrectly mounted crystals (i.e., the crystal can dry out or slip, which would change the unit cell parameters and the crystal orientation).

When does refinement occur?

To predict spot locations accurately (or just to be on the safe side), **dtintegrate** should be configured to refine spot positions periodically. **dtintegrate** allows the user to specify how often refinement is to take place and allows for an optional pre-refinement to take place before integration is started. The pre-refinement makes sure that the spot centroid predictions are good before starting the integration. The user is highly encouraged to enable this feature (**Predict reflections and refine before integration** in the **Advanced** tab).

Refinement Cycles

During refinement, an algorithm is used to detect convergence automatically. If the calculated spot centroids are already close to the predicted spot centroids, refinement may need to execute only a few cycles before terminating. This should be the case for most of the refinements executed during integration, with the possible exception of the pre-refinement done at initialization. If for any reason, the number of cycles should increase greatly, it is wise to investigate the input images for possible problems.

Mosaicity

Perhaps the single, most important refinement parameter is mosaicity. Mosaicity (discussed below in its own section) is refined along with other parameters and is used to predict the width of reflection spots. Unlike the other refinement parameters though, it is not uncommon for mosaicity to vary widely (but smoothly) during an experiment. The user should track the mosaicity (summarized in a table at the end of the dtintegrate log file) and check for abnormalities, such as sudden dips or jumps in mosaicity values. This is often symptomatic of goniometer head problems, slipping crystals, or mis-refinement of spot centroids.

Errors with refinement

Refinement usually functions well and need not be of concern for most data sets. Nevertheless, if problems or abnormalities in the data do occur, these problems often become obvious in refinement. If, for example, hardware related problems are experienced, it is likely that the refinement begins to display abnormally high residuals.

For this reason, a table summarizing the refinement parameters is printed at the end of **dtintegrate** to aid in troubleshooting. This table is helpful for spotting which images are beginning to manifest unusual refinement parameters and high root mean squared (RMS) residuals. It is highly unusual for refinement to fail after it has locked into a solution. If this should happen, it is likely that a problem occurred during data collection.

How often does refinement happen?

dtintegrate allows the user to specify how many images are processed between each refinement. The number of images selected should be to a small number (2-20 images) so refinement may take place frequently. This is specified in the **Refinement** parameter under **Images per batch**. If too many images are processed between refinements, it is possible that the detector parameters would change so much that refinement would be unable to lock in to a new solution. The most common instance of this occurs when the user fails to do a pre-refinement and, therefore, **dtintegrate** gets off to a bad start.

NOTE: Refinement requires a minimum of 50 reflections before it tries to execute. For smaller lattices, this might cause refinement to take longer than the specified number of images before executing a new refinement. Normally, this should not cause a problem, but may cause confusion if the user expects an integration to occur every few images and fails to get one. On

the other hand, an integration log file that completely lacks periodic refinement output tables is suspect and is a good indication that pre-refinement never locked in to a solution in the first place.

Box Dimensions

As mentioned in the general outline of **dtintegrate**, each spot is extracted from its data image by drawing a rectangle around the predicted two-dimensional (2D) centroid and placing all pixels inside the rectangle into memory. Therefore, be sure that the box is large enough to contain the entire spot, while simultaneously keeping the box small enough to avoid excessive memory usage (see section on Memory and disk).

Importance of choosing a correct box size

The Overview of this section (above) states that the 2D box is ideally the smallest box that covers most spots found in the images, with a little room to spare. Because of the wide range of images that **d*TREK** is designed to process, it is impossible to decide before hand (without looking at the spots on the images) how big this box should be. Probably a 40 x 40 pixel box would cover most cases, but this may be excessive. For this reason, **dtintegrate** makes an initial guess about the size of the box and dynamically refines its guess as it gathers a better idea of what spots look like on the detector.

Initial determination of global box size

An initial guess about a global box size (a box size that is appropriate for most spots) is determined by examining the average spot size found during the indexing stage of processing. If this information is not available, it is assumed that the average spot is 10 x 10 pixels. This average spot size is then multiplied by a spot multiplier, which takes a default value of 2.8. Thus, a default window is 28x28 pixels (provided that no information of spot size is passed to **dtintegrate**). This box size is used initially to start **dtintegrate**.

Dynamic determination of local box sizes

Once **dtintegrate** has a box size to work with, it can start collecting spots. As it collects more and more spots, it acquires a better idea of what the spots look like in different parts of the detector and can start choosing the box size of each spot as a function of its position on the detector. Each part of the detector is soon assigned a best box size on the basis of cumulative observations made as integration proceeds. This local method has advantages over using one global box size for the entire image. Experience has shown that spot sizes sometimes vary considerably over different parts of an image because of crystallographic and geometric factors. As with the initial global box size determination, the local box size is related to the local spot size by the window multiplier.

Neighboring spots

As a safeguard against box sizes that become too large, a method is used to limit box sizes so that they do not encroach too much on neighboring spots. In practice, a box should never be large enough to cover entirely a neighboring spot found on the same image. A box that becomes this large is too large and should be restricted to a reasonable size. To solve this problem, an algorithm is used to restrict each box size so that it does not grow to include more than one half of a neighboring spot.

NOTE: This restriction never goes into effect unless the spots are nearly overlapped on the image.

Errors with box dimensions

Most box dimension errors occur when the box dimensions are systematically chosen too small. The tell-tale sign of this is a high number of on-edge errors in the error tables at the end of **dtintegrate**. An on-edge error occurs when the spot found in a box is too large to be covered entirely, thus causing the spot to butt up against the edges of the box or actually to jump outside of it. In addition, a box that is too small

might have background estimation problems. The background of a spot is determined by using the pixels that are inside the box, yet outside the spot ellipsoid (see section on Ellipsoidal localization). Even if the box manages to barely cover the spot, very few pixels may be left for background determination, thus creating problems.

NOTE: The box size is a very crucial parameter and can greatly affect the number of reflections rejected in the data set. Fortunately, the automatic box size determination is robust, therefore the user hardly ever needs to override the default operation.

User specified box dimensions

On the rare occasion in which the user might wish to override the default operation, a fixed box size may be specified. When choosing this feature, the user effectively disables all automatic box size determinations and forces every box to have exactly the same size. This feature should be used with care and only when the automatic box size determination fails. The parameters are in the **Box** section of the **Main** tab.

Mosaicity and Reflection Widths

Mosaicity is a number that relates the position of each reflection on the Ewald sphere (as it is diffracting) to its expected width in degrees (i.e., the number of images that it intersects). The function that computes this relationship is known as the mosaicity model. This function also accounts for a variety of other effects, such as the x-ray spectral dispersion, errors in Lorentz factors, and beam divergence positions of the reflections. Fortunately, these other variables are either known fixed values or may be computed easily during the integration. Mosaicity, on the other hand, is not fixed and may be expected to vary over the course of an experiment.

As mentioned in the general overview, each reflection is actually the amalgamation of one or more spots collected on adjacent images. The number of images on which a reflection is found is a function of its mosaicity- dependent width. The larger the width, the more images the reflection lies on and the more spots that must be collected to obtain the entire reflection. Mosaicity is, therefore, the primary variable that determines how many 2D spots must be collected and processed for each reflection.

Importance of choosing a correct mosaicity

Mosaicity is important for two reasons:

1. For strong spots, it provides a way of determining which spots go with which reflections. For example, two spots on two adjacent images might belong to distinct reflections (i.e., the reflections have different hkl values). Nevertheless, if the mosaicity is too large, the spots might become lumped into a single reflection with a large width. This would be incorrect.

2. For weak spots (where the spots are perhaps only 2 sigma above background), it is important to know the mosaicity because the actual spots are not there to help us. When integrating weak spots, the calculated reflection width must be known. Fortunately, much of the weaker data spends less time in the diffracting condition and, thus, is likely to have a lower rotation width. This, in turn, helps to make the data less sensitive to the mosaicity value than one might expect.

Safeguards against bad mosaicity values

Even when the mosaicity value is unreliable, it should be noted that **dtintegrate** uses a variety of other techniques as safeguards against mosaicity values that predict incorrect or inaccurate rotation widths. First, **dtintegrate** allows the user to specify a padding value (**Padding** on the **Main** tab) that tells **dtintegrate** how many images beyond the current image it is allowed to search to find additional spots belonging to the reflection. This affects primarily the strong data containing spots that would otherwise be ignored because a reflection width was too small (i.e., the mosaicity was too small).

Second, **dtintegrate** makes sure that each spot is contributing to one reflection only. If for example, the mosaicity is too large, some spots lying half way between two reflection centroids might get included in both reflections. To solve this problem, a special algorithm, taking crystallographic considerations into account, assures that every spot is included in one reflection only.

These safeguards keep the effects of bad mosaicity values down to a minimum. However, it should be noted that weak data are much more susceptible to bad mosaicity values and are still affected.

Initial value of mosaicity in input to dtintegrate

So what is a good initial value for mosaicity? Mosaicity may not be well determined prior to integration. During the prerefinement step of integration, a reasonable initial estimate of the mosaicity is made (see section on Refinement). It is also possible to visually refine the mosaicity by running the **Predict Spots** steps with different **Mosaicity** values until the prediction looks good. The determination of mosaicity is probably the best reason to always do a pre-refinement. Once the mosaicity is set, it should vary only gradually as the experiment progresses.

Fluctuating mosaicity

The degree to which mosaicity can fluctuate without significantly affecting data quality is dependent mostly on the image rocking width and the size of the crystal lattice. In general, smaller lattices and/or larger rocking widths tend to exhibit greater fluctuations of the refined mosaicity value than larger lattices and/or smaller rocking widths. For example, smaller lattices (on the order of 20 to 40 Å on an edge) can sometimes exhibit statistical mosaicity variations exceeding 50% of the average mosaicity value!

This behavior should be regarded as being undesirable because it severely affects the integration of weak data, which must rely highly on the calculated rotation width. If this kind of randomly fluctuating mosaicity is exhibited during integration, it may be wise to disable the automatic mosaicity.

Fixing the mosaicity

Fixing the mosaicity is sometimes a necessary step for data sets that do not exhibit stable mosaicity profiles. Using the value from pre-refinement (or from a previous refinement done by **Refine Cell**) is often a wise choice that probably improves data quality. Ideally, however, one should rarely need to resort to this option.

Image Issues

d*TREK has been designed to accommodate a large range of detectors, image formats, and data collection techniques. A position-sensitive detector creates an image file from the photons that it has detected during an exposure of the sample. An image contains a 2D array of values that usually correspond to the number of counts detected at each pixel location on the detector. Images need to be corrected for deficiencies introduced by the detector and experiment. For example, electronic position-sensitive detectors geometrically distort the detected signal, such as a diffraction pattern. Each component of these detectors—phosphor, fiber-optic tapers, lens, image intensifiers, SIT tubes, and CCDs—introduces potential problems that are difficult to avoid when designing and building detectors. Software is used to correct for the deficiencies in images introduced by the hardware.

The deficiencies in the images are spatial distortions, pixel inhomogeneity of response, saturated pixels, defective pixels, and pixels blocked by beamstops. To treat these problems effectively, additional information, such as a mask file, needs to be specified to the processing program.

Defective pixels

Defective pixels arise for a number of reasons. A defective pixel is one that does not give a count that is proportional to the photons registered. Some pixels are dark, that is, they always yield zero or very low counts. Other pixels are always bright; they give high counts that are independent of the photons detected. Still, some other pixels can give random counts. Defective pixels must be flagged and not used by a data processing program. A mask file is used to flag defective pixels.

Saturated pixels

A saturated pixel is one for which the counts are beyond the range within which they are proportional to the detected photons. Each detector can exhibit both local and global saturations. The data processing software needs to detect and treat Bragg reflections with saturated pixels.

Spatial distortions

Spatial distortions need to be corrected so that pixels can be accurately mapped to millimeters and vice versa. These mapping functions predict where Bragg reflections fall on the detector and on the millimeter coordinates of peaks found in images. Most detectors (including all Rigaku detectors) produce corrected images, and a simple pixel size (e.g., 0.1 mm x 0.1 mm) may be used. For other images, **d*TREK** needs spatial distortion tables that **d*TREK** can create using the **dtdistor** or the **dtcalibrate** program.

Inhomogeneity of response

The inhomogeneity or nonuniformity of response needs correction so that counts in individual pixels may be correlated with the incoming signal (photons). Response may vary over the detector because of variations in phosphor thickness, fiber-optic taper properties, pixel area, fixed-pattern noise, and other electronic noise. Again, these corrections are usually made before an image is written to a file, but d*TREK can apply the corrections later if a nonuniformity of response file is available.

NOTE: This nonuniformity of response file cannot be created by the users because they lack the needed specialized calibration setup. The file is created by Rigaku after performing careful experiments with the **dtcalibrate** program. This is done when the detector is being tested and released from the factory, so generally you do not need to worry about this.

Varying beam intensity

Laboratory X-ray sources are stable and generally produce a stable beam. By contrast, synchrotron sources produce a beam that varies in intensity because of changes in the number of particles and their orbit within the synchrotron. Sometimes, a beamline has an incident beam monitor. The counts from such a monitor may be applied by the **dtintegrate** program.

Oblique incidence factors

X-rays are absorbed by matter, which includes the air between the X-ray source and the crystal, the air between the crystal and the detector, the phosphor of the detector, and any covering of the detecting element (i.e., black paper, beryllium, etc.). Corrections depend on the thickness of the absorbing matter and are of two types:

1. Type 1 reduces the number of counts seen in the detector, that is, X-rays are absorbed.

2. Type 2 increases the number of counts seen in the detector. This would occur if the travel path in a phosphor that converts X-ray photons to light photons is longer than a standard reference path.

Spot Localization

After a box has been drawn around each spot, it is the task of the ellipsoidal localization algorithm to find the smallest area that covers the diffracted spot. A smaller area is preferred because this reduces the contribution of background noise to the summed peak intensity. After each spot has been assigned an ellipse, another algorithm verifies that the spot has been mostly covered by the ellipse and that the covered peak shape is consistent with the expected profile of a diffraction peak (i.e., it is not a zinger or a streaked spot).

Choosing an ellipsoidal area for strong spots

For strong spots, the algorithm tries to draw an ellipse around the most intense pixels of the spot and, then, expand the ellipse to cover the less intense pixels in the spot. The resulting ellipse should be located well within the 2D box and should have its edges at or near the calculated background level (see section on Background Determination). At the same time, the ellipse should not violate crystallographic constraints by getting too near to other spots on the image plate. Given that all of these conditions hold, the spot intensity is calculated using all pixels falling inside the ellipse.

Choosing an ellipsoidal area for weak spots

Because the ellipse for strong peaks is fully determined by three numbers, it is possible to store and calculate the average of these three numbers and use the average to predict what an ellipsoid for the weak spots will be. Thus, for weak spots, the average ellipsoid shape of nearby strong spots is used to calculate an ellipse (because one cannot see what the weak spots look like, it is preferable to use the information from the strong spots, if they are available). At the same time, systematic differences between observed spot centroids and calculated spot centroids for nearby spots may be taken into account so that the weak spot centroid is shifted appropriately.

On-edge errors

On-edge errors occur when the spot ellipsoid jumps outside of the bounding box. This is caused either by badly refined detector parameters or by a box size that is too small (see section on Box Dimensions). If many on-edge errors occur, then these two things should be checked first. These errors have also been known to occur with very noisy images, when determining the ellipsoidal shape can become problematic.

Crystallographic bounds

Even for ellipsoids that lie well within the bounding box, problems can sometimes crop up when the ellipse covers pixels that should belong to the spots of other reflections. Whereas overlapped spots are eventually detected by the overlap detector (see the section on Overlaps under Merging reflections), one can still benefit by rejecting spots that seem to intrude into the space of other spots. Such spots might have large thermal diffuse scatter or streaks resulting from bad optics or detector problems. In either case, it is wise to reject them as soon as possible so that they do not affect spot shape calculations or centroid calculations. Any reflection containing spots that are rejected in this manner are flagged with an hkl error.

Weird spot shapes

Sometimes, spots that are affected by zingers or twinned reflections have bizarre spot shapes. These reflections sometimes have a poor bounding ellipse that only partially covers all statistically significant pixels. If, for any reason, the ellipse fails to cover some of the spot, it is rejected.

Fuzzy spot shapes

On the other hand, spots that do not fit fully into their ellipse are not necessarily abnormal. A noisy detector or a detector with raster problems sometimes has fuzzy edges that might cause the ellipse to cover too few pixels. If this is the case, then the spot is flagged as being fuzzy but is still processed. Fuzzy spot errors are printed in the summary table at the end of **dtintegrate**.

Merging Spots into Reflections

Perhaps the single, most difficult task in integration is deciding which spots belong to which reflections. As mentioned in the mosaicity section, this task is accomplished by looking at the reflection widths calculated as a function of mosaicity. However, there are other finer points that must be considered when merging spots. In particular, one must ascertain that the spots merged actually represent significant data, and that the spots do not overlap other reflections. Checking these things when merging the spots can improve data quality.

Integrating noise

Reflections that are composed of three or more spots sometimes contain most of their intensity in the centermost spot and only a little bit of residual intensity in the endmost spots. If the integration routine used all of these data, there is a possibility that the variance of the data would actually go up as a result of the extra background noise that is inevitably added. For this reason, strong reflections (say 5 Sigma above background) do not include weak spots (say 2 Sigma above background). On the other hand, weak spots continue to use all the data available.

Diffuse tails

When merging the spots in a reflection, the spots that lie farthermost from the reflection's rotation centroid are called the tails. The spots in the tails are often significantly weaker than spots located close to the rotation centroid, but are visible only on strong reflections. Sometimes, however, very strong reflections exhibit abnormally long diffuse tails that can drag on much farther than the actual width of the spot calculated from the mosaicity model.
These diffuse tails may be caused by thermal diffuse scatter or by X-ray source problems that affect the Ewald sphere. Unfortunately, this phenomenon might mislead integration to think that the mosaicity is larger than it actually is. To correct for this, the rotation widths that are used to refine the mosaicity are trimmed so that they include only the strong spots in each reflection. This tends to result in a slight underestimation of the mosaicity, but this is corrected for when padding is used.

Detecting overlapping spots

When spots are close together (perhaps as the result of a large crystal lattice), they sometimes intersect on the same image. On the other hand, if the reflections have large rotation widths, they might intersect with each other on different images. In either case, one can imagine that the overlapping pixels that are shared by any two reflections are, in fact, somewhere in the no man's land between them.

Reflections containing overlapping spots are called overlaps and are treated all at once immediately before profile fitting. A special algorithm traverses through the accumulated integration data and attempts to find pixels that are contributing to more than one reflection. For each pair of intersecting reflections, a sum is made of the total intersecting intensity. For each of the reflections, if this sum is larger than a user specified fraction (see section on -checkoverlaps option) of the reflection intensity, then the reflection is rejected as being overlapped. This may sometimes lead to the rejection of one of the intersecting reflections but not of the other. If for example, a weak reflection intersects a strong reflection, it might be that the weak reflection's intensity is compromised more than the strong reflection, leading to the rejection of only the weak reflection.

Large numbers of overlaps

If the integration output should happen to report a large number of overlaps, it is recommended that the user investigate the spots in the image for overlaps. If overlaps are not immediately visible, it might be that the overlap checker has been configured to be too sensitive. Consult the Overlaps table to determine the best value for the overlap parameter.

Background Determination

This section gives a brief description of how background is calculated in **dtintegrate** and of possible problems that might arise during the computation of the background.

Local background determination

It is generally acknowledged that using a local background near a spot is preferable to using backgrounds calculated far away from the spot. Also, because of changes in beam intensity or in detector plane fluctuations, it is best to accommodate the possibility that background might change for the same pixel on different images. Thus, the best algorithm calculates backgrounds on a per-spot basis, using pixels near the spot. Assuming that all pixels in a spot's bounding box are near, one can simply use pixels that are not on the spot (but in the bounding box) to calculate backgrounds. This is another reason why it is important to choose a sufficiently large spot multiplier (see section on Box Dimensions).

Fitting to planes

A two-step algorithm is used to compute backgrounds:

1. An average and standard deviation is computed for all pixels in the bounding box. These numbers implicitly define a set of 3 sigma pixels that are marked as being significant and are not used in the background determination.

2. All pixels adjacent to a significant pixel are flagged as border pixels. These pixels are also excluded from the background calculations.

Also a best fit plane is computed using all unflagged pixels in the bounding box. Deviations are computed from this plane to obtain a background variance to be used when calculating spot statistics.

Compute time required for background calculations

Background determination is the most time consuming process in **dtintegrate** requiring perhaps one third of the total execution time. Although computationally simple, the floating point calculations are time consuming and must be determined for each spot. The algorithms have been designed to reject any pixels that might affect the accuracy of the background.

Failures to find background

Most failures to find a background are reported when a spot butts up against the image edges or when the bounding box size has been fixed to a number that is too small. In **dtintegrate**, it is required that at least 10% of the pixels in each bounding box contribute to the background. Thus, background errors are reported when a very large spot occupies a very small box. Also, noisy images tend to produce faulty backgrounds because the noise is interpreted as being significant and, therefore, is not used in the background.

Disk and Memory Requirements

As **dtintegrate** processes the input data, it is doing three different tasks, each requiring memory and disk space:

1. Read the input images one at a time.
2. Copy pixel values from the image into a 3D array for each Bragg reflection
3. Integrate and profit profile fit the reflections.

Of these three tasks, the first one needs very little memory, but the second and third use quite a bit of memory. Fortunately, **dtintegrate** offers ways of minimizing the memory usage or the disk usage, depending upon which is the limited commodity (see sections below on How to save memory and How to save disk space).

Memory for 3D reflection integration

By far, most memory required by **dtintegrate** is used to store the pixel values around the predicted position of the Bragg reflection. Thus, all of the memory for reflections (including bounding boxes) must be queued in memory. This means that the amount of memory required by **dtintegrate** is a function of the average number of pixels in each reflection and of the average number of reflections per image. Images with many reflections and a high mosaicity require the most memory, whereas images with only a few reflections require less. Likewise, larger unit cells require more memory.

Memory for profile fitting

Profile fitting can also require a lot of memory. Most of this memory is used to load in and build reflection profiles, which must apply to overlapping images (see section on Profile Fitting). Because profile fitting is performed at the end of integration from data written periodically to scratch files during integration, the memory used can skyrocket until the profile fitting has terminated.

Abnormal termination from lack of memory or disk space

Integration tries to exit gracefully when the disk drive is full or when it fails to allocate memory. Nevertheless, many operating systems have difficulty recovering when all available disk space has been used up, and some either hang or terminate applications when all available memory is exhausted.

Profile Fitting

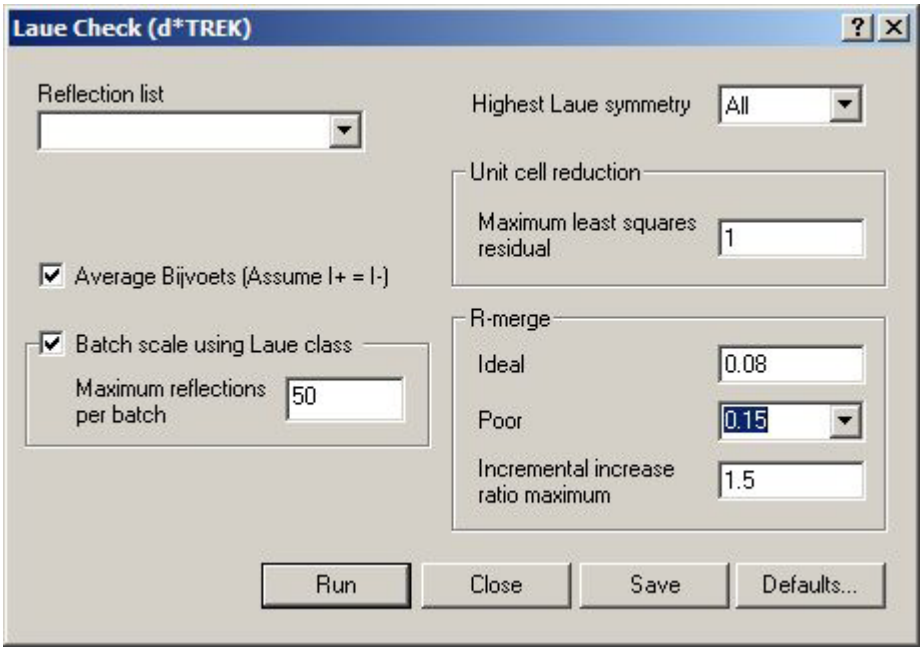
After integration, a post-processing algorithm is used to profile the shape of each reflection. A profile is a mathematical model of what a spot looks like on the detector and is a tool used to obtain better intensity estimates for weakly diffracting data. The basic principle that underlies profile fitting is that reflections that are near each other on the detector have similar spot shapes and similar intensity distribution within those spot boundaries as they pass through the diffract condition. Thus, a weaker reflection (one that diffracts less) should be a less intense copy of some nearby strongly diffracting reflection.

The assumptions of profile fitting are that reflection positions can be predicted accurately that accurate profiles can be obtained by averaging non-weak spot shapes and that the significant data in the weak spots cover several pixels, it follows that the estimated variance of a weak spot obtained from profile fitting is lower than the estimated variance obtained from summing up pixel counts.

Laue Check

The LaueCheck step analyzes the integrated data to determine the Laue symmetry for the crystal. The Laue symmetry determination is done by averaging the data for each Laue symmetry group, and then comparing the R_{merge}s to determine the correct Laue symmetry.

Laue Check Dialog

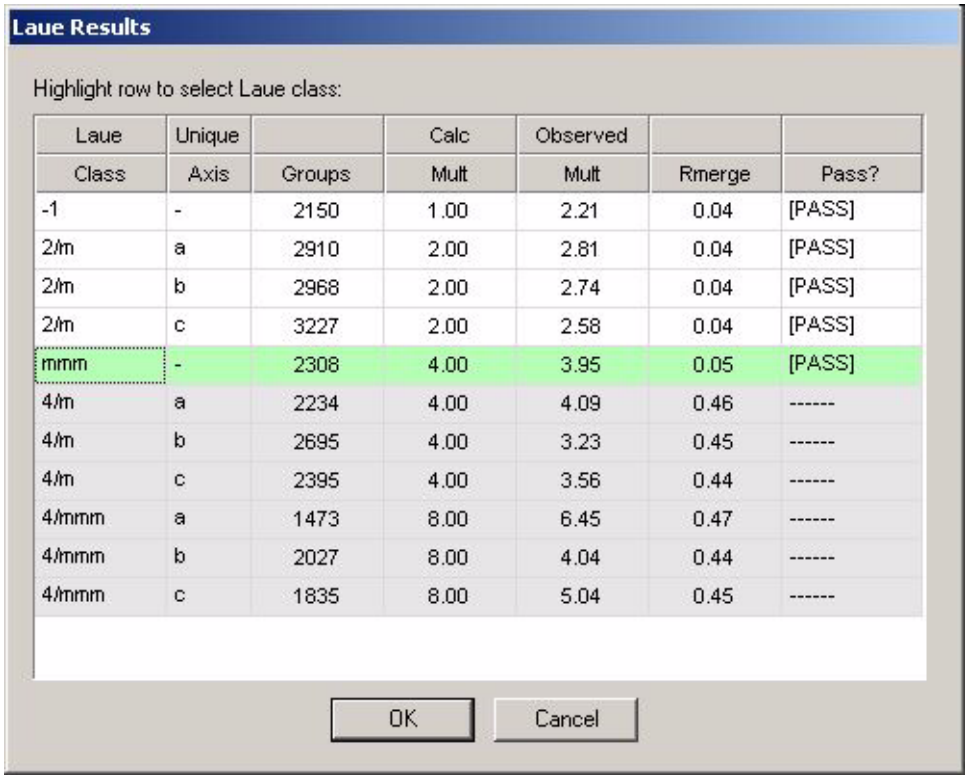


Parameter	Description
Reflection list	The normal reflection list to use is dtprofit.ref. But it is possible to use other reflection files.
Average Bijvoets	Select the **Average Bijvoets** option if you want to force the check to average I^+ and I^- in Bijvoet pairs. If this option is not selected and the crystal has a significant anomalous signal, the check may select the incorrect Laue symmetry.

Parameter	Description
Batch scale using Laue class	Select the **Batch scale using Laue class** option to enable batch scaling of reflection data during the check. If this option is selected, enter the number of reflections to use per batch in the **Maximum reflections per batch** field. If this option is not selected and there is a significant change in the scale factor for the data, resulting from decay or a poorly centered crystal, the check may select the incorrect Laue symmetry.
Highest Laue symmetry	This is a list of the Laue symmetry groups that are reported. You can set this to restrict the results to lower symmetry groups.
Unit cell reduction	In the **Maximum least-squares residual** field, enter the value for the largest least-square residual that a reduced cell is allowed to have for inclusion in the log file output.
R-merge	In the **R-merge** section, specify the values to use in the R_{merge} test during the check. In the **Ideal** field, enter the largest R_{merge} for which Laue symmetry is automatically included in the check. In the **Poor** field, enter the maximum R_{merge} for which a Laue symmetry is conditionally included in the check. All Laue symmetries with an R_{merge} greater than this value are always excluded from the check. In the **Incremental increase ratio maximum** field, enter the maximum ratio used for the conditional test. Laue symmetries with R_{merge} greater than the ideal, but less than the maximum, are included in the check only if the ratio of their R_{merge} to the R_{merge} for the next lower symmetry is less than the maximum ratio specified.

Laue Check Results Table

After you have run the Laue Check step, the following table is displayed. You should select the Laue class that you want to use and then click OK.

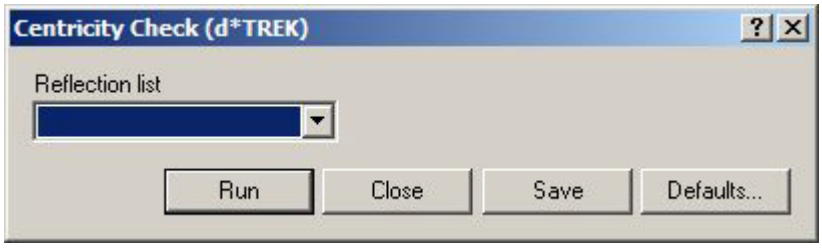


Laue Class	Unique Axis	Groups	Calc Mult	Observed Mult	Rmerge	Pass?
-1	-	2150	1.00	2.21	0.04	[PASS]
2/m	a	2910	2.00	2.81	0.04	[PASS]
2/m	b	2968	2.00	2.74	0.04	[PASS]
2/m	c	3227	2.00	2.58	0.04	[PASS]
mmm	-	2308	4.00	3.95	0.05	[PASS]
4/m	a	2234	4.00	4.09	0.46	------
4/m	b	2695	4.00	3.23	0.45	------
4/m	c	2395	4.00	3.56	0.44	------
4/mmm	a	1473	8.00	6.45	0.47	------
4/mmm	b	2027	8.00	4.04	0.44	------
4/mmm	c	1835	8.00	5.04	0.45	------

Centricity Check

After the Laue check you can then check the centricity. Macromolecular users may want to skip this step, since macromolecules always have an acentric space group.

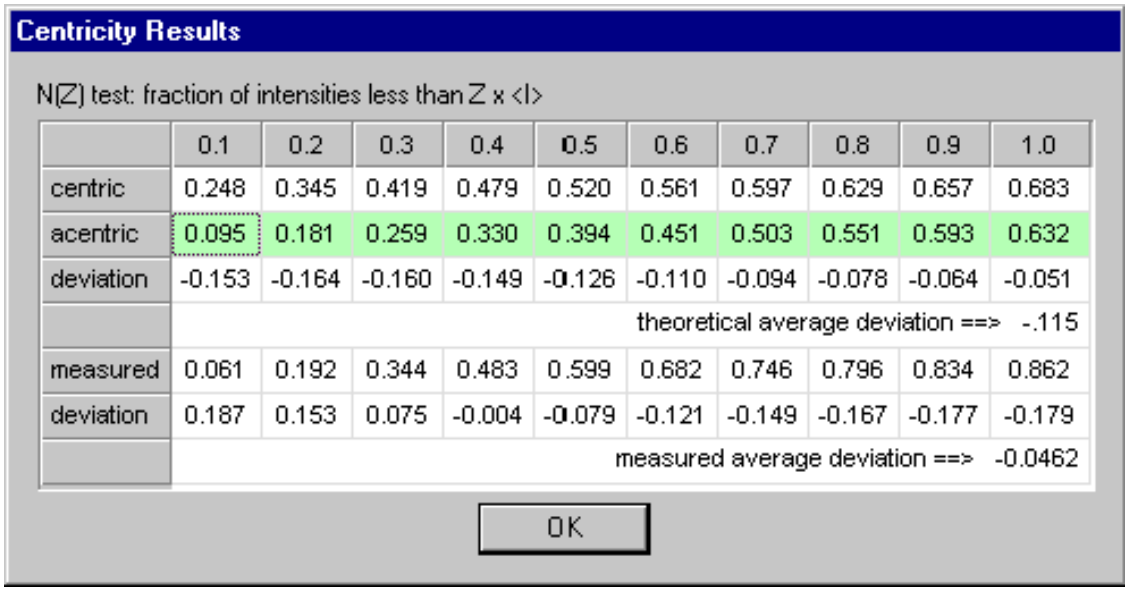
Centricity Check Dialog



Parameter	Description
Reflection list	The normal reflection list to use is dtprofit.ref. But it is possible to use other reflection files.

Centricity Check Results Table

Once you have run the **Centricity Check** step, the following table opens. Select centric or acentric and click **OK**.

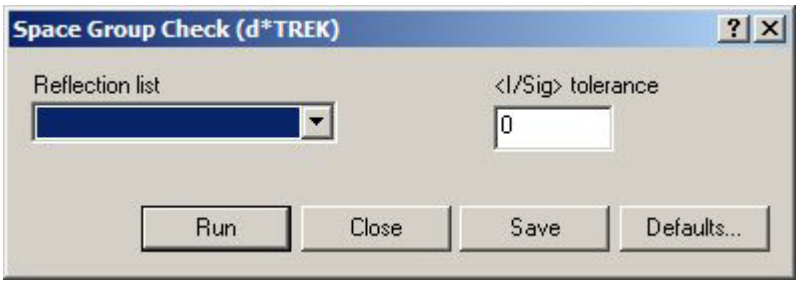


	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
centric	0.248	0.345	0.419	0.479	0.520	0.561	0.597	0.629	0.657	0.683
acentric	0.095	0.181	0.259	0.330	0.394	0.451	0.503	0.551	0.593	0.632
deviation	-0.153	-0.164	-0.160	-0.149	-0.126	-0.110	-0.094	-0.078	-0.064	-0.051
									theoretical average deviation ==>	-.115
measured	0.061	0.192	0.344	0.483	0.599	0.682	0.746	0.796	0.834	0.862
deviation	0.187	0.153	0.075	-0.004	-0.079	-0.121	-0.149	-0.167	-0.177	-0.179
									measured average deviation ==>	-0.0462

Space Group Check

The **Space Group Check** step analyzes the integrated data to determine the space group for the crystal. The reflections are examined to determine any systematic absences consistent with the Laue symmetry and centricity specified.

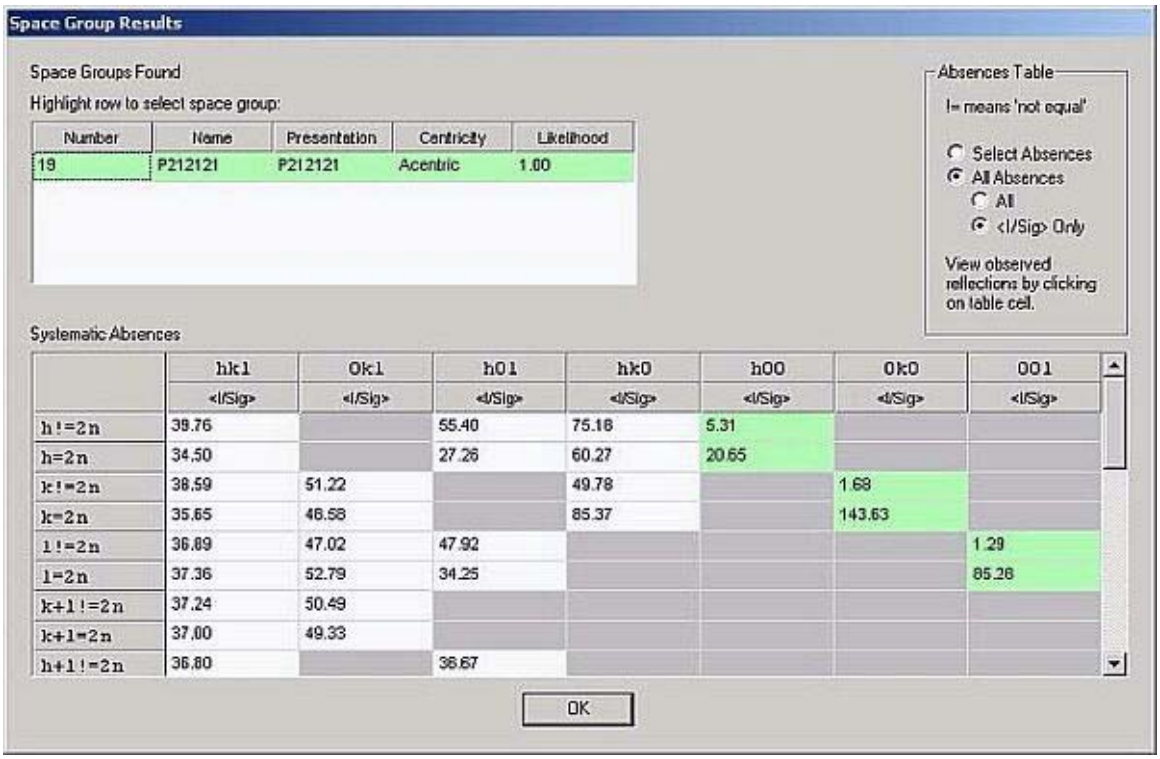
Space Group Check Dialog



Parameter	Description
Reflection list	The normal reflection list to use is dtprofit.ref. But it is possible to use other reflection files.
I/Sig tolerance	In the **I/Sig tolerance** field, enter the maximum I/σ(I) for a reflection to be considered absent. 0 means use the d*TREK default

Space Group Results Check Table

Once the **Space Group Check** step has run, the following table opens.

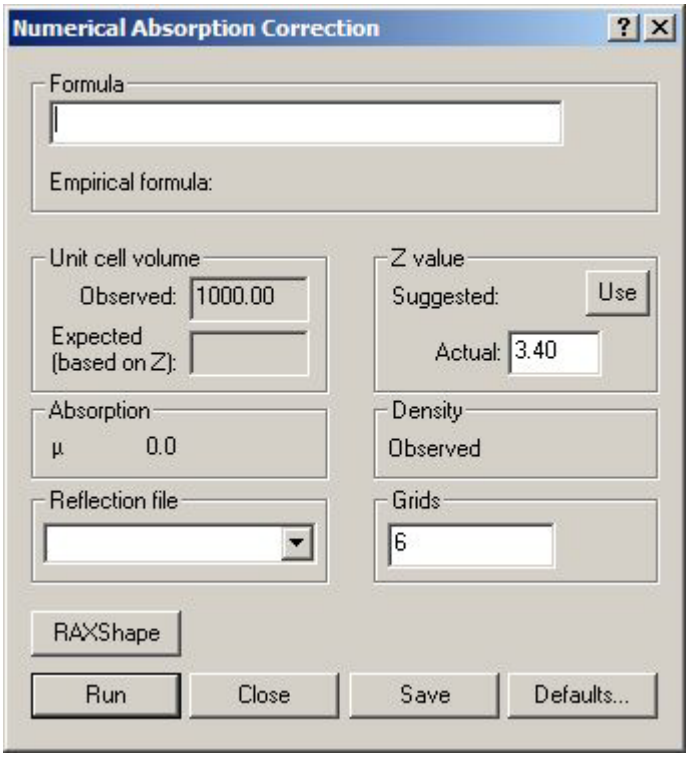


Double-clicking on any field displays outlier data supporting the absence condition. When the space group is not uniquely determined by systematic absences (either due to incomplete data or space groups having identical systematic absences), all possible space groups are displayed. The likelihood column relates the statistical representation of reported structures in the space groups (and should sum to 1).

Num. Abs. Correction

For small molecule work, if you know the formula and shape of your crystal, you can do numerical absorption correction. The normal way to determine the shape of your crystal is to run RAXShape. RAXShape requires a video camera and a compatible video capture card.

Numerical Absorption Correction Dialog



Parameter	Description
Formula	If you entered the molecular formula during Setup, it shows up here. If you did not or you need to modify it, you can enter the formula at this time. CrystalClear accepts the formula in a variety of formats.
Unit cell volume	This is just user information provided to make sure your formula is compatible. • **Observed**. The volume calculated from the unit cell obtained during indexing. • **Expected (based on Z)**. This is the estimated volume estimated based entirely on the formula (and Z value)
Z Value	The Z value (number of molecules in the unit cell) based on the formula is reported here. • **Suggested** The Z value based on the formula is reported here. If this is the value you want click **Use**. • **Actual** This is the actual Z value that is used. If you click **Use** the **Suggested** value updates the **Actual** field. Or you can just type in the value you want to use.
Absorption	This is the calculated absorption in cm^{-1}.
Density	Density as calculated using the molecular formula, Z value, and observed unit cell volume.
Reflection file	This allows you select the reflection file to use for numerical absorption correction. The default file is dtprofit.ref.

Parameter	Description
Grids	The numerical absorption uses a gridding method. By increasing the number of grids, the computation is made slower, but more accurate.
RAXShape	This starts up the RAXShape program that you can use to determine the shape of the crystal. In order for RAXShape to run you must have a video camera with a capture card and have RAXVideo installed.

Running Numerical Absorption Correction

The Run button starts the process; each reflection in the input reflection file is read and the absorption correction calculated based on the integrated path length of an incident and reflected beam for each grid point. The output reflection file is dtnumabs.ref; to use the results of the numerical absorption correction, be sure to use this new reflection file as the input for the next step.

Scale and Absorption

This is the small molecule version of the Scale and average step. The only difference is that this version does not allow the user to change the name and format of the output reflection file. See the **Scale and Average** description in the next section for details.

Scale and Average

The **Scale and Average** step:

1. Calculates and applies scale factors to different batches of reflections in a list of raw integrated data
2. Averages symmetry-equivalent reflections
3. Calculates merging and completeness statistics
4. Creates a list of unique reflections

There are three basic reasons why raw integrated data should be scaled:

1. Correction of systematic errors
2. Rejection of outliers
3. Validation of the error model, or σs

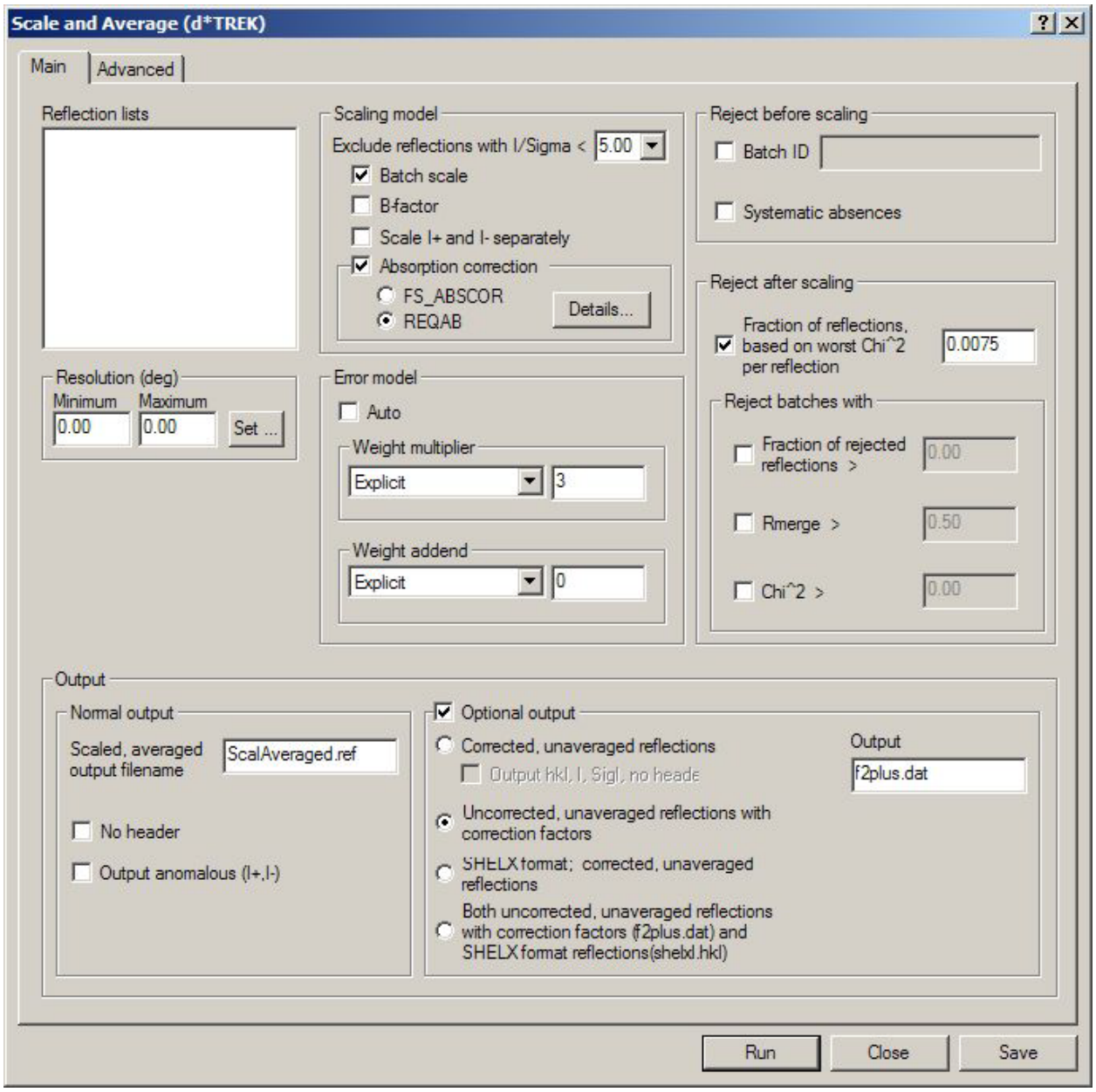
Corrections for systematic errors generally fall into one of three categories:

1. Absorption correction – errors caused by the physical geometry of the crystal
2. Decay correction – errors caused by prolonged exposure to X-rays
3. Batch scaling – errors caused by effects not related to the other two types

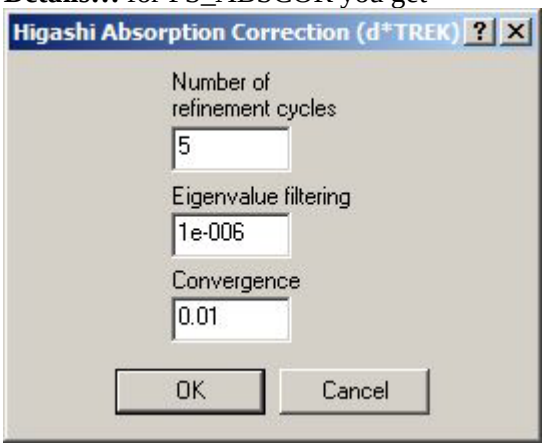
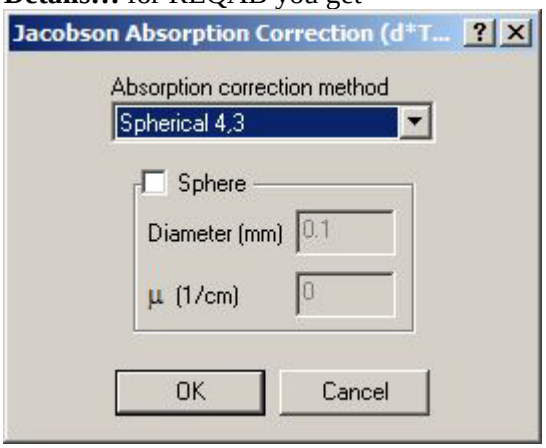
The **Scale and Average** step contains algorithms for addressing each of these types of systematic errors. The basic idea of outlier rejection is to reject measurements that differ significantly from the average obtained from redundant measurements. By removing outliers from the data, the observed reflections should more closely match the true diffraction from the crystal.

Scaling uses an error model to adjust the σs of the raw integrated data, so that they match the observed deviations among theoretically equivalent measurement. In a valid model, the variance calculated for each reflection matches the observed variance obtained from multiple measurements of the reflection. In this case, the reduced chi squared ($|\chi^2|$) statistic has an expected value of 1.0. Because systematic errors affect the calculated σs, correction parameters E_{add} and E_{mul} are required to scale the calculated σs to obtain $|\chi^2| = 1.0$. Determining the correct error model is necessary to ensure that the data is properly averaged *and* properly weighted during scaling.

Scale and Average Main Tab



Parameter	Description
Reflection lists	This box contains all the .ref files in the sample. The last .ref files written (e.g., dtprofit.ref) are the default.
Resolution	You can use the standard resolution controls to limit the resolution for this operation.
Scaling Model	This section provides you with a variety of parameters to control the scaling and absorption correction. • **Exclude reflections with I/Sigma .** Enter the I/σ(I) value to be used for excluding reflections from being used in scale factor determination. Note, however, that these reflections are included in the final statistics tabulated after averaging. • **Batch scale.** You can scale on a variety of properties. Batch scale is

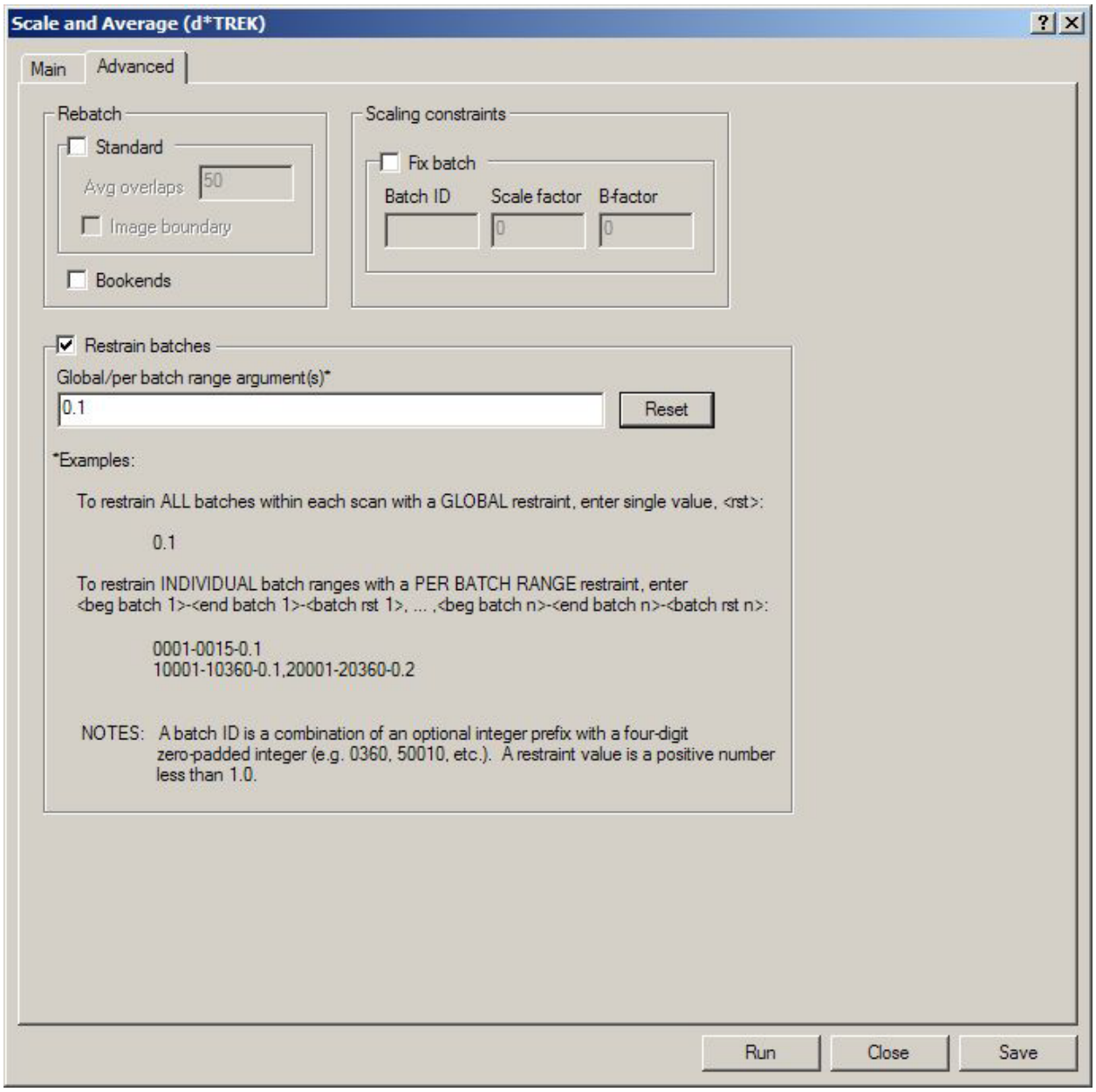
Parameter	Description
	defaulted to on. • **B-factor**. Select the **B-factor** the option if you want to enable B factor, or decay correction, scaling. This should generally only be used if your data was collected at room temperature, or your shows signs of decay. • **Scale |+ and |- separately**. Select the **Scale I+ and I- separately** option if you do not want to average I^+ and I^- before scaling. Selecting this option also affects any absorption correction that is being applied. • **Absorption Correction**. If this is checked, one of two absorption correction programs is run. o **FS_ABSCOR.** This works well for small molecule data, but is quite slow for macromolecule. o **Details…** for FS_ABSCOR you get  o ▪ **Number of refinement cycles**. The default is 5. ▪ **Eigenvalue filtering**. Threshold of small eigenvalues to be filtered. Any eigenvalue less than this is filtered. Default value is $1x10^{-6}$. ▪ **Convergence**. For value *t*, when $\|\Delta p\| < t.\sigma(p)$ for all parameters, the program assumes that the least-squares calculation has converged. Default value is 0.05. o For details, see the following references: D. Stuart & N. Walker (1979). Acta Cryst. A35, 925. N. Walker & D. Stuart (1983), Acta Cryst. A39, 158. o **REQAB.** Recommended for macromolecules. o **Details…** for REQAB you get  o ▪ **Absorption correction method**. Several alternative

Parameter	Description
	methods are listed in this drop down. But Spherical 4,3 is recommended nearly all the time. ▪ **Sphere**. For the Alternate Spherical method this specifies the **Diameter** and absorption coefficient, μ, of the sphere completely enclosed by the crystal volume.
Error Model	Check the **Auto** box if you want to d*TREK to automatically determine the error model. If **Auto** is not checked, you must select the **Weight multiplier** and **Weight addend**. The error model adjusts the input sigmas. For a valid error model, the variance calculated for each reflection should match the observed variance obtained from multiple measurements of the reflection. When this holds, a reduced chi squared ($|\chi^2|$) statistic has an expected value of 1.0. Since calculated sigmas are affected by detector gain, correction parameters Eadd and Emul are required to scale the calculated sigmas to obtain $|\chi^2|$ == 1.0. The parameters Eadd and Emul can be set manually, or determined automatically. In the dropdown list for the **Weight multiplier** field, select the option to use for the error model multiplier, E_{mul}. Some options require you to set an additional parameter in the field next to the dropdown list. The options for the multiplier are: Auto – intelligent: E_{mul} is calculated automatically Auto – redundancy: E_{mul} is calculated automatically, using reflections with a minimum redundancy. You must specify the redundancy to use. Auto – fraction: E_{mul} is calculated automatically, using a fraction of reflections. You must specify the fraction of reflections to use. Explicit: E_{mul} is not calculated, but must be specified explicitly. Typical values range from 1.5 to 7 and relate to the gain of the detector. In the dropdown list for the **Weight addend** field, select the option to use for the error model addend, E_{add}. Some options require you to set an additional parameter in the field next to the dropdown list. The options for the addend are: Auto – intelligent: E_{add} is calculated automatically Auto – redundancy: E_{add} is calculated automatically, using reflections with a minimum redundancy. You must specify the redundancy to use. Auto – fraction: E_{add} is calculated automatically, using a fraction of reflection. You must specify the fraction of reflections to use. Explicit: E_{add} is not calculated, but must be specified explicitly. Typical values start with the R_{merge} of the data.
Reject before scaling	If you check Batch, then in the **Batch IDs** field enter the batch IDs of the batches that you want to reject from scaling. Batch IDs must be entered as individual batches (*e.g.*, 101, 102, 103) or as a range or batches (*e.g.*, 101-103). You can combine individual batches and ranges by separating them with commas (,) (*e.g.*, 101,110-114,125). Batch numbers are listed in dtscaleaverage.log and usually have 5 or 6 digits. Select the **Systematic absences** option if you want to reject systematically

Parameter	Description
	absent reflections from scaling.
Reject after scaling	This specifies the criteria for rejecting reflection after they have been scaled. **Fraction of reflections based on worst Chi^2 per prediction.** Select this option if you want to reject reflections on the basis of their χ^2 value. In the field next to the option, enter the maximum fraction of reflections to be rejected because of their χ^2 value. Typical data uses 0.0075. When a crystal has evidence of pathological problems, consider increasing this value. Note: increasing this value with anomalous data may remove a weak anomalous signal. The following options reject entire batches. Select the **Fraction of rejected reflections >** option if you to reject batches based on the number of rejected reflections that they contain. In the field next to the option, enter the minimum number of rejected reflections value to use for rejection. Select the **Rmerge >** option if you want to reject batches on the basis of their R_{merge} values. In the field next to the option, enter the minimum R_{merge} value to use for rejection. Select the **Chi^2 >** option if you want to reject batches on the basis of their χ^2 values. In the field next to the option, enter the minimum χ^2 value to use for rejection
Normal Output	This section is not included in Small Molecule modes where these values are fixed. • **Scaled, averaged output file name**. The default is ScaleAverage.ref and some programs expect that. This reflection files lists h, k, l, average I and average SigmaI. • **No Header**. The header may cause problems for some programs, so you can eliminate it. • **Output anomalous**. This allows you to output the anomalous information which can be used for SAD phasing. This reflection files lists h, k, l, average I and average SigmaI, I+, SigmaI+, I-, and SigmaI-.
Optional Output	If you need output for other programs (e.g., structure solution for small molecule) you can also specify an additional output file. These are all unaveraged reflections in one of the following formats • Corrected, unaveraged reflections. • Uncorrected, unaveraged reflections with correction factors • SHELX format: corrected, unaveraged reflections • Both corrected, unaveraged reflections with corrections factors (f2plus.dat) and SHELX format reflections (shelxl.hkl) • Output. For the first two options, the default is f2plus.dat, and for the third option it is shelxl.hkl. For the Both option, you don't get to specify file names and both files are generated.

Scale and Average Advanced Tab

The **Advanced** tab offers a few more parameters which can control the **Scale and Averag**e process.



Parameter	Description
Rebatch	Select the **Standard** option if you want the scaling to automatically rebatch scans that do not have a sufficient number of overlaps for scaling. If you select this option, you must also enter the average number of overlaps required for rebatching in the **Average overlaps** field. Rebatching groups batches together so that the specified number of average overlaps occurs between batches. If you select the **Image boundary** option, the batches are created so that they lie on image boundaries. Select the **Bookends** option if you want the scaling automatically rebatch scans that do not have a sufficient number of overlaps for scaling, constraining the rebatching to only use those batches that have a size of less than half the average batch size.
Scaling Constraints	If you check **Fix batch** then you can specify a batch number and a **Scale**

Parameter	Description
	factor and **B-factor** for that batch.
Restrain batches	Select the **Restrain batches** option if you restrain the scale factors. If you select this option, you must also specify the weights to use for the restraint using the syntax explained in the dialog. Restraining the scale factors reduces the magnitude of their variation, but does not keep them from varying. Note that batches are not be restrained across scans.

Once scaling and averaging has finished, you should examine the log file output to determine if any problems occurred.

Improving Scaling and Averaging

Batch scale factors should vary smoothly within a scan. You may want to reject batches that have unusually large or small scale factors, high $|\chi^2|$, high R_{merge}, or a large number of rejected reflections. Batches are identified by a batch ID number, which is usually a number with four or more digits (*e.g.*, 0001, 20123). The last four digits of the batch ID are usually the image sequence number. The preceding digits are the batch prefix from integration. Rerun **Scale and Average**, listing the batch IDs to be rejected in the **Batch IDs** field.

Sometimes the first or last scaling batch does not have enough overlaps during scaling, causing it not to scale well. In this case, either reject those batches or turn on rebatching (using the **Rebatch** or **Bookends** options).

Reducing the resolution cutoff for the data may also improve scaling. The data are automatically divided into 10 resolution shells. If many of your resolution shells have weak data, this may adversely affect scaling. Check the $|\chi^2|$ values for the resolution shells to make sure that they are near 1, otherwise the $I/\sigma(I)$ values may not be reasonable. Cut the resolution off at the shell where the unaveraged $I/\sigma(I)$ drops to 2. This is usually the point at which the R_{merge} for the shell is between 0.30 and 0.40. Rerun **Scale and Average**, setting the resolution cutoff in the **Resolution** section and setting the **Max** value to be the maximum resolution of the shell selected.

If you have a low redundancy in the data set, or all of the data are very weak, you may want to consider turning off all scaling. You may also want to consider turning off scaling if you have an unusually high or low R_{merge}. In the former case, the existence of a few bad reflections may cause an invalid correction to be applied (potentially lowering the R_{merge}). In the latter case, it may not be beneficial to apply empirical corrections, as they may increase the R_{merge}.

If the entire crystal is bathed in the X-ray beam, you may not want to use batch scale factors. This assumes that you have only used one crystal throughout the data collection and that the exposure time was constant for the entire data collection.

Depending on the redundancy of the data, valid $|\chi^2|$ values might lie between 0.8 and 1.8. For data sets with an average redundancy of less than 3, it is probably not worth trying to adjust the error model so that $|\chi^2|$ is within a small tolerance of 1.0.

If the crystal was only rotated though a small angular region during collection, you may want to turn off absorption correction, as there may not be enough of the absorption surface present in the data to accurately determine the absorption surface.

If you are scaling data from more than one crystal, do an independent absorption correction on each crystal separately, and then use batch scaling only on the combined data.

If the data contains only random errors and the σs are correct, $|\chi^2|$ is 1.0. However, if the σs are correct but $|\chi^2|$ is larger than 1, then this suggests the presence of systematic errors in the measurements.

Additional tips on scaling and averaging, as well as in-depth descriptions of the concept used in scaling can be found in the d*TREK documentation.

REQAB Issues

The REQAB option for absorption correction provides a number of parameters for the modeling of absorption, crystal decay and incident beam intensity drift. These are all modeled within the absorption correction algorithms provided in REQAB.

The goal of the absorption correction algorithm is to deduce an absorption surface associated with a crystal by requiring that this surface minimize the difference in intensities between symmetry related reflections. In practice, this is done by minimizing the weighted differences between the right and left-hand sides of the equation:

(1)

$$I_i A_i = \frac{\sum_{j=1}^{k} w_j I_j A_j^0}{\sum_{j}^{k} w_j}$$

where *I*'s are the intensities and *A*'s are the absorbencies. The right hand side represents a sum over the *k* symmetry related intensities in a group of equivalent reflections, and the subscript i represents any member in the group. One seeks to find an absorbance A_i to minimize this difference and A_j^0 represents an initial estimate of the absorbance. As implemented in the program the initial A^0 values correspond to that of the spherical crystal; when a new absorption surface is obtained, these replace the original A^0 values and the process is repeated until convergence is obtained.

The absorbance *A* consists of a product of the absorbance of the primary beam and the absorbance of the scattered beam, i.e.

(2)

$$A = e^{-\mu t} = e^{-\upsilon(t_p + t_s)} = A_p \cdot A_s$$

These absorbencies could also be expanded in a Taylor series,

(3)

$$A = A^o + \sum_j \frac{\partial A}{\partial p_j} \Delta p_j + \text{higher order terms}$$

If the usual approximation is made of assuming that the higher order contributions are negligible, then by a least squares approach, the Δp_j values can be calculated and the whole process repeated until convergence is obtained. We assume the absorption surface is smoothly varying in reciprocal space. In order to enforce this powerful constraint two sets of analytical functions are used.

Spherical harmonics are one convenient set of analytic functions for *As*. The maximum order is limited to 8, four even term and four odd terms:

(4)

$$A(\phi, \upsilon) = a_0 + \sum_{l=1}^{l \le 8} \sum_{m=0}^{l} a_{lm} y_{lm}(\phi, \upsilon)$$

where a_{lm} represents the coefficients to be determined by refinement and y_{lm} are the corresponding spherical harmonics, and φ and υ are the equatorial and azimuthal angles of the diffracted x-ray beam, respectively.

An alternate analytic function employs a Fourier series to represent absorbance:
(5)

$$A(\phi_p, \phi_s, \upsilon_p, \upsilon_s = Q_0 + \sum_{n}^{n\,max} \sum_{m}^{m\,max} P_{nm}(\sin(n\phi_p + m\upsilon_p) + \sin(n\phi_s + m\upsilon_s)) + \\ + Q_{nm}(\cos(n\phi_p + m\upsilon_p) + \cos(n\phi_s + m\upsilon_s))$$

Here the coefficients P_{nm} and Q_{nm} are fit via a least squares procedure. The maximum values of n and m are 8 and 4, respectively. In the case of the Fourier method, both the primary (or incident) and scattered beams are modeled.

In addition to absorption, the apparent intensity of the primary or incident beam may be affected by decay, beam in homogeneity and source instability. *CrystalClear* provides two methods for modeling these effects, simple batch scaling, which is applicable to either the spherical harmonics and Fourier methods, and circular harmonics, which applicable to only with only the spherical harmonics.

In the batch scaling method the data are first scanned to find the batch with the greatest overlap. This batch becomes the reference batch and the absorbencies described in Equation 3 are allowed to vary with respect to the reference batch. That is, the derivatives of A_{pj} are zero unless j corresponds to a batch and the reference batch.

Alternatively, if the azimuthal angle in equation 4 is fixed at 0, the spherical harmonics collapse into the equatorial plane becoming circular harmonics and the resulting function $A(\phi,0)$ can be used to model incident beam absorption.

The options are found in the REQAB Dialog and are described in below**.** The Spherical options seem to be best for small molecule data, but the Fourier method is often the best method for macromolecule data. But if one method does not provide the results you were expecting try one of the other methods.

Absorption options

Option	Description
Spherical 4,3	This option uses 4th order even and odd spherical harmonics to describe the diffracted beam absorption and 3rd order even and odd circular harmonics to model incident beam absorption.
Spherical 3,2	This option uses 3rd order even and odd spherical harmonics to describe the diffracted beam absorption and 2nd order even and odd circular harmonics to model incident beam absorption
Fourier 8,3	This option uses Fourier coefficients to model diffracted beam absorption and simple batch scaling to model incident beam absorption, decay, etc.
Alternate Spherical	In this method the primary absorption surface is calculated using the ideal sphere then one of the other methods is used perturb the ideal correction. Furthermore this method puts the resultant transmission factors on an absolute scale.

Fitting the absorption surface using spherical harmonic coefficients or Fourier coefficients via singular value decomposition is very robust and, in most cases, leads to a satisfactory result. However, there are some cases in which error messages are displayed.

Absorption correction trouble shooting guide

Error Message	Cause and remedy
Number of outliers exceeds 2000	The assumed symmetry is too high. Use **Data analysis -> Laue Group** to automatically determine the Laue group and rerun. The sample is a strong absorber, μR > 2. In the Advanced Menu set outer rejection criterion to 500 or 1000. This ensures that reflections that are strongly affected by absorption are used in the calculation of the surface, improving the overall correction. The crystal is split and some reflections overlap and some do not. Try to find a better crystal.
No convergence in SVDCMP	The redundancy is low. If the sample is triclinic try to collect at least one scan of 180°. If the sample is of higher symmetry collect more data in a different region of ϕ or χ. The diffraction data are poor. The algorithm uses significant data to calculate the absorption surface reliably and there are not enough to do this. Recollect the data with longer exposure times.
Spherical harmonics methods do not complete S Del2 > 10^6**	Multiple scans have different exposure times. You may be able to recover by repeated runs of "Fourier". The crystal died or fell off, the generator went down, etc. Inspect your images to determine if diffraction stopped at some definite point in time and reintegrate up to that point. The assumed symmetry is too high. Change the putative space group.
Fourier methods do not complete. S Del2 > 10^6**	Multiple scans have different exposure times. Recovery unlikely. The crystal died or fell off, the generator went down, etc. Inspect your images to determine if diffraction stopped at some definite point in time and reintegrate up to that point. The assumed symmetry is too high. Change the putative space group.

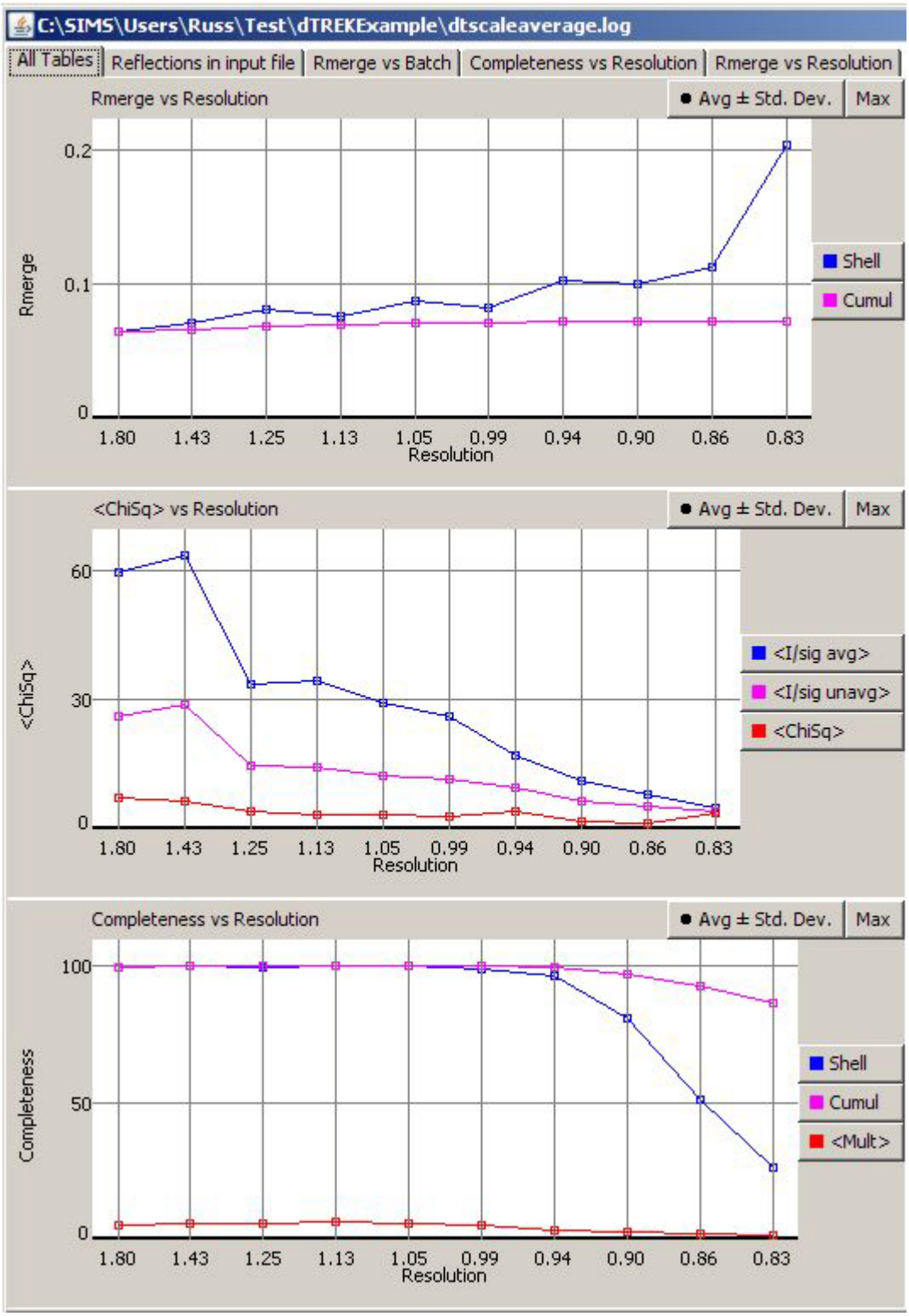
JDtplot for Displaying Scale and Average Results

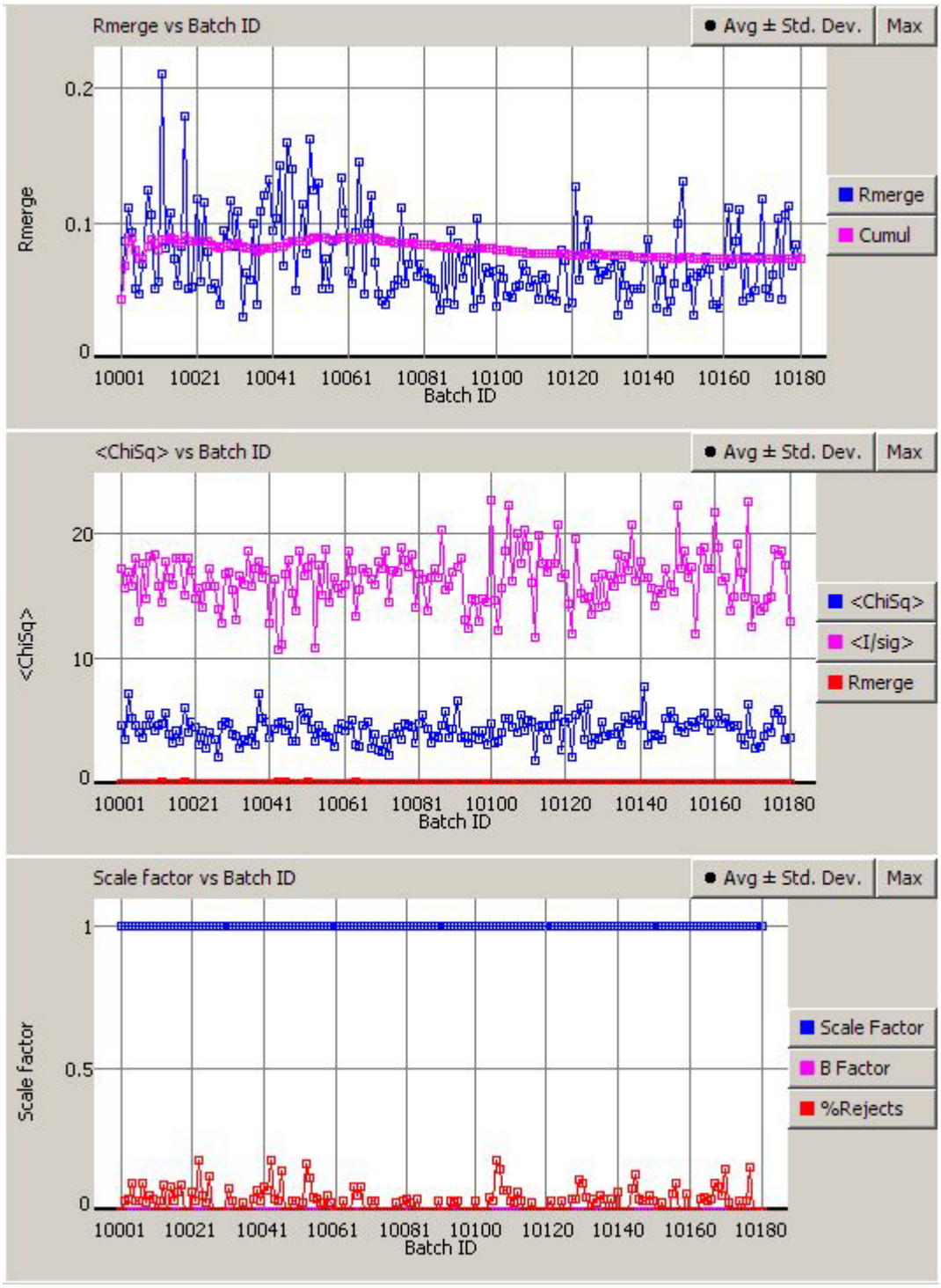
JDtplot is an auxiliary program for plotting the results of **Scale and Average** results. At the end of **Scale and Average** this program is automatically started if you have selected JDTPlot in the User Preferences (which is the default). You have the option to be prompted first that you can set in Preferences (see chapter 2). The older CrystalClear plotting option is now obsolete. You can also run JDtplot through the menu under **Processing>Run JDtplot** or **View>>Scale/Average Plots>JDtplot**. However, you must have run **Scale and Average** before this runs successfully.

JDtplot is a Java program and requires a compatible JRE (Java Runtime Environment). Normally the JRE you have installed by default in Windows should work fine. If you need to load a different JRE, you must also edit CrystalClear.configuration to specify the path to this JRE.

JDtplot looks at the log file from **Scale and Average** and creates various plots. The figures below split the two columns that make up the normal display in order to fit them better on the page.

JDtplot Display


C:\SIMS\Users\Russ\Test\dTREKExample\dtscaleaverage.log
All Tables
Reflections in input file
Rmerge vs Batch
Completeness vs Resolution
Rmerge vs Resolution
Rmerge vs Resolution
Avg ± Std. Dev.
Max
Rmerge
0.2
0.1
0
1.80 1.43 1.25 1.13 1.05 0.99 0.94 0.90 0.86 0.83
Resolution
Shell
Cumul
<ChiSq> vs Resolution
Avg ± Std. Dev.
Max
<ChiSq>
60
30
0
1.80 1.43 1.25 1.13 1.05 0.99 0.94 0.90 0.86 0.83
Resolution
<I/sig avg>
<I/sig unavg>
<ChiSq>
Completeness vs Resolution
Avg ± Std. Dev.
Max
Completeness
100
50
0
1.80 1.43 1.25 1.13 1.05 0.99 0.94 0.90 0.86 0.83
Resolution
Shell
Cumul
<Mult>
Rmerge vs Batch
Completeness vs Resolution
Rmerge vs Resolution



Using JDtplot

The default display is for all 6 graphs to be displayed in the JDtplot main window. If you want to examine just one plot in detail you can either click **Max** for that graph, or click the tab for that graph. Once the large graph is displayed you can click **Min** or go back to the **All tables** tab to get back to the combined display.

When you mouse over the graphs, you get a display of the value under the mouse.

If you click **Avg +- Std. Dev**, a semi-transparent box pops up with the average and standard deviation for each of the properties that is plotted.

The color code for each property is shown with the property name along the right side of the graphs. If you click these buttons, the display of the property is toggled off or back on. The Y axis might be rescaled to best display the remaining properties.

Menu Operations

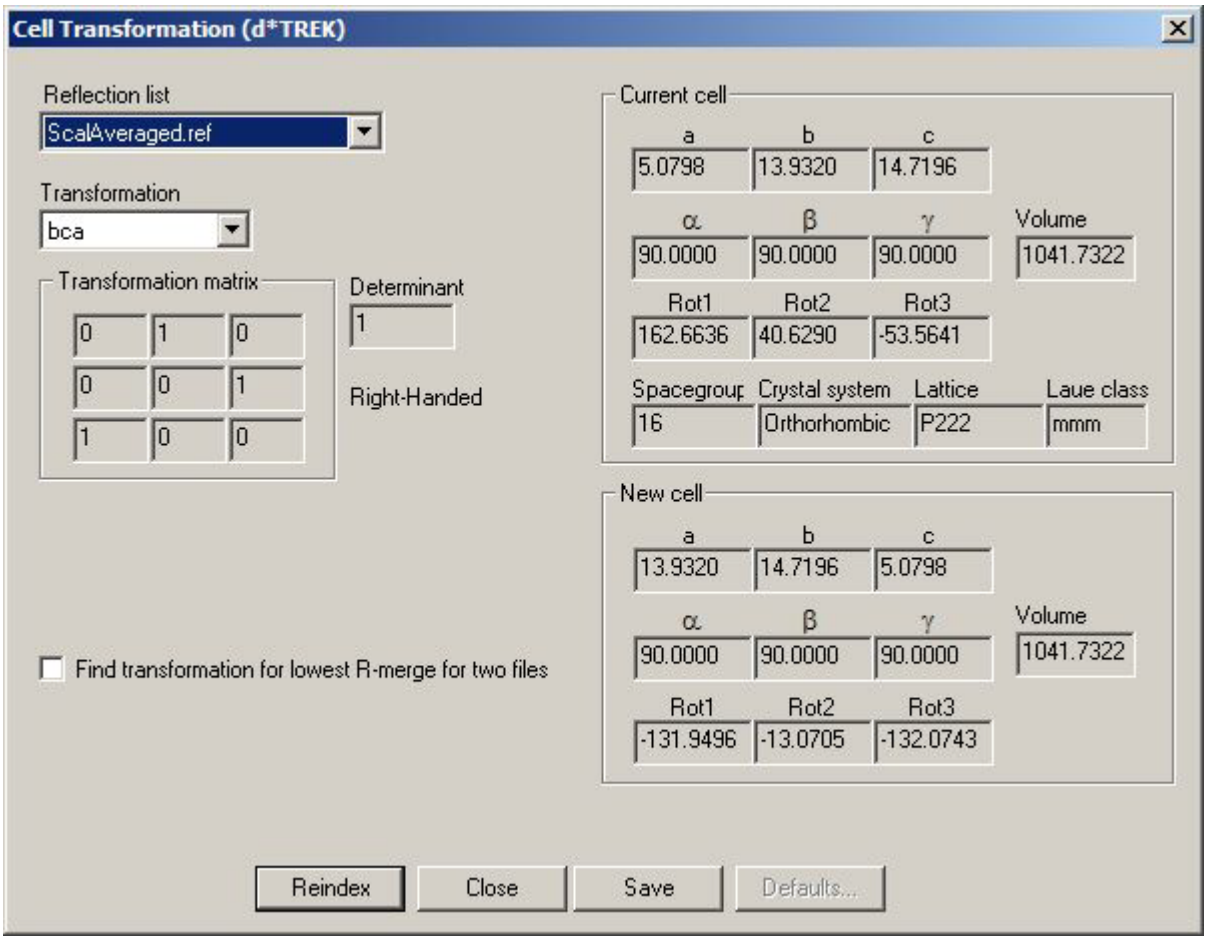
There several functions that are accessible through the **Processing** menu, but do not have explicit steps in flow bars. These include:

- **Cell Tools**
  - **Transform**
- **Reflection List**
  - **Merge Reflections**. This combines reflection lists in separate files into a single file.
  - **Reflection File Tools**. This provides various options for filtering a reflection list.
- **Rank Crystal**
- **Run d*TREK Interactive**

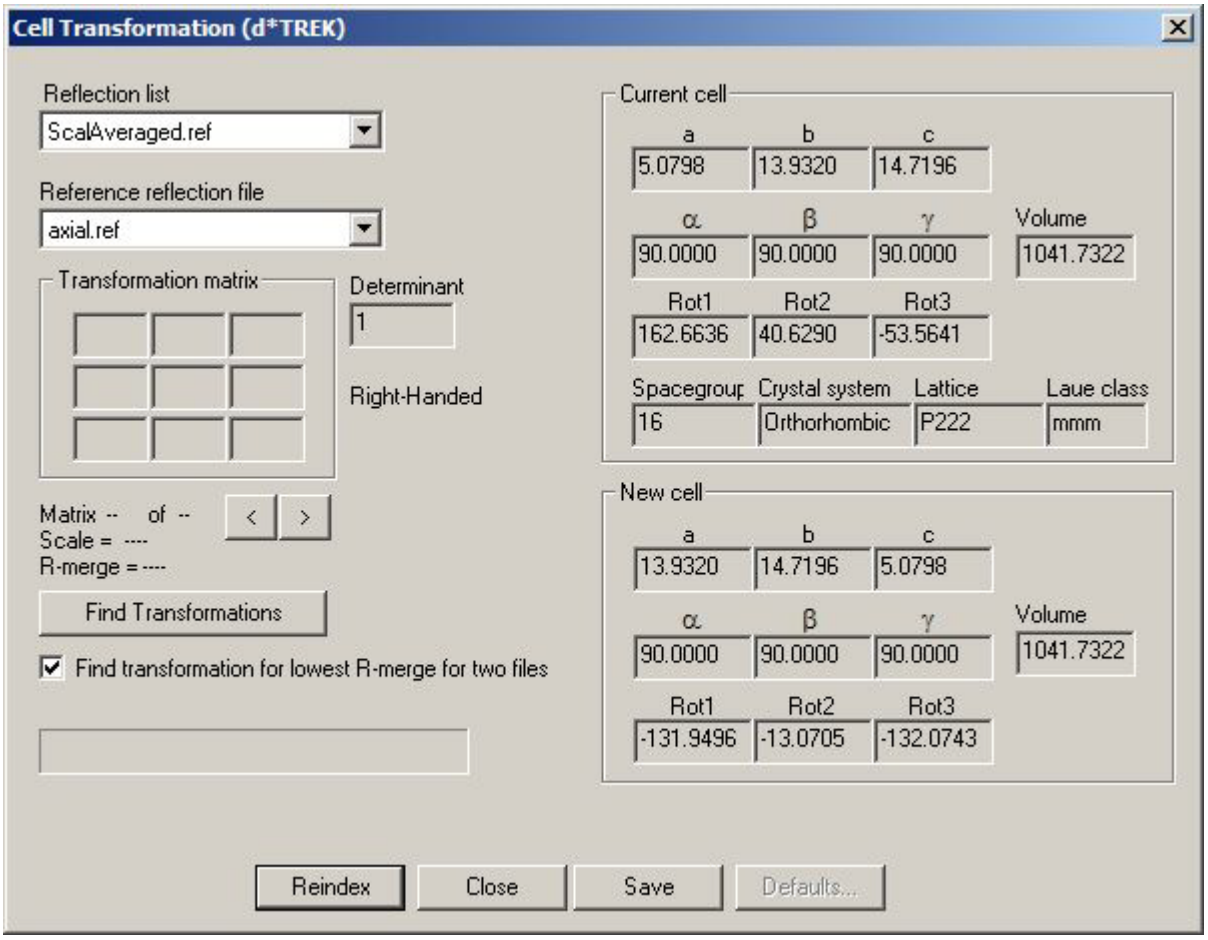
Cell Tools>Transform

This tool allows you to re-index a cell under a given transformation.

Cell Transformation Dialog



When **Find transformation for lowest R-merge for two files** is checked, there are additional fields.



Parameter	Description
Reflection lists	This drop down list contains all the .ref files in the sample. This is normally done after all the integration and scaling, so you should normally use ScalAveraged.ref.
Reference reflection file	This is displayed only when the **Find transformation…** box is checked. It specifies the file that is to be used to minimize the R-merge assuming that this file and some transformation of the Reflection file were to be merged.
Transformation	Select the transformation you want to perform. This control is not displayed if you click **Find transform for lowest R-merge for two files**.
Transformation matrix	This is a read only display of the transformation matrix unless you choose **Custom Matrix** as your **Transformation** option. The **Determinant** and Right-Handed or Left-Handed are displayed but not editable.
Find Transformations	Click this button to compute the transform that would result in the lowest R-merge for the **Reflection** and the **Reference reflection** files. The results are show above this button and you can use the <> buttons to examine the ranked solutions.
Find transformation for lowest R-merge for two files	This check box activates the **Reference reflection** file and **Find Transform** controls so that you can use these tools to determine the transform you want to use.
Current Cell	This is a read only display of the current cell.
New Cell	Unless you select **Custom Cell** as the option for the Transformation, this is just a read only display of the new cell. If you do select **Custom Cell**, you can enter the basic cell parameters.

Re-indexing Using a Standard Cell Transformation

The most basic kind of **Cell Transformations** involve permuting (flipping) axes, changing centering and changing monoclinic cell type. These operations are prevalent enough to have common names that should be recognizable by most crystallographers. The transformations provided are:

Label Description	Identity Transformation
CAB **BCA** **BAC** **ACB** **CBA**	Flips or permute cell lengths. For example, CAB takes (a,b,c) to (c,a,b).
mI -> mC **mC -> mI**	Moves between I centered and C centered monoclinic cells.
hR -> hP **hP -> hR**	Moves between primitive rhombohedral and triply-primitive cells.
hP -> oC (C1) **hP -> oC (C2)** **hP -> oC (C3)** **oC -> hP (C1)** **oC -> hP (C2)** **oC -> hP (C3)**	Moves between primitive hexagonal and C-centered orthohexagonal cells.

The standard transformations can be used to flip cell axes, move from primitive to centered cells (or vice versa), and re-index into different orientations.

- Select the desired transformation from the **Transformation** list. Be aware that not all transformations in the list are valid for every kind of lattice and centering. The special cells named **Custom Matrix** and **Custom Cell** are described in a separate section below.
- Once a transformation is selected, the matrix appears in the **Transformation Matrix** item and the real space cell that would result from applying this transformation should appear in the **New Cell** item box.
- Go to the **Reflection List** item and select a reflection list. This is optional, but must be specified if you wish to apply the transformation to previously collected data. Only one reflection file can be specified.
- Click **Re-index**.
- A message box inquires if the operation is satisfactory. Any reflections that map to a fractional HKL value (a real possibility when transforming from centered to uncentered cells) are displayed along with I/sig statistics for those reflections. To cancel the operation, click **No**. To proceed with the operation, click **Yes**.
- If you specified a reflection list to index, then you are prompted with the reflection list to use. Enter the name of the **Reflection List**.
- After the operation, the **Current Cell** and the **New Cell** match. The **Transformation** automatically resets itself to the identity transformation.

Re-indexing Using a Custom Cell Transformation

Uncommon **Cell Transformations** can be specified by providing an HKL transformation matrix. This matrix describes the transformation taking each HKL value to a new HKL value. Specify the 3x3 matrix [A] multiplying the column vector HKLOld from the left (i.e. HKLNew = [A] HKLOld).

Care should be taken when specifying transformation matrices. Be aware that left-handed transformations yield left-handed crystal orientation matrices, and those transformations that change volume can possibly corrupt reflection files. A custom **Cell Transformation** can be entered if none of the standard **Cell Transformations** are satisfactory. The procedure is similar to that used for standard **Cell Transformations**.

- Go to **Transformation** control group and select **Custom Matrix**.
- Enter the matrix elements. Allowable values consist of integers and small fractions.
- Go to the **Reflection List** and select a reflection list. This is optional, but must be specified if you wish to apply the transformation to previously collected data. Only one reflection file can be specified.
- Click **Re-index**.
- A message box inquires if the operation is satisfactory. Any reflections that map to a fractional HKL value (a real possibility when transforming from uncentered to centered cells) are displayed along with I/σ statistics for those reflections. To cancel the operation, click **No**. To proceed with the operation, click **Yes**.
- If you specified a reflection list to index, then you are prompted with the reflection list to use. Enter the name of the **Reflection List**.
- After the operation, the **Current Cell** and the **New Cell** should match. The Transformation automatically resets itself to the identity transformation.

Re-indexing Using a Specific Cell

This feature automatically finds the transformation matrix that would transform the one cell to another. This can be useful for changing axis lengths or getting the *CrystalClear* cell to match the cell from some other source.

NOTE: Use the **Cell Reduction** dialog if you only wish to change the cell orientation angles.

This is a very powerful option that allows you re-index to any district cell (within reason). Instead of entering in a literal HKL transformation matrix, you specify a cell, and the program figures out the matrix that would produce a close match to that cell.

- Go to **Transformation** and select **Custom Cell**. **New Cell** becomes active. Enter cell constants a, b, c, α, β or γ.
- Once the cell has been specified, click **Find Matrix**.
- A message queries to verify the operation. The proposed HKL transformation is specified along with the resulting cell and a residual error. At this time, you may cancel by clicking **Cancel,** otherwise click **Use Matrix.**
- If you click **Use Matrix** in (c), then **Transformation** changes to **Custom Matrix**, and **New Cell** changes to the new target cell. Proceed as if you entered the **Cell Transformation** yourself, and follow the steps above.

Re-indexing to Find the Lowest R-merge

NOTE: This is an advanced tool and should be used with caution.

This tool attempts to solve this ambiguity by finding the relative transformation matrix that would have to be applied to one data set to get a low R-merge.

This feature compares two data sets collected with possibly different orientation angles. When two data sets from a crystal with a polar Space Group are combined, this can help make sure they have the same polarity (ie. P2, P3, P6, P321 vs. P312). This might not be the case if the data sets were collected on two different machines, or if the orientation angles of one of the data sets are unknown.

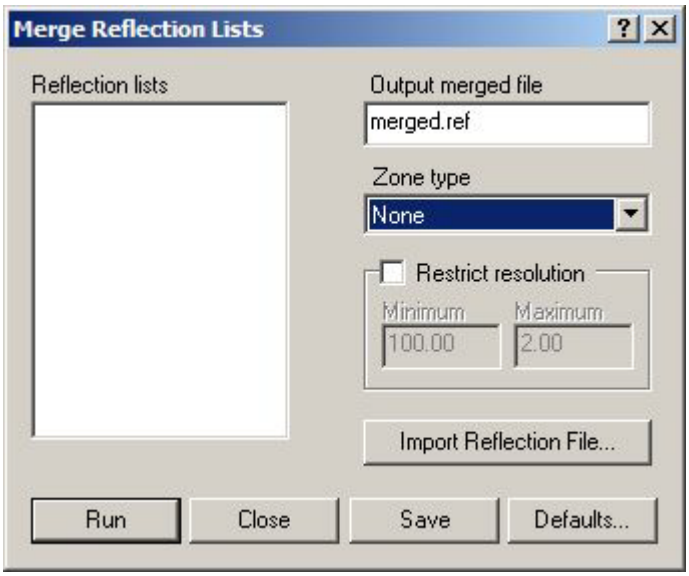
This feature can be used to find the best transformation between two data sets having an anomalous signal and different crystal orientation angles. It can also be used to scale data that are collected at different exposure times.

- Click **Find Transformation for Lowest R-merge for Two Files**. Several new dialog boxes appear when the box is checked.
- Select reflection files to compare. The **Reference** reflection file is not re-indexed and issued as the benchmark. The transformation resulting from this operation is applied to the other reflection list, and to the *CrystalClear* cell.
- Click **Find Transformations**. **NOTE**: This operation takes some time to complete.
- When the operation is done, use the Arrow keys to move between the different Cell Transformations. The R-merge and scale factor for each transformation is printed.
- Click **Re-index**.
- A message box inquires if the operation is satisfactory. Any reflections that map to a fractional HKL value (a real possibility when transforming from uncentered to centered cells) are displayed along with I/σ statistics for those reflections. To cancel the operation, click **No**. To proceed with the operation, click **Yes**.
- If you specified a reflection list to index, then you are prompted with the reflection list to use. Enter the name of the **Reflection List**.
- After the operation, the **Current Cell** and the **New Cell** should match. The **Transformation** automatically resets itself to the identity transformation.

Reflection List>Merge Reflections

If you have integrated reflections from separate scans into separate files or have multiple reflection files that need to be merged for any other reason, this step can be selected in the menu under **Processing>Reflection Lists>>Merge Reflections**….

Merge Reflections Dialog

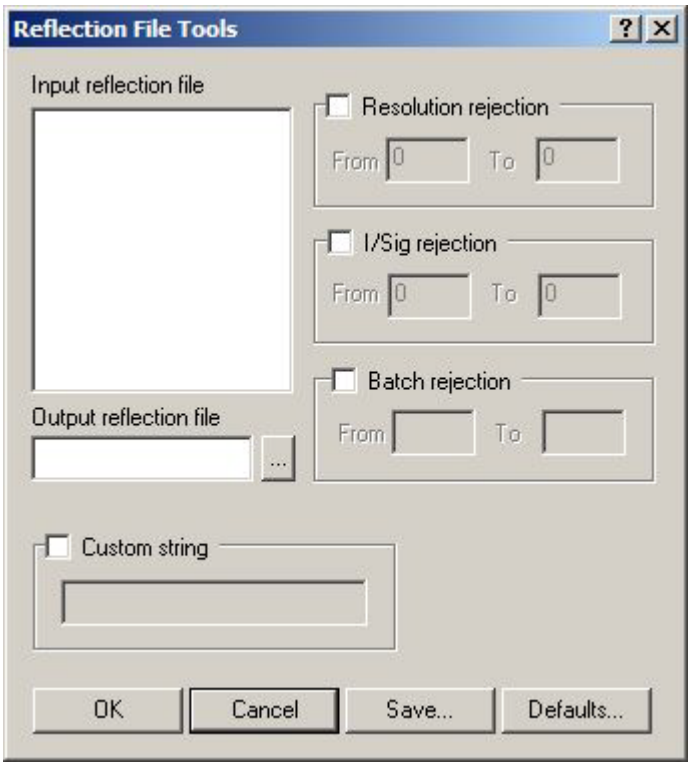


Parameter	Description
Reflection lists	These are the reflection lists that are currently in the sample directory. If you want to get reflection lists from another sample, use **Import Reflection File…** You can select multiple files from this list using standard Windows conventions.
Output merged file	You can give your merged file a name.
Zone type	If you want to restrict the reflections to certain zones of reciprocal space you can select the zone restriction from this drop down list.
Restrict Resolution	This does not use the global CrystalClear d*TREK resolution controls, but they are very similar and allow you set the **Minimum** and **Maximum** resolution.
Import Reflection File…	A file browser opens which allows you to import reflection files into the sample. Once they have been imported they show up in the Reflection lists.

Reflection Lists>Reflection Tools

This dialog allows you to specify various filters to be applied to a reflection file.

Reflection File Tools Dialog



Parameter	Description
Input reflection file	These are the reflection files that are currently in the sample directory. You can only select a single file in this list.
Output reflection file	You can specify a file name within the current sample, or click the … button to browse to another location to write the file outside the sample.
Resolution rejection	If you select this option, the reflections are filtered to lie between the **From** and

Parameter	Description
	To resolutions. 0 means no limit.
I/Sig rejection	This allows you to filter on I/Sigma. Again 0 means no limit
Batch rejection	If you need to remove one or more batches from the reflection file, you can check this box and provide the **From** and **To** bounds.
Custom string	This allows you to enter custom rejection criteria. See the d*TREK dtreflnmerge command for more details.

Ranking

The Ranking step allows you to determine a quantitative measure of the quality of a crystal sample. Ranking uses a series of pre-defined rules to determine the quality of a crystal. Depending on whether or not the rule's criteria are met, a number of bonus or penalty points are included in the calculation of the rank. The rules used in ranking are:

- Spot count in resolution shells
- I/σ(I) in resolution shells
- Spot sharpness
- Spots in strong ice rings
- Spots in diffuse ice rings
- Percentage of spots indexed
- RMS residuals after refinement
- Mosaicity
- Percentage of spots refined
- Spot count (predicted and observed) in resolution shells after refinement
- I/σ(I) (predicted and observed spots) in resolution shells after refinement
- Strategy total rotation width

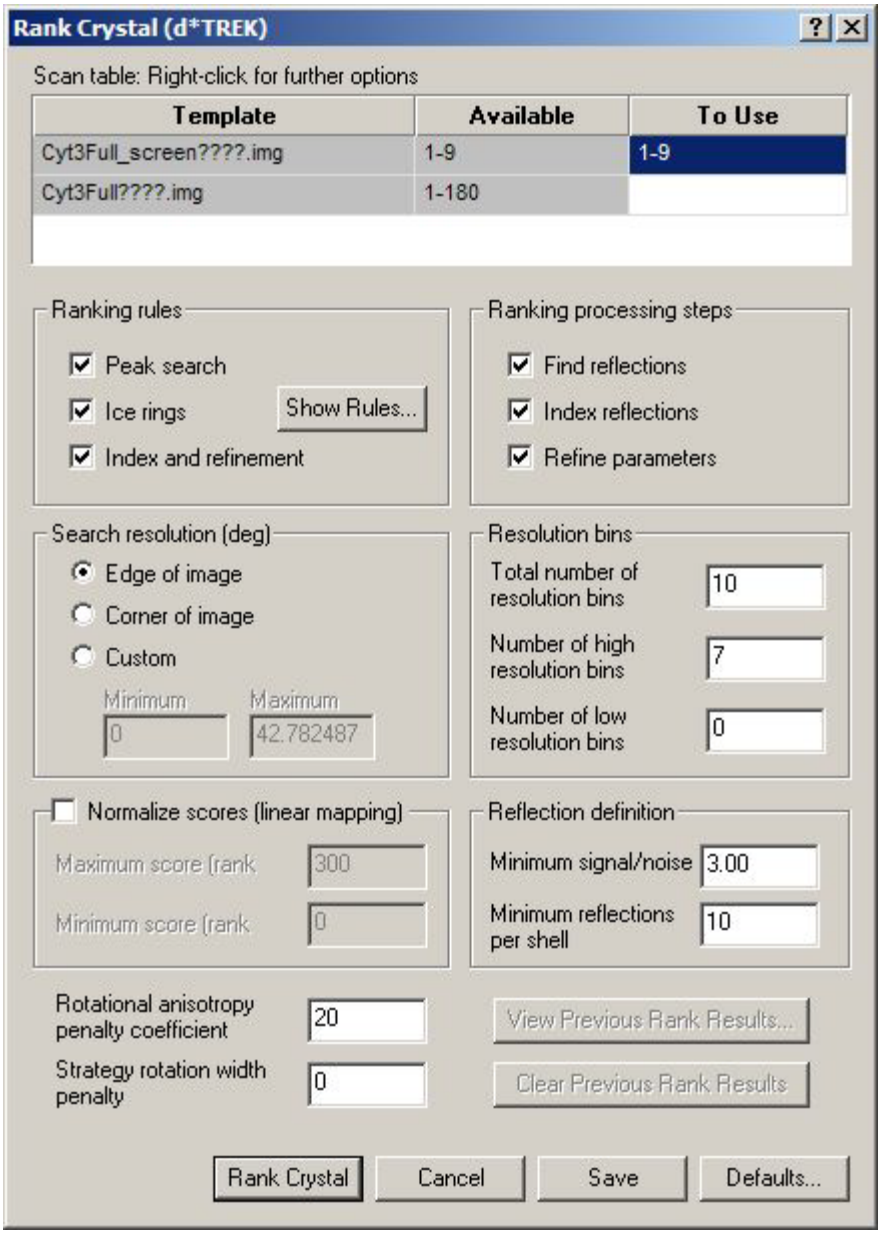
The rank calculated for a crystal can be used to evaluate samples of the same crystal type to determine which sample is likely give the best data set.

Ranking assigns a single integer rank to a crystal based on an analysis of the screen images. This is not normally very useful in manual work where you are sitting in front of CrystalClear. You can usually just look at your images and decide how good they are. But as soon as you automate the data collection process (e.g., by using Director with an ACTOR robot to drive CrystalClear), this becomes a vital step which allows the automatic determination of the best samples to collect and to prevents wasting time collecting samples that are unlikely to provide useful data.

Ranking is most useful when you are comparing similar crystals. The various ranking rules add both positive and negative points to the rank. So for comparison purposes it is best to make sure the same ranking rules are used for all the samples in the set. For example if you have three crystals that are same, you might want to screen each one and then rank them to decide the best one to use for data collection. This is especially useful if you are using Director on an ACTOR system.

Because Ranking is not used in normal interactive CrystalClear operation, it is only available through the menu by selecting **Processing** and then **Rank Crystal.**

Rank Crystal Dialog

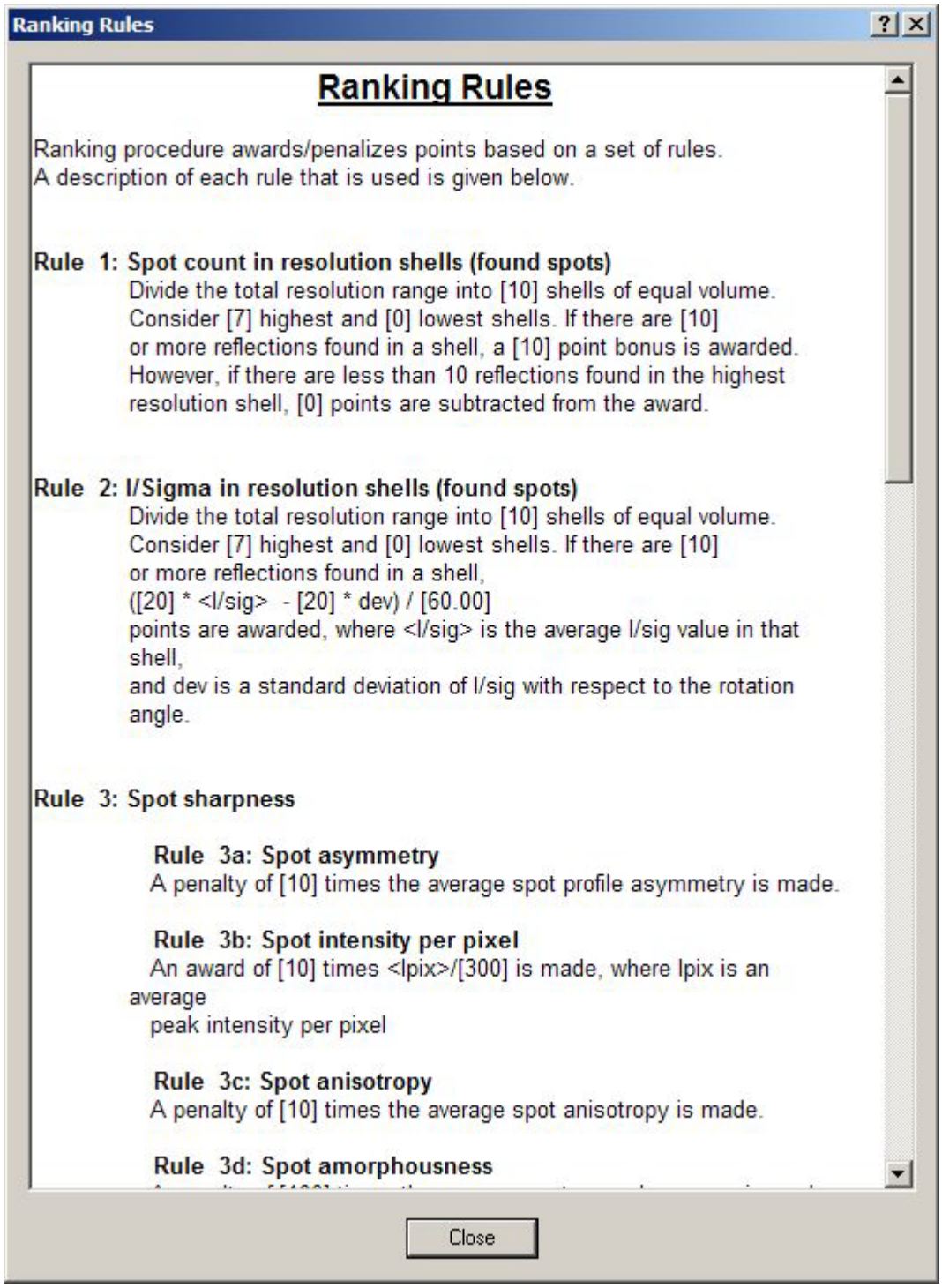


Parameter	Description
Scan table	This is the standard scan table for selecting the images on which to rank. Ranking is normally done on screen images before data is collected.
Ranking rules	The ranking rules fall into groups. You can turn each group off by unchecking the box. The options are: • **Peak Search**. How many peaks are found, how are they distributed through resolution shells, how good are the peaks, etc. This is probably the most useful criterion, so it is unlikely to be useful to uncheck it. • **Ice Rings**. Negative points if ice rings are detected. • **Index and Refinement**. Can the crystal be indexed? How good is the refinement?
Show rules	This displays a text viewer with the current ranking rules. An example is shown

Parameter	Description
	below.
Search resolution	You can specify the resolution to use in searching for peaks. If you know the crystal does not diffract very far, you might want to limit the resolution so that it won't be penalized for not having high resolution peaks. The standard CrystalClear resolution options are provided.
Normalize Scores	If you want to normalize your scores over a particular range you can specify a **Maximum** and **Minimum** score. This is only really useful if another program is looking at these scores and expects a fixed range.
Rotational anisotropy penalty coefficient	This is the weight applied to the penalty for rotational anisotropy.
Strategy rotation width penalty	If rank performs a Strategy step, then this specifies the penalty that is applied for samples that require more images to be collected. It is not currently used.
Ranking processing steps	Depending on the rules used and the steps that have already been performed, ranking performs several steps to determine the properties that go into the rank. • **Find reflection**s. Necessary for the **Peak search** rules. • **Index reflections**. Necessary for the **Index** rules. • **Refine parameters**. Necessary for the **Refine** rules.
Resolution bins	For the Peak search rules, ranking sorts the peaks into resolution bins and then bases the score on the number of peaks in each bin. This section allows you to control this process. • **Total number of resolution bins.** • **Number of high resolution bins.** • **Number of low resolution bins.** Not all bins are either high or low, so the high and low does not need to add up to the total. Low defaults to 0.
Reflection definition	These specify what it takes for a peak to be considered a reflection. • **Minimum signal/noise**. This is the minimum I/Sigma that a peak must have to be included. • **Minimum reflections per shell**. A shell is a resolution bin. This is the cutoff below which the sample is penalized for not having sufficient reflections for a particular shell.
View Previous Rank Results	This is used primarily for testing ranking results, but can be useful for comparing samples.
Clear Previous Rank Results	Clears out the list used for comparison.
Rank Crystal	This performs the actual ranking. Depending on the Ranking processing steps checked this might run dtfind, dtindex, and dtrefine as well as dtranker.

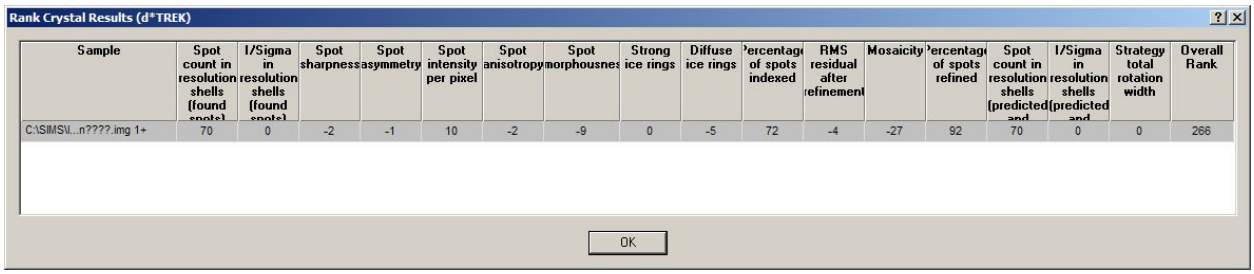
Rank Rules

When you click **Show Rules** you get the following display.



Rank Results

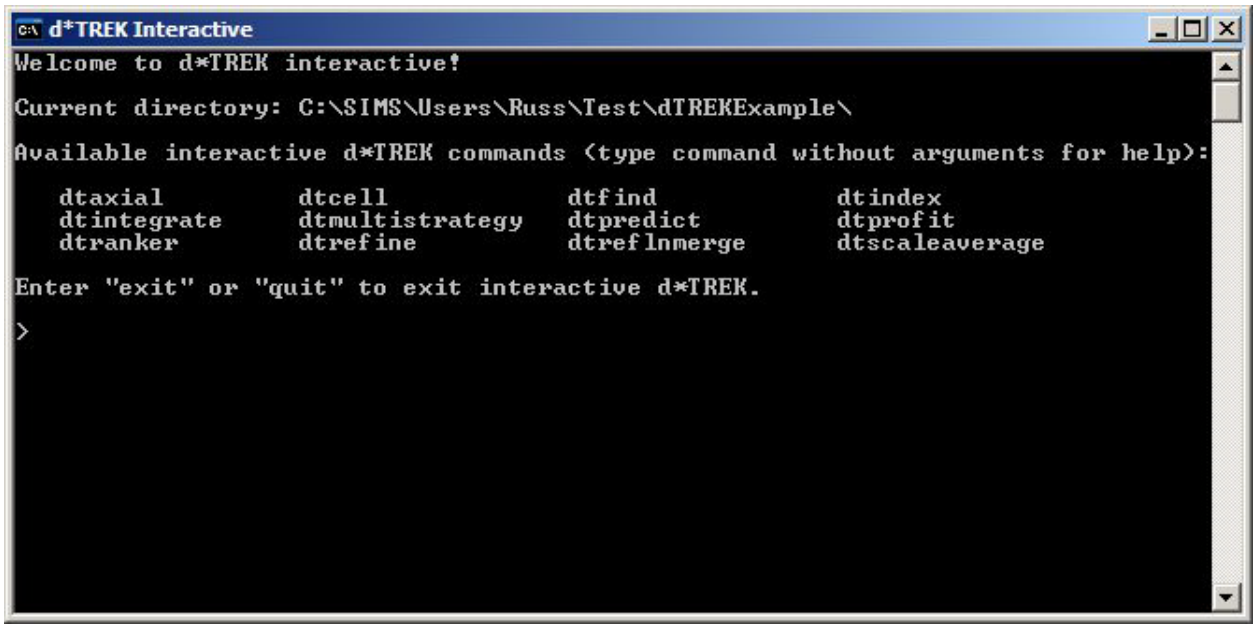
Once the ranking is done you get the following display.



If you have run rank several times, you see the results for all the samples. You can clear this table with **Clear Previous Rank Results**.

Run d*TREK Interactive

If you need to use a d*TREK option that is not supported by the CrystalClear, then you can use this tool to bring up a DOS window that allows you to type in d*TREK commands directly. The results of these commands are not used to update the state and the log files are just be shown in the DOS box, so this has limitations. It is an escape hatch for the brave who know a lot of d*TREK and is not recommended for mere mortals.



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9. FS_PROCESS Steps

Fine Slice PROCESS (FS_PROCESS) is based on Dr. Higashi's PROCESS program. Dr. Higashi and other scientific staff members at Rigaku in Japan have reworked much of the old PROCESS program and added many new features and refinements for processing fine sliced images. FS_PROCESS continues to be very effective for wider images (> 2 degrees) and is the recommended package for processing wide RAPID and SPIDER images for small molecule work.

Assign Unit Cell

The three steps in the Assign Unit Cell group include:

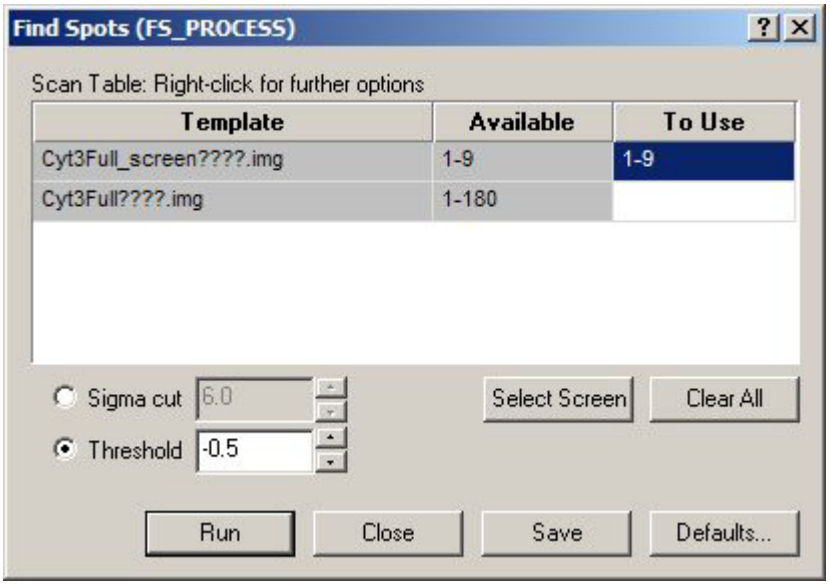
- Find Spots
- Index Spots (includes refinement in FS_PROCESS)
- Predict Spots

Find Spots

The first step after having collected screen images is to find spots to be used in indexing. These spots are written into a file called fsFind.ref.

In order for subsequent **Operations** to function properly (i.e. **Index Spots** and **Refine Cell**), using 50-70 reflection centroids in **Find Spots** is recommended. The exact number needed can vary depending on the centroid accuracy and location in reciprocal space.

Find Spots Dialog



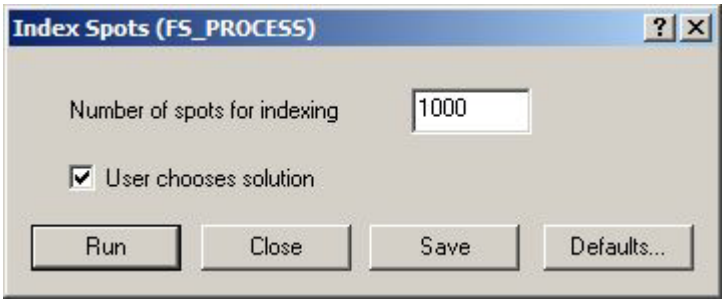
Parameter	Description

Parameter	Description
Scan Table	This is the standard scan table for selecting images to be used for the **Find Spots** step. By default the _screen images are selected.
Sigma cut	One option for specifying the criterion for a pixel value to be considered a potential part of a spot is the I/Sigma of the pixel.
Threshold	The other option is a threshold number

Index Spots

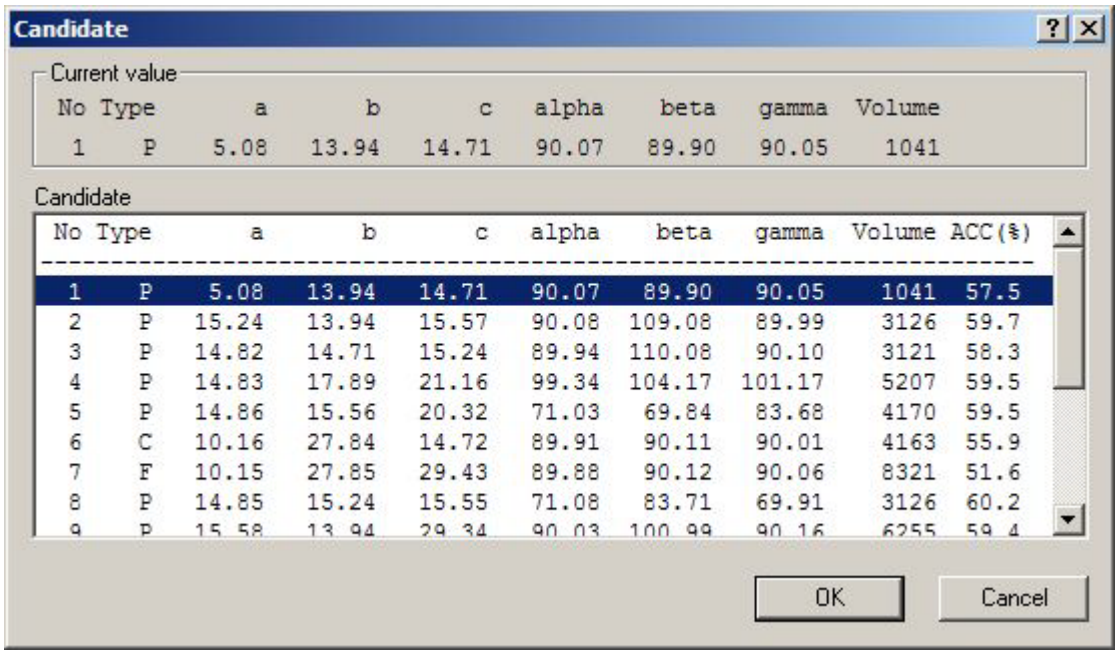
FS_PROCESS index requires very few parameters. After the solution has been selected (either by the user or automatically) it is refined, so no separate refinement step is necessary.

Index Spots Dialog



Parameter	Description
Number of spots for indexing	You can set a maximum number of spots to be used for indexing. Of course, if fsFind.ref has fewer spots, then the actual number of spots used may be smaller.
User chooses solution	If this is checked a table of candidate solutions is presented and you can select the solution that you think is right. If this box is not checked, then the most likely solution (the top line in the table if it had been displayed) is chosen.

Indexing Solution Candidate Table

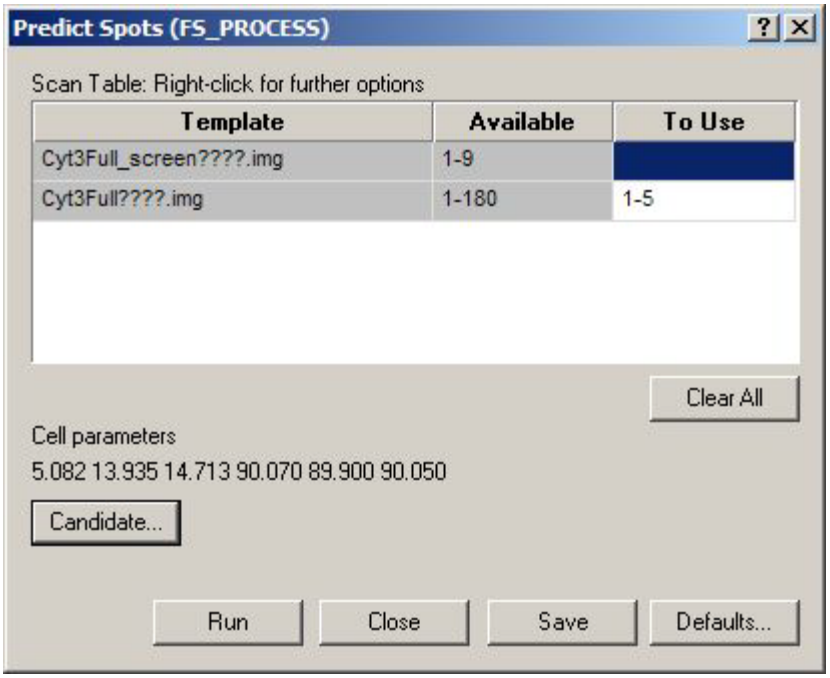


Look over the **Candidate** table to select the desired cell. The ACC(%) parameter is the percentage of reflections that fit the unit cell. Higher is better.

Predict Spots

It is a good idea to predict spots to make sure that they match the spots on the images. If you have already collected data, it is best to predict on collected images.

Predict Spots Dialog



Parameter	Description
Scan Table	This is the standard CrystalClear scan table for selecting the images you want to predict on.
Cell parameters	FS_PROCESS reports the current cell parameters in this area.
Candidate…	If you did not check **User chooses solution** in Index Spots or you decide at this point that you do not like the cell, you can click Candidate and you get the **Index Solution Candidate Table** described in the last table which gives you a chance to change the selected cell.

Integration and Scaling

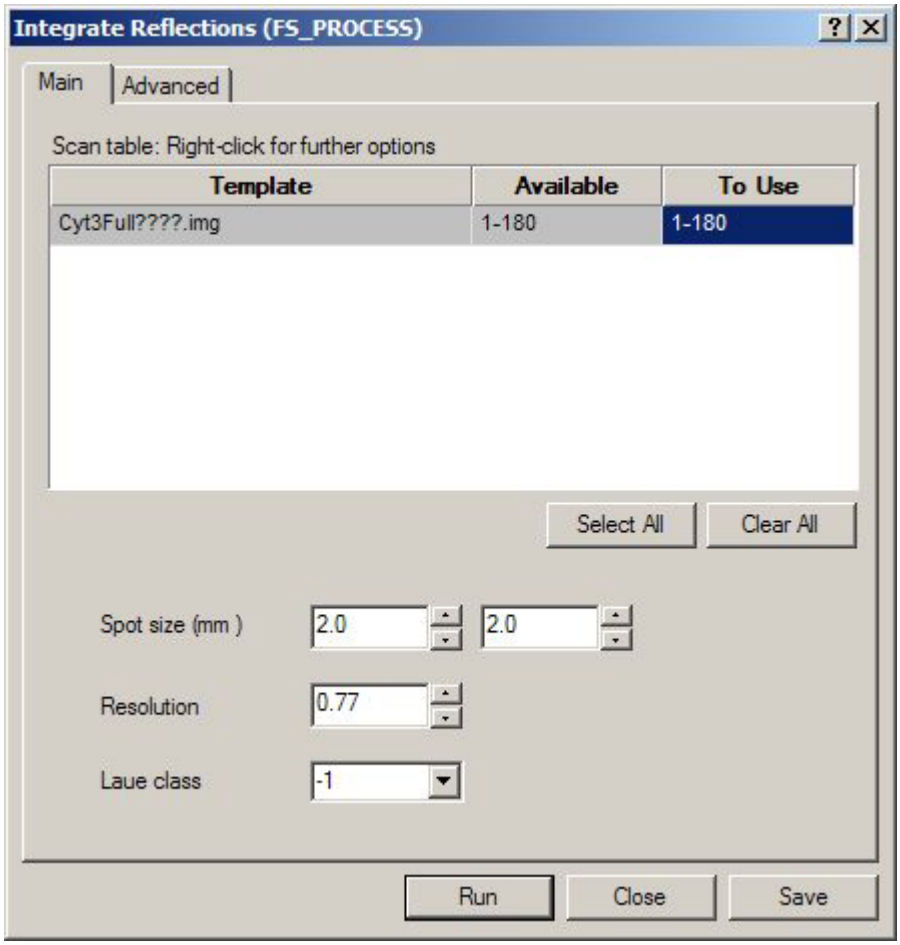
The last two steps in the FS_PROCESS flow are

- Integrate Reflections
- Scale and Average (includes multiple additional steps)

Integrate Reflections

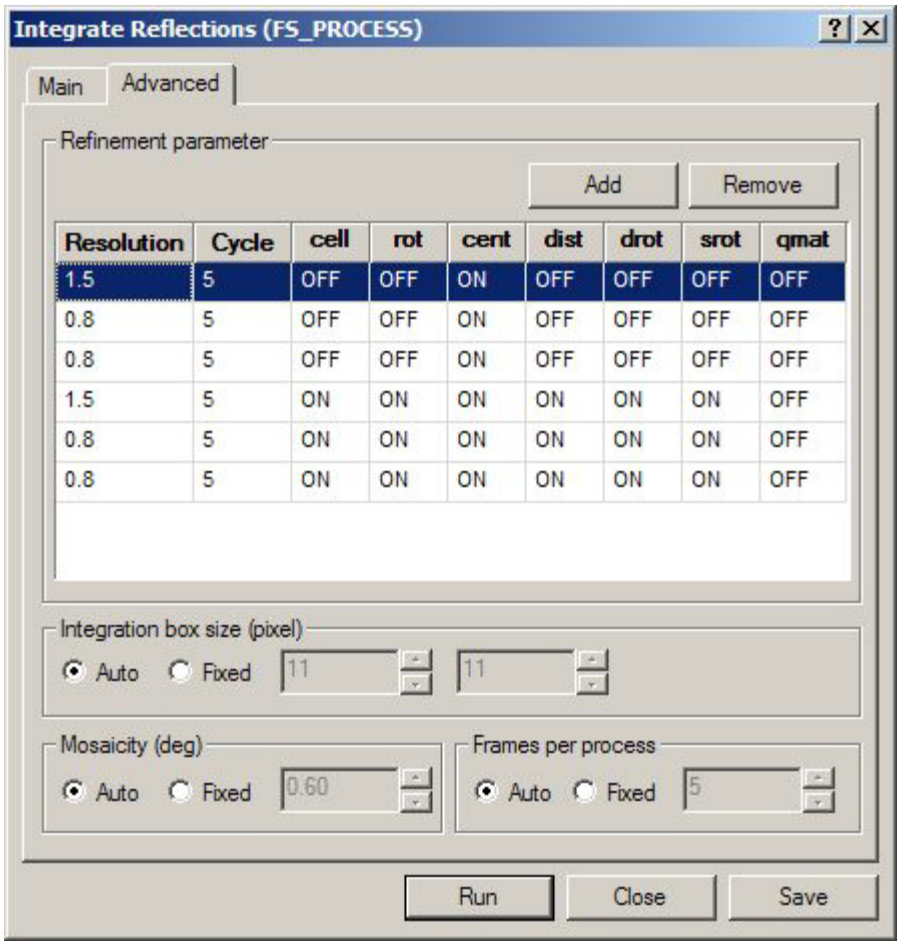
FS_PROCESS integration works well for both fine sliced and wide sliced images. Note that for consistency with FS_PROCESS log files the interface uses **Frames** where d*TREK and most the collection dialogs use Images. FS_PROCESS also uses **Process** rather than batch.

Integrate Reflections Main Tab



Parameter	Description
Scan Table	This is the standard CrystalClear scan table for selecting the images you want to integrate. By default all the images without _screen in the name are chosen.
Select All	A short cut to select all images.
Clear All	A short cut to clear all image selection.
Spot size	This is the average spot size in millimeters. FS_PROESS judges overlapped reflections based on these two parameters. These values should be set based upon the size of reflections and unit cell. Values that are too small may not calculate the overlap properly. Values that are too large may reject too many reflections.
Resolution	This is the maximum resolution for integration. Usually 1Å is used for refinement and 0.77Å and 0.83 Å are used for integration for Mo and Cu, respectively.
Laue class	You can specify the Laue class at this stage which can improve the quality of the integration.

Integrate Reflections Advanced Tab



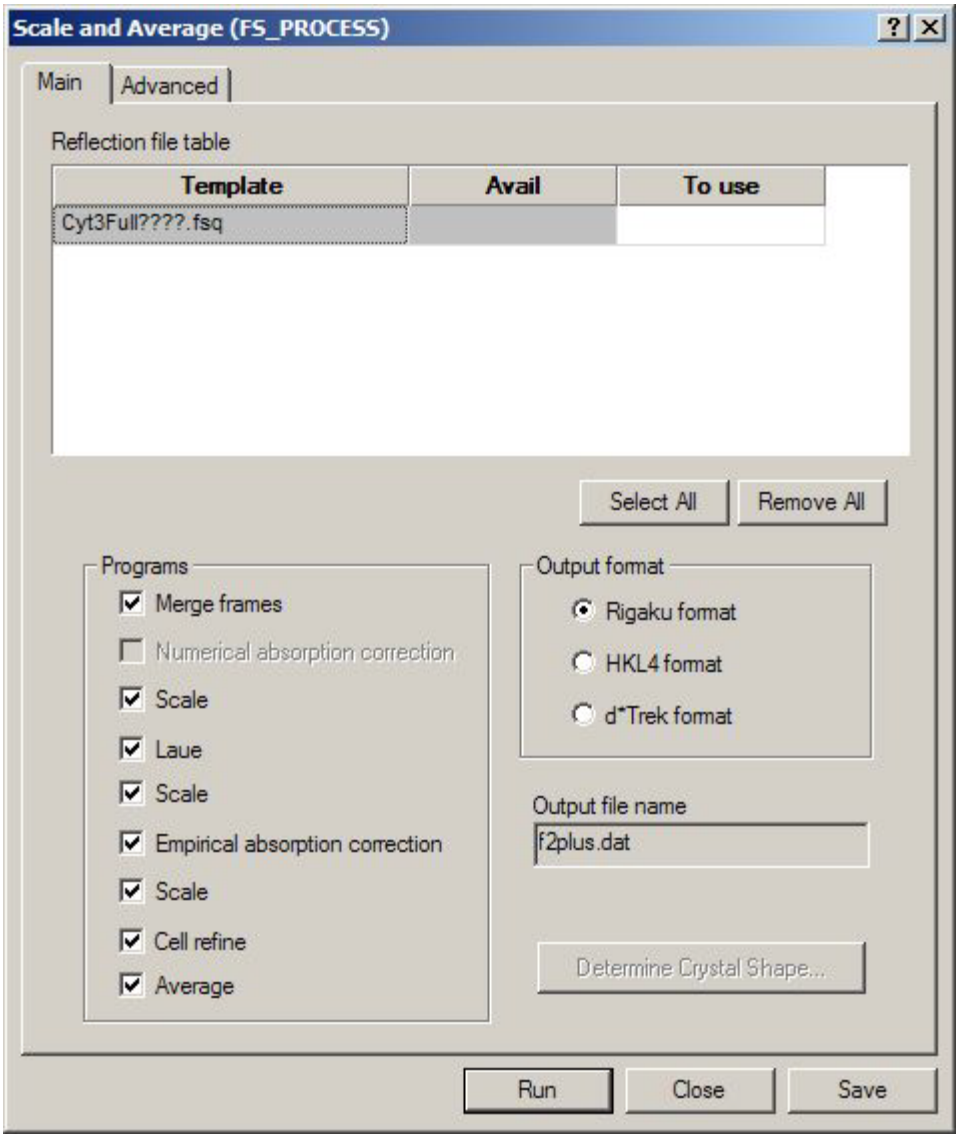
Parameter	Description

Parameter	Description
Refinement parameter	This table specifies the refinement procedure that is executed before integration starts and then before each process. Each row in the refinement specifies a different refinement step. You can edit this table by using the **Add** button to add a row or the **Remove** button to remove the selected row. For each step you specify: • **Resolution** The resolution cutoff for that step • **Cycle** The maximum number of refinement cycles to run. • **Cell** (ON/OFF) ON means refine cell parameters • **rot** (ON/OFF) ON means refine crystal mis-setting angles • **cent** (ON/OFF) ON means refine the direct beam position • **dist** (ON/OFF) ON means refine distance parameters • **drot** (ON/OFF) ON means refine the detector rotations • **srot** (ON/OFF) On means refine the source rotations • **qmat** (ON/OFF) On means refine the q matrix, which describes the nonunformity of the x and y pixels.
Integration box size	• **Auto** which is the normal default means the integration box size is determined automatically. • **Fixed** means that you specify a fixed box size in pixels.
Mosaicity	• **Auto** means that the mosaicity is estimated by FS_PROCESS as it is working. • **Fixed** means that you fix the mosaicity to the specified value.
Frames per process	• **Auto** means that FS_PROCESS automatically figures out when it needs to re-refine. • **Fixed** allows you to specify the number of frames (images) in each process (batch).

Scale and Average

In FS_PROCESS the Scale and Average Step runs a selected set of programs and thus replaces several steps in d*TREK.

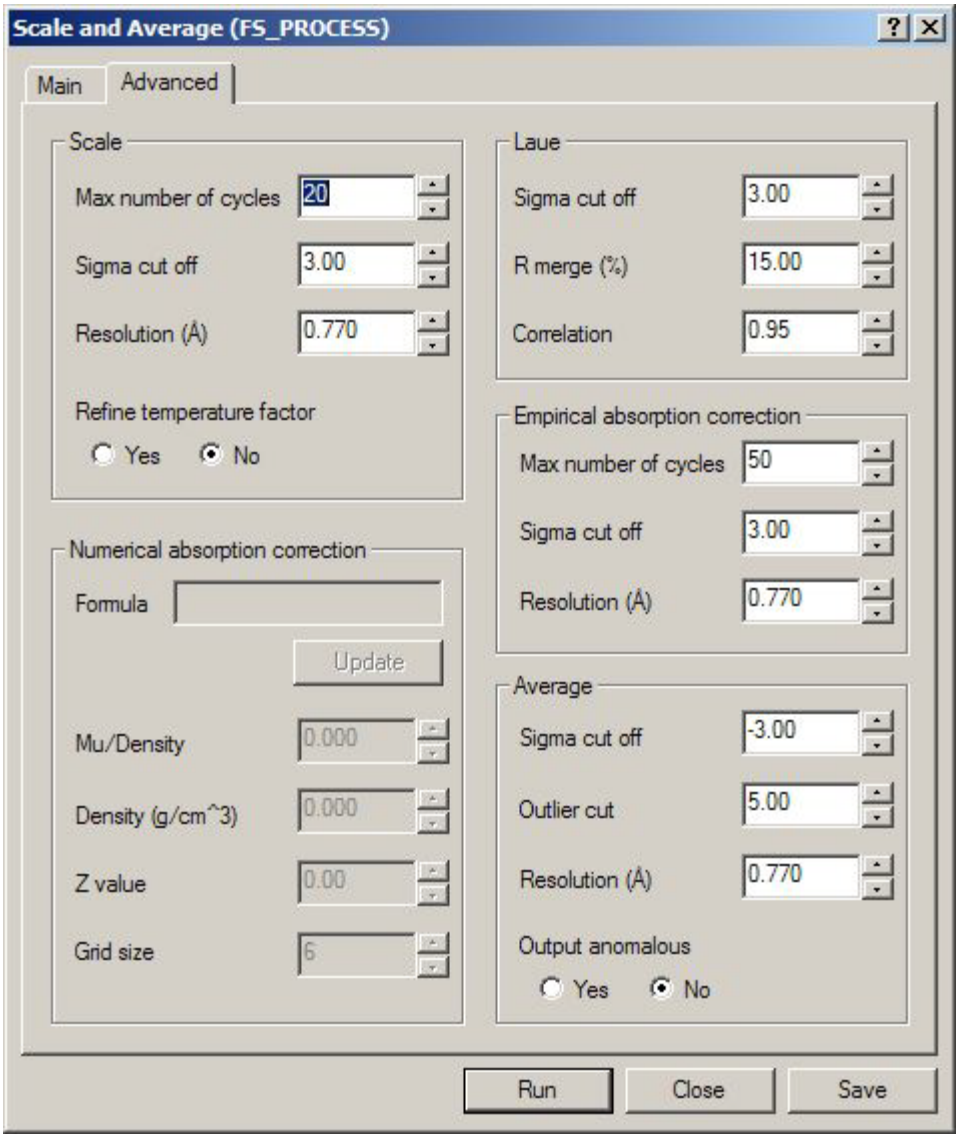
Scale and Average Main Tab



Parameter	Description
Reflection file table	FS_PROCESS produces a reflection file for each frame (image). This table behaves much like the scan selection table and allows you to select which reflection files are included in the **Scale and Average** step.
Select All	A short cut to select all reflection files
Remove All	A short cut to remove all reflection files selection.
Programs	The Scale and Average step runs a list of program. You should check the programs you want to run. If a program is grayed out, it is not valid to run at this point. • **Merge frames**. Merges the individual reflection list associated with each frame into a single reflection list. • **Numerical absorption correction**. Calculates the absorption effect within the crystal and applies absorption correction based on the shape of the crystal obtained by the shape tracing software shape. This requires that you have used RAXShape to define the shape of the

Parameter	Description
	crystal. • **Scale**. This is an initial scaling step. Scale can be repeated several times in this process. • **Laue**. Performs a Laue group check. • **Scale**. • **Empirical absorption correction**. This runs the FS_PROCESS absorption collection program. (Numerical absorption and empirical absorption are mutually exclusive). • **Scale**. • **Cell refine**. A final cell refinement. • **Average**. The final step is to average the reflections.
Output format	Several output file formats are available. Each format defaults to a different (and fixed) output file name. • **Rigaku format** – Standard output for CrystalStructure in f2plus.dat • **HKL4 format** – Legacy format in f2.dat • **d*TREK** – Compatible with dtscaleaverage.ref, but named dtrek.dat.
Output file name	This is a read only display of the file name that is used for output. It is based on the **Output format** and cannot be changed.
Determine Crystal Shape…	This button starts up RAXShape so that you interactively determine the crystal shape. This requires that you have a video camera and video capture card on your system and that you have RAXVideo installed.

Scale and Average Advanced Tab



Parameter	Description
Scale	The Scale program is often executed multiple times. These parameters apply to each time it is run. • **Max number of cycles**. • **Sigma cut off**. The I/Sigma value that each reflection must have to be considered in scaling. • **Resolution**. Reflections that have higher resolution than this are not considered for scaling. • **Refine temperature factor**. In this is not checked only the scale factor is refined, and if it is checked, the scale factor and temperature factors are refined. The temperature factor may be applied when intensities decay isotropically, such as protein crystals.
Numerical absorption	These parameters apply to the Numerical absorption correction program.

Parameter	Description
correction	• **Formula**. You should enter the molecular formula and click the **Update** button to update the computed Z value and other computed values. • **Mu/Density.** Mass attenuation factors • **Density** • **Z value.** This is computed from the molecular formula, but you can edit it as needed. • **Grid size**
Laue	The parameters apply to the FS_PROCESS Laue check program. • **Sigma cut off**. Reflections with a lower I/Sigma value are not included in the Laue evaluation. • **Rmerge**. • **Correlation.** This is the deformation correlation lower-limit value for judging the symmetry.
Empirical absorption correction	FS_PROCESSS has its own empirical absorption correction program that works especially well for small molecules. The parameters for this include: • **Max number of cycles.** • **Sigma cut off**. Reflections with I/Sigma lower than this value are not used to calculate the absorption correction. • **Resolution**. Reflections with higher resolution than this are not used in absorption correction calculation. The resolution is in Angstroms.
Average	For the averaging program the parameters include: • Sigma cut off • Outlier cut • Resolution • Output anomalous. If this is Yes, the log file includes information about the anomalous differences for Bijvoet pairs (I+, I-).

Scale and Average Step Description

Merge frames

Merges intensity data files from each image and generates a merged reflection file. Merge uses the intensity data selected at **Reflection List**.

Cell Refine

Cell Refine determines precise cell constants and the orientation matrix with the post refinement method.

Scale

Performs scaling of each image.

Laue

Examines the symmetry of intensity data and determines the Laue symmetry. If the program has failed to determine the Laue symmetry, the following reasons are possible.

1. There are not enough pairs to judge the symmetry due to the limited measurement region within the reciprocal space.

2. The merged file contains intensities obtained under different conditions but not well scaled. The intensities should be scaled prior to running Laue.

3. The absorption effect on intensities is large. In this case, the intensities should be corrected for absorption before Laue.

Numerical absorption correction

Numerical absoprtion calculates the absorption effect within the crystal and applies absorption correction based on the shape of the crystal obtained by the shape tracing software shape. In order to run this you must have run RAXShape to get a shape description file. If you have not done this then this step is disabled.

Empirical absorption correction

Empirical absorption correction approximates the absorption surface by a Fourier series. The Fourier coefficients are determined by the least squares fitting assuming that the intensity differences among equivalent reflections are caused by absorption effect.

Average

Averages intensities of equivalent reflections and writes the averaged intensity to the output file. If the anomalous diffraction option is specified, the intensity difference originated from anomalous diffraction effect is determined and delivered to the output file.

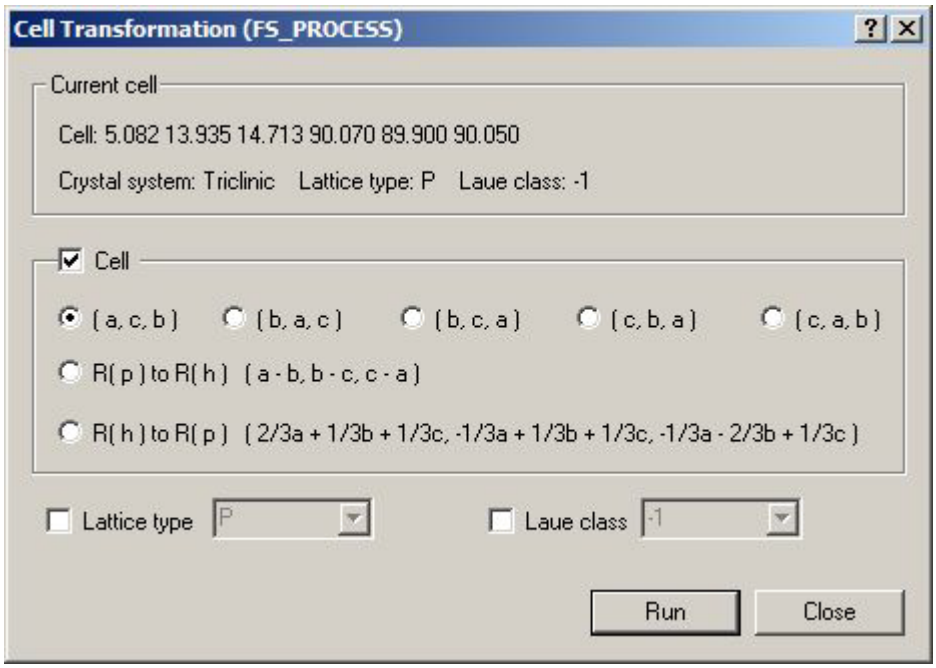
Menu Operations

The operations included in this section are accessible through the menu, but do not have corresponding steps in the flow bars so they have not been documented elsewhere.

Cell Tools>Transform

The Transform Cell command is different for depending on the processing suite. For FS_PROCESS the options are somewhat different than they are for d*TREK or TwinSolve.

Cell Transform Dialog

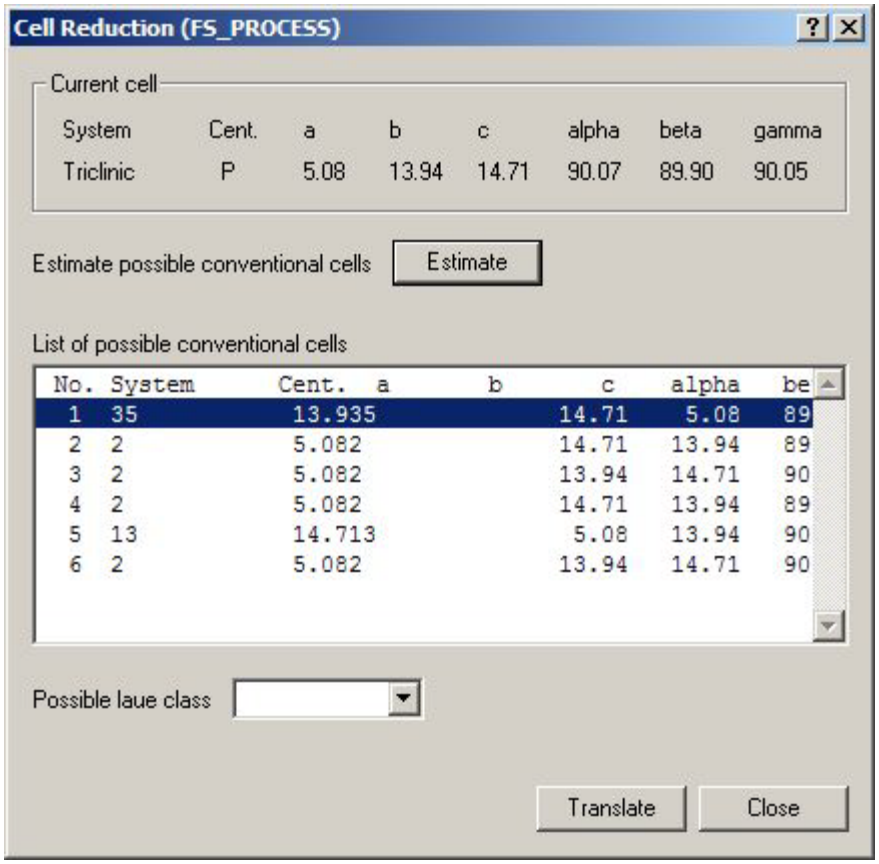


Parameter	Description
Current Cell	The current cell information is taken from the sample state. This reports the cell parameters, **Crystal system**, **Lattice type** and **Laue class**.
Cell	If **Cell** is checked, then the selected transformation is applied and the sample re-indexed. FS_PROCESS provides a set of standard transformations from which you can choose.
Lattice type	If you check this you are able to change the Lattice type in the transformation by selecting another Lattice type from the drop down list.
Laue class	This allows you to select a new Laue class for the transformation.

Cell Tools>Reduce

In the FS_PROCESS flow bars cell reduction is not a built in step. However there is a command available to reduce the cell to a conventional cell.

Cell Reduction Dialog



Parameter	Description
Current Cell	The current cell information is taken from the sample state. This reports the cell parameters, **System** and **Centering.**
Estimate	Click this button to generate a list of possible conventional cells based on the current cell.
List of possible conventional cells.	This list is filled out once **Estimate** has finished.
Possible Laue class	If the Laue class can be determined, there is drop down list of possible of Laue classes from which you can select.
Translate	This translates the current cell to the selected conventional cell.

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10. TwinSolve Steps

TwinSolve was developed by Dr. Christer Svensson. It is licensed separately from CrystalClear, but usually included for most small molecule customers. As its name implies, TwinSolve is especially well suited to dealing with twinned small molecule crystals.

The CrystalClear interface allows the user to study the images from a twinned crystal. The twin relationships (twin laws) are generated for each pair of components. Reflections from each twin component are integrated, including the overlapped reflections. Absorption corrections for each component are applied using shape factors (based on measurements of equivalent reflections) expanded in terms of spherical harmonics (up to order 8).

TwinSolve properly handles both fine- and wide-slide image data from either CCD or image-plate detectors. In addition to Rigaku image data, **TwinSolve** processes images from Bruker, MAR, ADSC, and Nonius detectors.

TwinSolve is not limited to use with twinned crystals; it also produces high-quality integrated, absorption corrected data from single crystals.

Assign Unit Cell

TwinSolve has a separate step for cell refinement like d*TREK. The five steps for assigning the unit cell include:

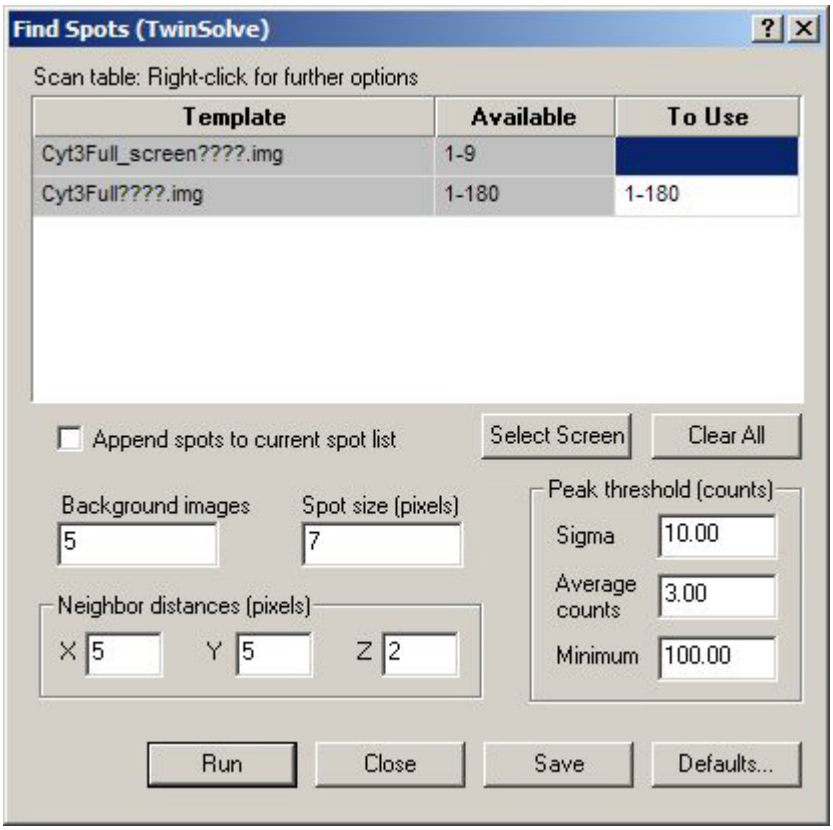
- **Find Spots**
- **Index Spots**
- **Refine Cell**
- **Get Twin Relations**
- **Predict Spots**

Since TwinSolve is used primarily for twinned crystals, it is common to create a process only sample which refers to the image data from the original sample where it was created. TwinSolve **Find Spots** is normally performed using the collected images rather than the screen images. When you **Index Spots** with TwinSolve it picks out the spots which belongs to the major twin component to be used in indexing. It then removes them from the spot list. You should next **Refine Cell** on that component and then return to **Index Spots** for the next component. You should continue to loop through **Index Spots** and **Refine Cell** until no significant twin components are left.

Find Spots

Normally you select all collected images for the **Find Spots** step.

Find Spots Dialog



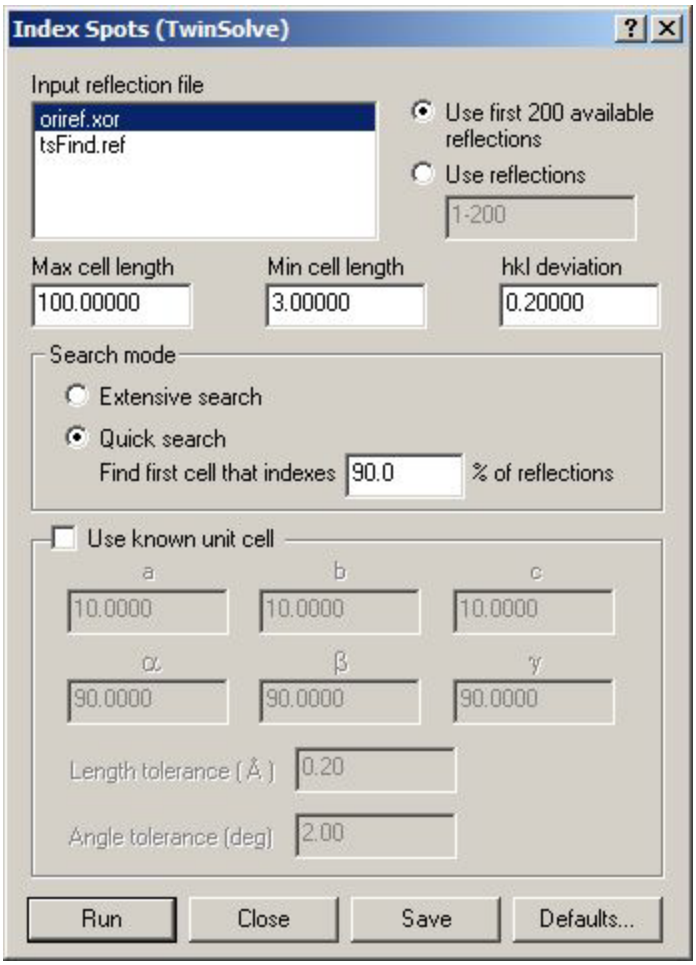
Parameter	Description
Scan Table	This is the standard scan table for selecting images to be used for the **Find Spots** step. By default the collected images are selected rather than the screen images. This is the normal and suggested practice for TwinSolve.
Select Screen	This is a short cut to select all the screen images. It is included for consistency with the other processing suites, but in TwinSolve it is often better to use the collected images.
Clear All	This short cut clears all image selections so that you can start over.
Append spots to current spot list	If you have done a **Find Spots** on a partial data set and want to add more images without starting over, you can check this box and then just select the new images.
Background Images	Number of images to use to calculate an average background image.
Spot Size	This is the number of pixels for an average spot.
Neighbor distance	These are the X, Y, and Z average distances between neighboring spots measured in pixels.
Peak threshold	These are the criteria for choosing peaks. • **Sigma**. The I/Sigma above which pixels are considered as belonging to a peak. The I/Sigma is calculated after the non-uniformity correction has been applied. You can specify **Sigma** as 0, TwinSolve does not use this criterion which speeds up the process because it does not need to calculate an average background and standard deviation. However, a Sigma cutoff can often be useful, so generally you should set this to a reasonable value.

Parameter	Description
	• **Average Counts** above the background average. • **Minimum**. This specifies the minimum count that a pixel must have to be considered part of a peak. A pixel must be above this minimum and have an I/Sigma higher than specified (if Sigma is not 0) in order to considered as part of a peak.

Index Spots

The TwinSolve **Index Spots** step indexes the major component, if any, from the remaining spot list. The indexed spots are then removed from the reflection file and this step is normally repeated (after doing a **Refine Cell**) until no major component is found.

Index Spots Dialog

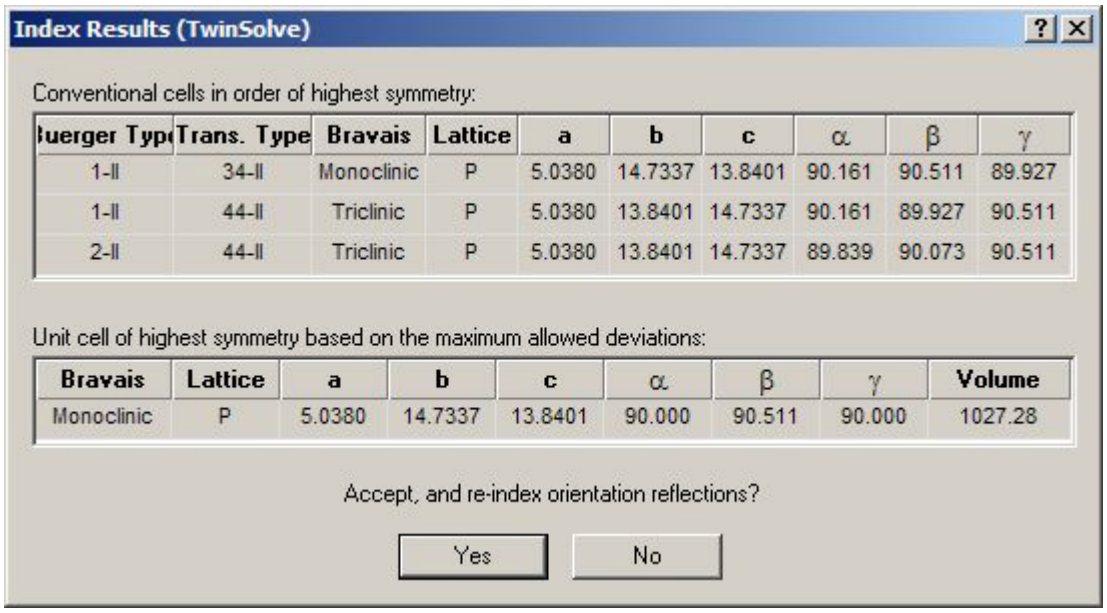


Parameter	Description
Input reflection file	The internal oriref.xor file should normally be selected. TwinSolve updates this file after indexing to mark the spots that have been used in the twin component indexed during the last run.
Use first 200 available reflections	The normal and recommended number of reflections to use is 200.
Use reflections	You can also specify a range of reflections to use (e.g., 100-500) if you don't

Parameter	Description
	want to use the first 200 for some reason.
Max cell length	If you enter 0, TwinSolve does not reject possible indexing because the cell is too big. It is usually best to specify a reasonable maximum cell dimension in Å.
Min cell length	If you want TwinSolve to reject cells that are two small enter a non 0 minimum cell dimension.
Search mode	There are two search modes available • **Extensive**. This takes longer but may find cells missed by the **Quick** mode. • **Quick**. This mode requires you to specify the percentage of reflections that must be indexed to cutoff the attempt to find a solution.
Use Known Cell	If you check this box, you must enter the cell parameters for the known cell. You should also specify: • **Length tolerance**. This specifies the deviation that is allowed from the specified lengths in Ångstroms. If you enter 0, a 3% error is used. • **Angle tolerance**. This specifies the deviation from the specified cell angles in degrees. If you enter 0, a 3% error is used. If there is a cell specified in the header it is first converted to a reduced primitive cell to provide defaults for the known cell. TwinSolve searches for a match to this reduced primitive cell.

Index Spots Results Table

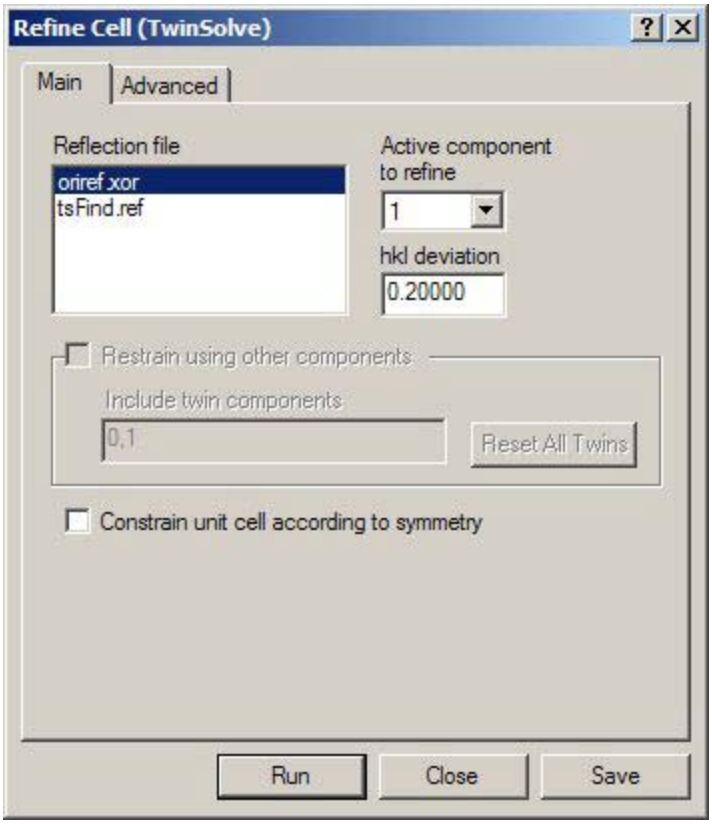
The results of the TwinSolve indexing are displayed in a table. You are given the chance to accept this indexing scheme which updates the reflection list to mark the reflections that have been indexed by this solution. If you don't accept this solution, then you can either return to change the index parameters or continue on with the components that you have found.



Refine Cell

After each component has been indexed, the cell should be refined by running the **Refine Cell** step.

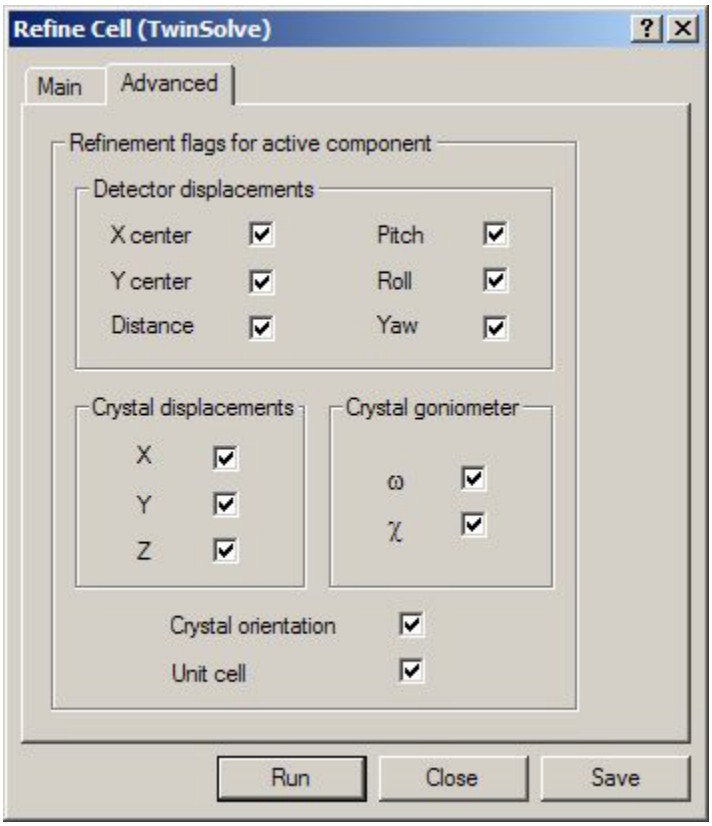
Refine Cell Main Tab



Parameter	Description
Reflection file	The internal oriref.xor file should normally be selected. TwinSolve refines on reflection files rather than images.
Active Component	The twin components are numbered as they are created by **Index Spots**. Normally you refine the last component you created.
hkl deviation	This is the maximum allowed deviation that a reflection can have from the expected position and still be considered part of this component. When refining twins, it may be necessary to reduce this parameter to prevent overlapped reflections from biasing the refinement.
Restrain using other components	This causes the refinement to be restrained so that it is compatible with other twin components. This assumes that the twin components are just different orientations of the same unit cell. The components you want to use to restrain the refinement should go in a list separated by commas into the **Include twin components** edit box. The **Reset All Twins** buttons uses the refined cell for all twin components.
Constrain unit cell according to symmetry	Check this to force the refined cell to conform to the current symmetry class for this component.

Refine Cell Advanced Tab

This tab allows you to select the parameters to refine. If you need to refine only some of them at a time, you can start with a subset of these parameters checked and then add more checks and rerun for each step in the refinement process.



Parameter	Description
Detector displacements	The detector options include • **X center, Y center** (Direct beam) • **Distance** • **Pitch, Roll, Yaw**
Crystal displacements	The crystal displacement options include: • **X, Y, Z**
Crystal goniometer	The crystal goniometer options include: • **Omega, Chi** (Chi may be Kappa on your goniometer)
Crystal orientation	Rot1, Rot2, Rot3
Unit cell	This may have been constrained or restrained by the options on the main tab.

Refine Cell Results

After it runs Refine Cell displays an extensive table of results and gives you the option to accept or reject them.

Refine Results (TwinSolve)

Statistics

Reflections

Total	3270	C	0 / 0
Used	2598	Q	2598 / 3270
Indexed, not used	434	Not indexed	238
Indexed, but extinct for this Bravais lattice	0		

RMS Deviations

h	0.007	Position	0.0800
k	0.019	Rotation	0.1100
l	0.016		

Goodness of fit: 0.9938

Maximum Λ/σ: -1.141 (b* length)

Statistics for 2598 Q reflections

Ave 0.00 RMS 0.23 Max offset 0.50

hkl deviations from integers and rotation errors for 2598 indexed reflections:

	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.40	0.45	0.50
h	2598	0	0	0	0	0	0	0	0	0
k	2547	51	0	0	0	0	0	0	0	0
l	2574	24	0	0	0	0	0	0	0	0
r	1160	716	288	185	113	93	43	0	0	0

Refined parameters, standard deviations(σ), and final shifts (Λ)

Direct cell:

	a	b	c	α	β	γ	Volume
Result	5.0732	14.6971	13.9065	90.044	90.003	89.964	1036.88
σ	0.0014	0.0040	0.0038	0.010	0.010	0.010	0.49
Δ	0.0015	0.0045	0.0030	0.003	0.002	0.001	0.84

Reciprocal cell:

	a*	b*	c*	α*	β*	γ*
Result	0.1971	0.0680	0.0719	89.956	89.997	90.036
σ	0.0001	0.0000	0.0000	0.010	0.010	0.010
Δ	-0.0001	-0.0000	-0.0000	-0.003	-0.002	-0.001

Detector:

	Beam X	Beam Y	Distance	Pitch	Roll	Yaw
Result	-0.161	-0.209	0.113	0.001	0.164	-0.049
σ	0.003	0.002	0.013	0.012	0.007	0.016
Δ	0.000	-0.000	0.014	-0.004	0.002	0.002

Omega/Chi zero point offsets (deg):

	ω	χ
Result	-0.000	-0.000
σ	0.000	0.000
Δ	0.000	0.000

Crystal orientations (deg):

	Rot1	Rot2	Rot3
Result	36.385	-49.296	107.416
σ	0.009	0.010	0.018
Δ	0.002	-0.003	-0.003

Crystal Displacement Origin (mm):

	X	Y	Z
Result	0.000	0.000	-0.000
σ	0.000	0.000	0.000
Δ	0.000	0.000	0.000

Orientation matrix:

-0.1202980	-0.1284991	0.0887178
0.0220041	-0.0492042	-0.0415250
0.0520290	-0.0163587	0.0468641

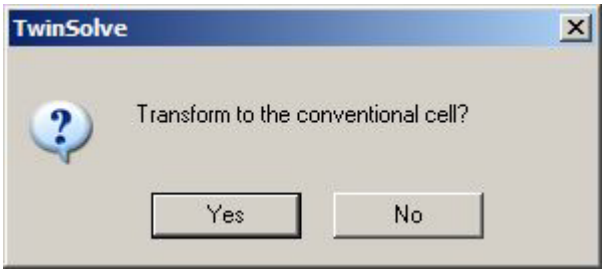
Additional information:

Twotheta range: 2.93 to 48.07 deg; d range: 0.871 to 13.894 angstroms
Refinement flags: 1111110000011111111

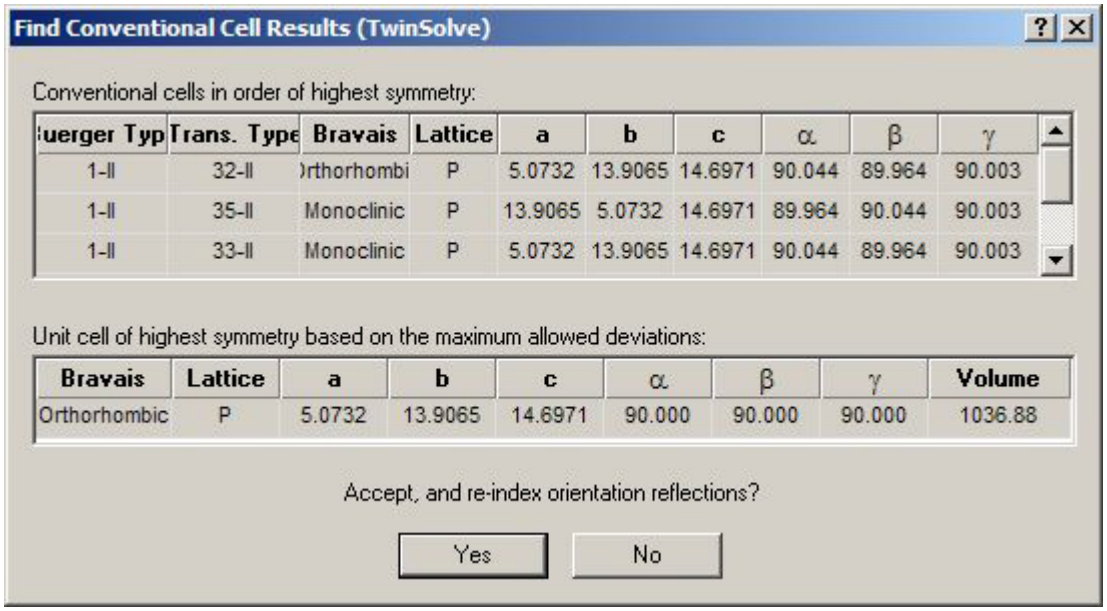
Accept these parameters?

Yes No

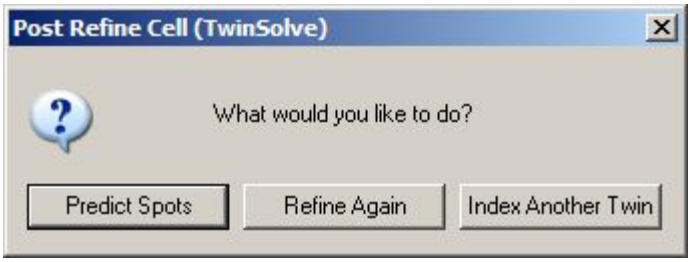
If you accept these results, you are asked if you would like to transform this to a conventional cell.



If you click Yes you get an updated Index results table.

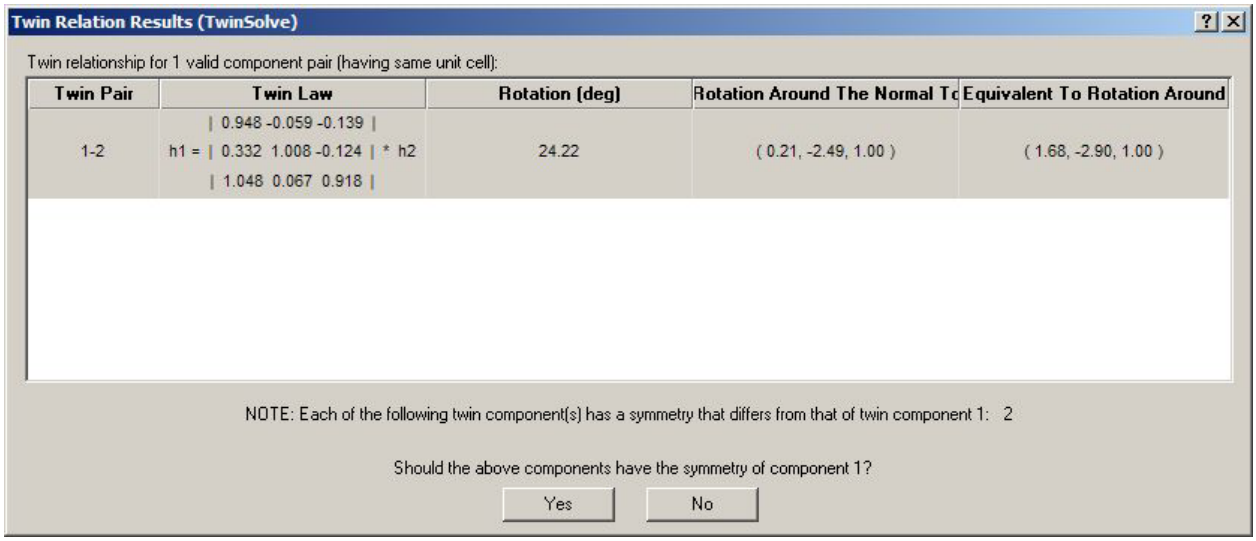


Finally you are asked what you want to do next.



Get Twin Relations

If you have indexed more than one component, before you **Predict Spots** you are given the chance to review the twin relations in the **Get Twin Relations** step. When you click this step it runs immediately and shows you the results.

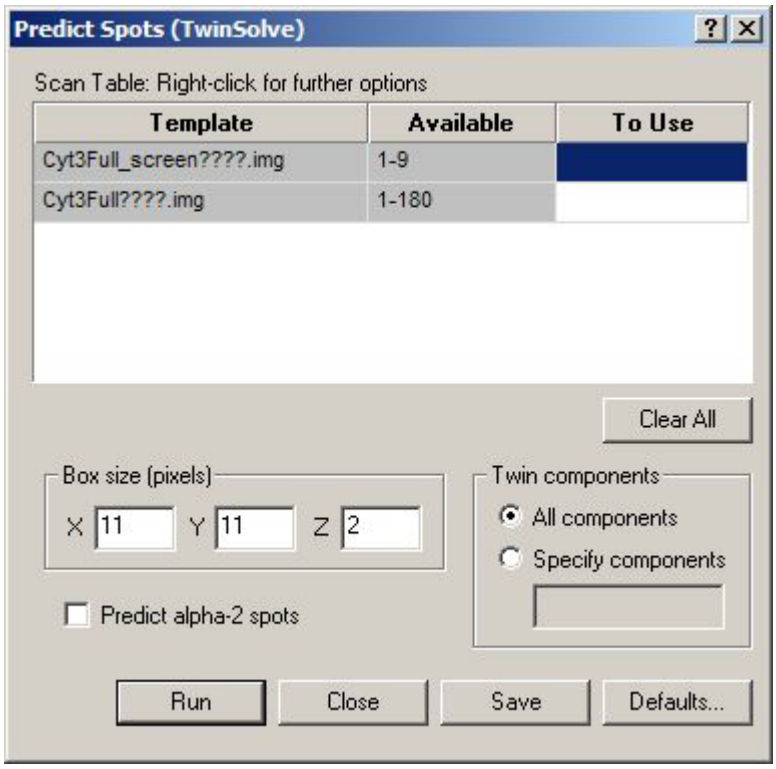


For each Twin Pair, this table reports a Twin Law and associated information. You are also given the chance to force all twin components to belong to the symmetry group of the first component.

Predict Spots

Once you have completed indexing and refining all the components, you can go on to Predict Spots to check the indexing.

Predict Spots Dialog



Parameter	Description
Scan Table	This is the standard scan table for selecting images to be used for the **Predict**

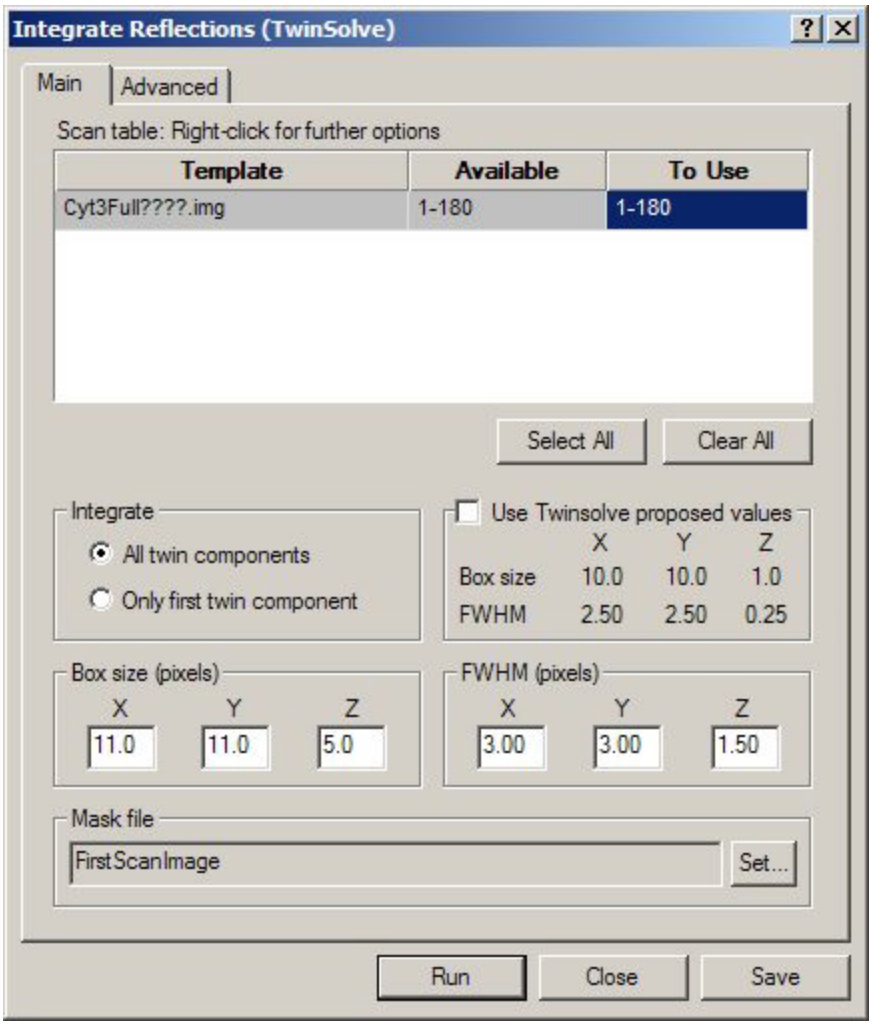
Parameter	Description
	Spots step. No images are selected by default, so you can choose the scans and images you want to predict for.
Clear All	This short cut clears all image selections so that you can start over.
Box size	The box size is used the starting point for the box size calculation for integration.
Twin components	You can either predict with **All components** or Specify components by listing them in the edit box separated by commas (e.g., 1,3,4)
Predict alpha-2 spots	If you are getting significant alpha-2 radiation onto your sample in your instrument you may want to predict the alpha-2 spots to make sure they are as expected.

Integrate

Integrate Reflections

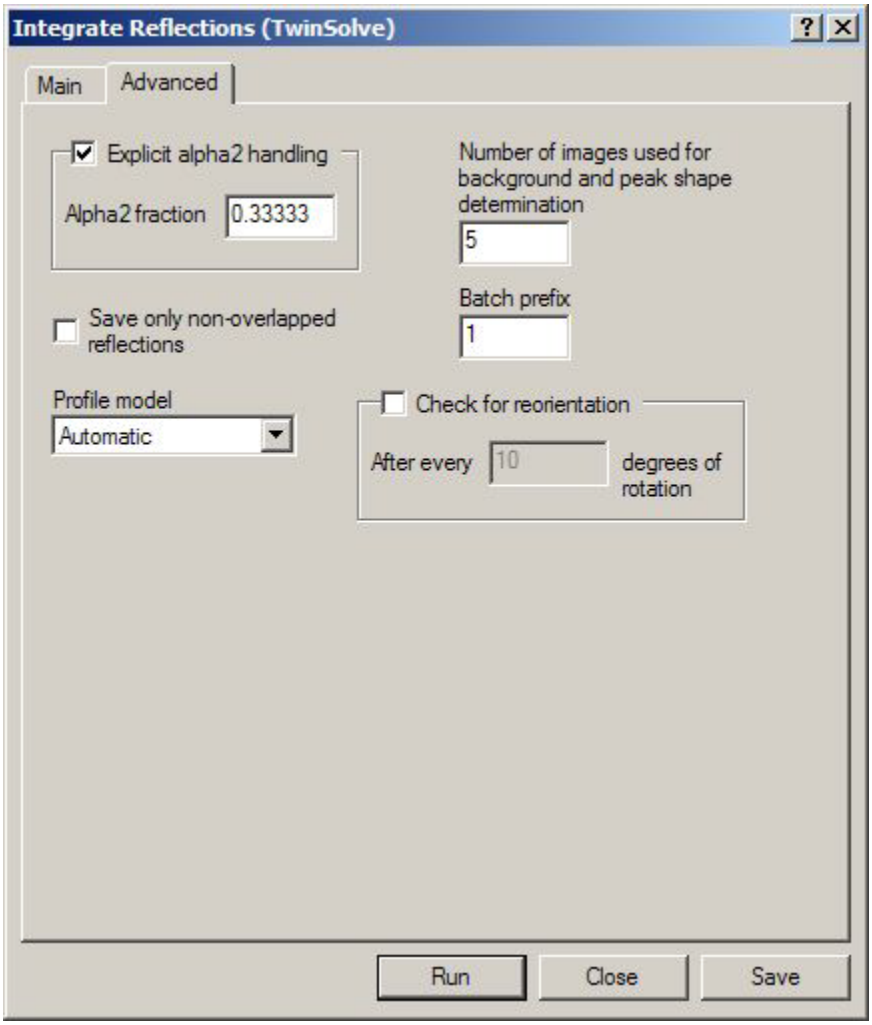
TwinSolve is able to integrate all components of a twinned crystal in a single run.

Integrate Reflections Main Tab



Parameter	Description
Scan table	This is the standard scan table for selecting images to be used for the **Integrate Reflections** step. Screen scans are not included in the **Scan table**.
Select All	This is a short cut to select all the images in the sample.
Clear All	This short cut clears all image selections so that you can start over.
Integrate	You can chose between **All twin components** and **Only first twin component**.
Use TwinSolve proposed values	Check this box is you want to use the values that TwinSolve proposes for the Box size and Full Width Half Maximum (FWHM). If you do not check this box, you should make sure that the values in the dialog below are as you intend.
Box size	This is the integration box size in **X, Y**, and **Z** in pixels.
FWHM	This is the Full Width Half Maximum (FWHM) in pixels.
Mask file	You should usually select a mask file to mask out the shadow of the beam stop. The mask file can be created using the Image Display. The Set… button opens a file browser so that you can search for the mask file you would like to use.

Integrate Reflections Advanced Tab



Parameter	Description

Parameter	Description
Explicit alpha2 handling	Check this box if you want to explicitly predict and integrate alpha 2 reflections. You must specify the **Alpha2 fraction**.
Save only non-overlapped reflections	Check this box if you only want to integrate reflections that do not overlap with other reflections.
Profile model	The two options are: • **Automatic**. The profile is adjusted automatically by TwinSolve. • **Default Peak Shape**. Use the default peak shape for all reflections.
Number of images used for background and peak shape determination	Background and automatic peak shapes are determined using multiple images. This parameter lets you adjust the number of images that are used.
Batch prefix	The prefix for the first batch.
Check for reorientation	If you check this box the orientation is refined periodically. When this happens is controlled by After every specified degreed of rotation. Note that this control is in degrees in the scan axis and not in number of images which might be different.

Analyze Data

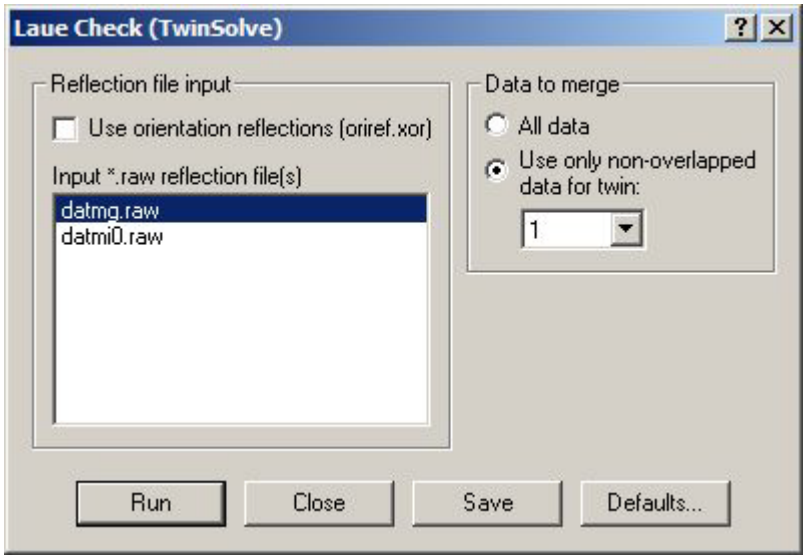
Once the data have been integrated there are two steps in the **Analyze Data** group.

- Laue Check
- Space Group Check

Laue Check

The **Laue Check** step is used to determine the Laue group of the sample.

Laue Check Dialog



Parameter	Description
Reflectin file input	You can either **Use orientation reflections** (oriref.xor) or select one or more *.raw files from the list to use for the **Laue Check** step.
Data to merge	Reflection data is merged before the **Laue Check** is performed. The selection for **Data to merge** is either **All data** or **Use only non-overlapped data for twin**. If you choose this second option you must select a twin component.

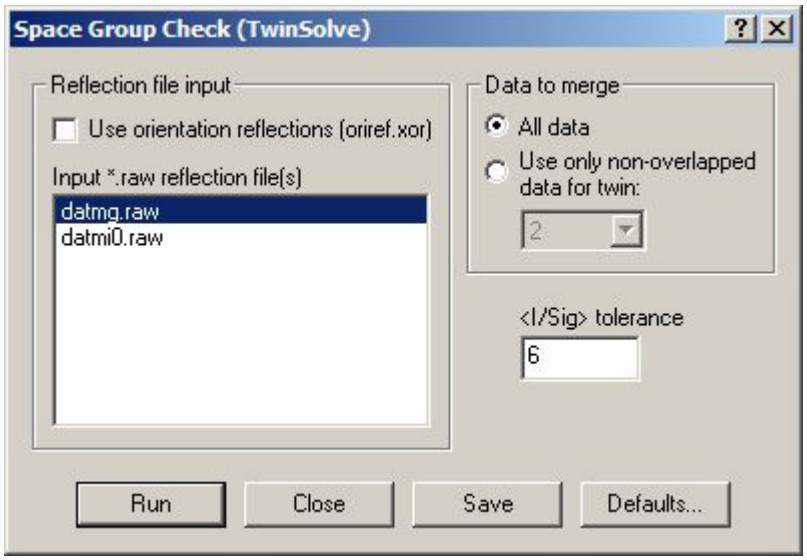
Laue Check Results

The **Laue Check** results are shown in the **Space Group Results** table described in the next section.

Space Group Check

After the Laue Check, you should normally do a space group check.

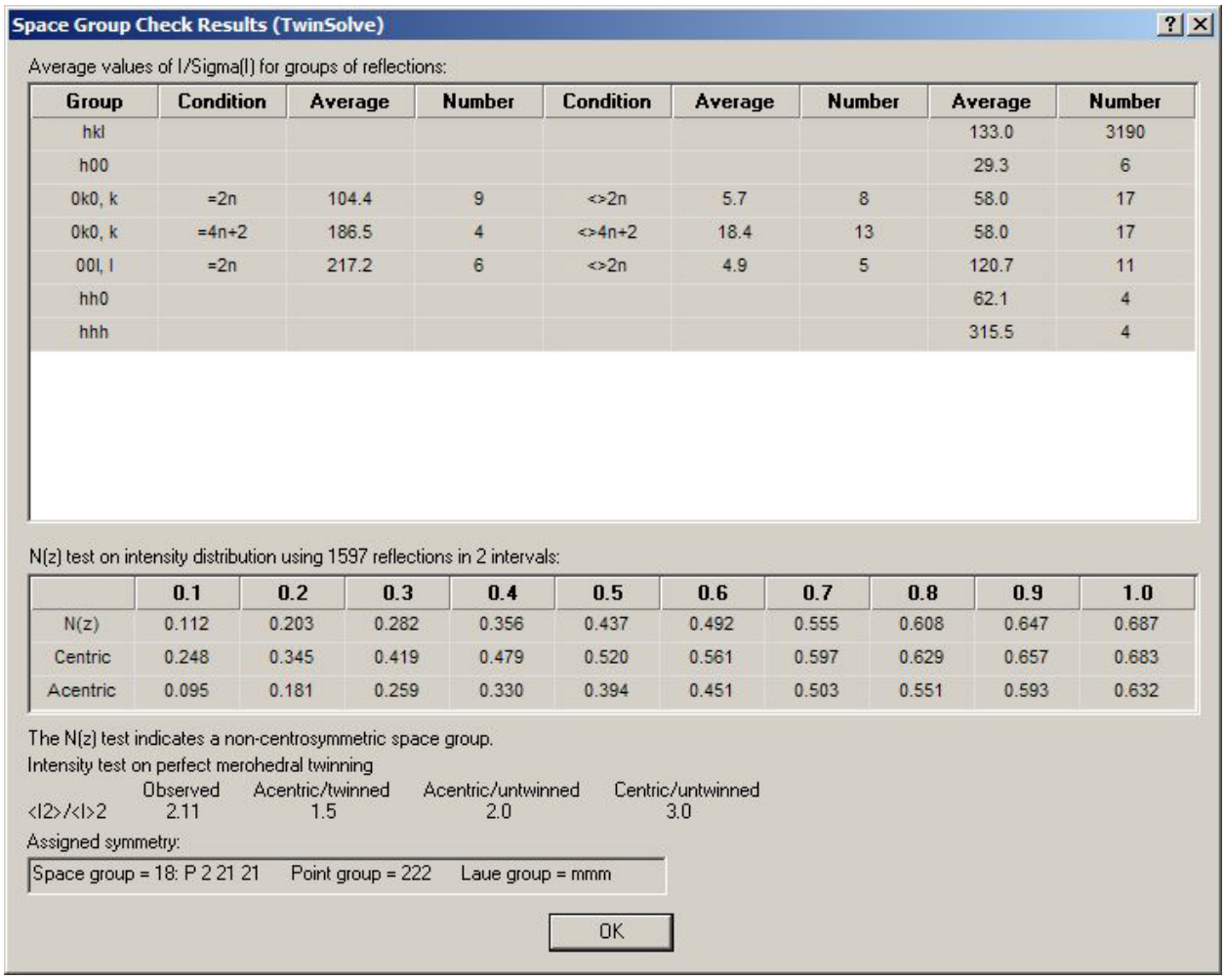
Space Group Check Dialog



Parameter	Description
Reflection file input	You can either **Use orientation reflections** (oriref.xor) or select one or more *.raw files from the list to use for the **Space group** step.
Data to merge	Reflection data is merged before the **Laue Check** is performed. The selection for **Data to merge** is either **All data** or **Use only non-overlapped data for twin**. If you chose this second option you must select a twin.
I/Sig tolerance	This is the minimum I/Sigma that a reflection must have to be included in the space group determination.

Space Group Check Results

After running the Space Group Check, you see the results in a table.



Space Group Check Results (TwinSolve)

Average values of I/Sigma(I) for groups of reflections:

Group	Condition	Average	Number	Condition	Average	Number	Average	Number
hkl							133.0	3190
h00							29.3	6
0k0, k	=2n	104.4	9	<>2n	5.7	8	58.0	17
0k0, k	=4n+2	186.5	4	<>4n+2	18.4	13	58.0	17
00l, l	=2n	217.2	6	<>2n	4.9	5	120.7	11
hh0							62.1	4
hhh							315.5	4

N(z) test on intensity distribution using 1597 reflections in 2 intervals:

	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0
N(z)	0.112	0.203	0.282	0.356	0.437	0.492	0.555	0.608	0.647	0.687
Centric	0.248	0.345	0.419	0.479	0.520	0.561	0.597	0.629	0.657	0.683
Acentric	0.095	0.181	0.259	0.330	0.394	0.451	0.503	0.551	0.593	0.632

The N(z) test indicates a non-centrosymmetric space group.

Intensity test on perfect merohedral twinning

	Observed	Acentric/twinned	Acentric/untwinned	Centric/untwinned
<I2>/<I>2	2.11	1.5	2.0	3.0

Assigned symmetry:

Space group = 18: P 2 21 21 Point group = 222 Laue group = mmm

OK

When you have reviewed the space group information click OK and this table is closed.

Scale and Average

The final steps in processing the data with TwinSolve through the CrystalClear interface include:

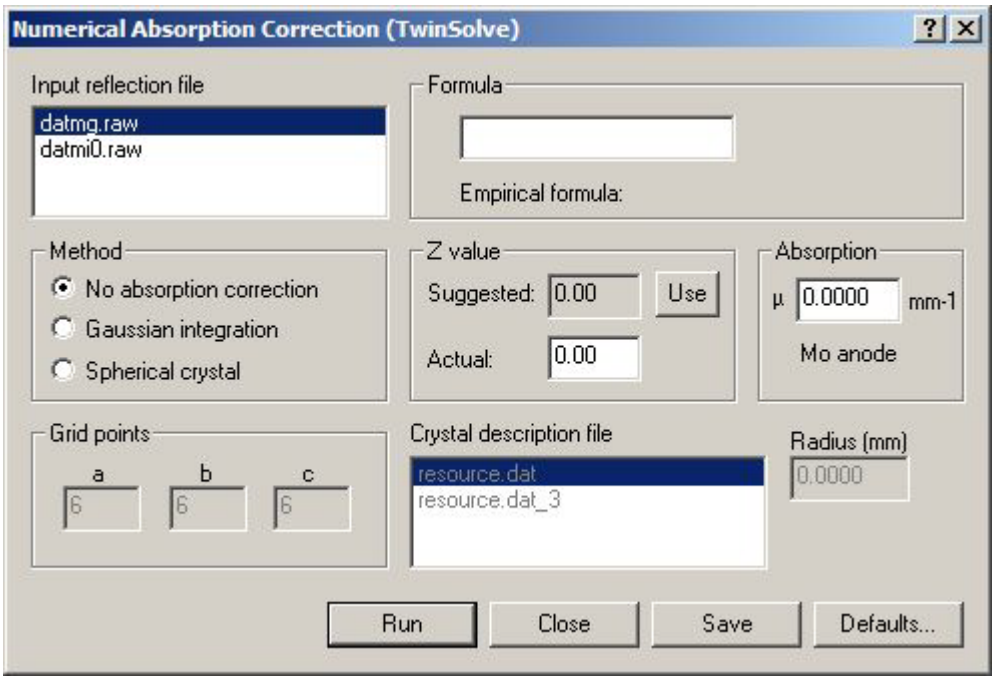
- **Num. Abs. Correction**
- **Scale and Shape**
- **Reject and Average**
- **Final Cell Refine.**

The Final Cell Refine is just another instance of the Refine step described in the **Refine Cell** step, so it is not documented separately in this section.

Num. Abs. Correction

TwinSolve has its own numerical absorption correction program which is run by this step.

Num. Abs. Correction Dialog



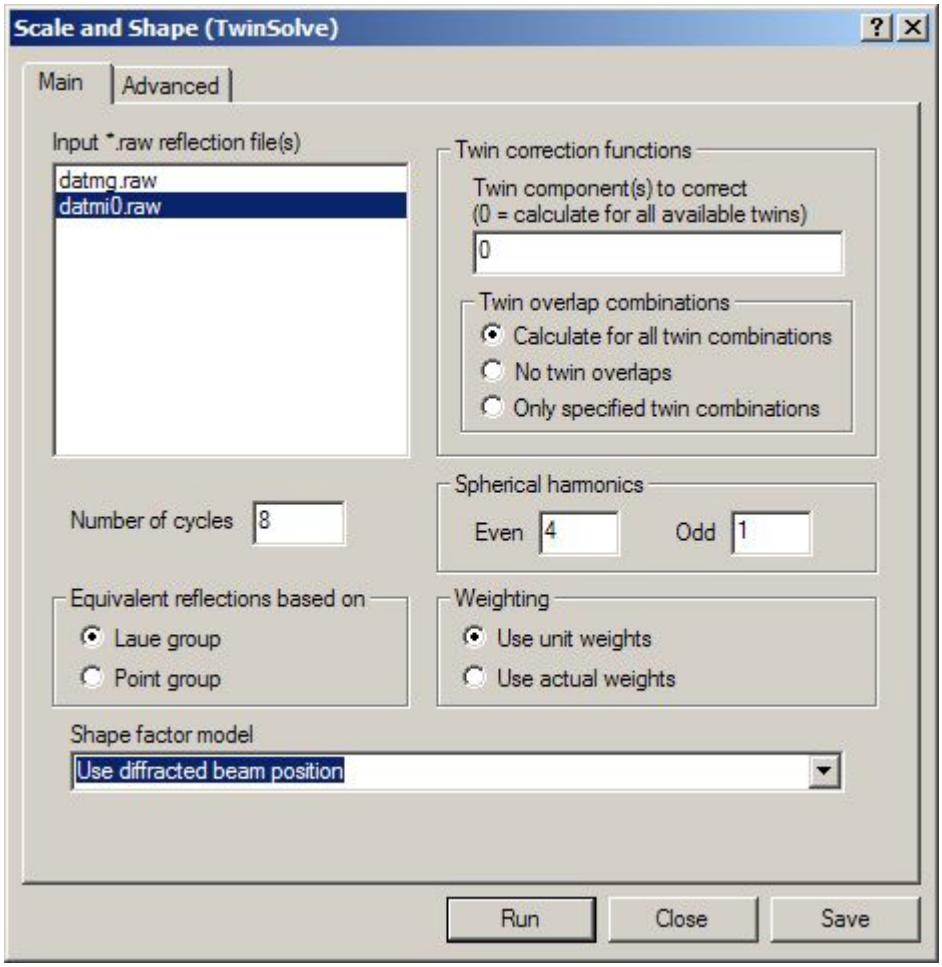
Parameter	Description
Input reflection file	For this step you need to select an *.raw file for input.
Formula	You need to enter a molecular Formula here if you have not done so in **Setup**.
Method	You have three options for the method of absorption correction to be used: • **No absorption correction** • **Gaussian integration** • **Spherical crystal**
Z value	A **Suggested** Z value is calculated based on the **Formula**. You have the option of using this (click **Use**) or entering another Z value in **Actual**.
Absorption	You can specify the **Absorption** coefficient mu in inverse millimeters.
Grid Points	For Gaussian integration you can specify the size of the grid used in the calculation.
Crystal description file	For Gaussian integration there is also a list of crystal description files from which to choose. You can create a description file by running RAXShape.
Radius	The **Radius** is used for the Spherical crystal option. It is in mm.

Scale and Shape

Scale calculates and applies scale factors to different batches of reflections in a reflection list, averages symmetry equivalent reflections, calculates merging and completeness statistics, and creates a reflection list of unique reflections.

The data set as specified is divided into scale groups based on the scan/batch number and the rotation angle value. Scale factors are then determined such that the deviations between intensities for equivalent reflections are minimized. Then these scale factors are used, and in the same way **Shape** factors (absorption anisotropy) are determined based on the directions of the primary and diffracted beams through the crystal. First the shape factors are applied and new scale factors are determined. The shape factors are expanded in terms of Cartesian spherical harmonics up to the order 8.

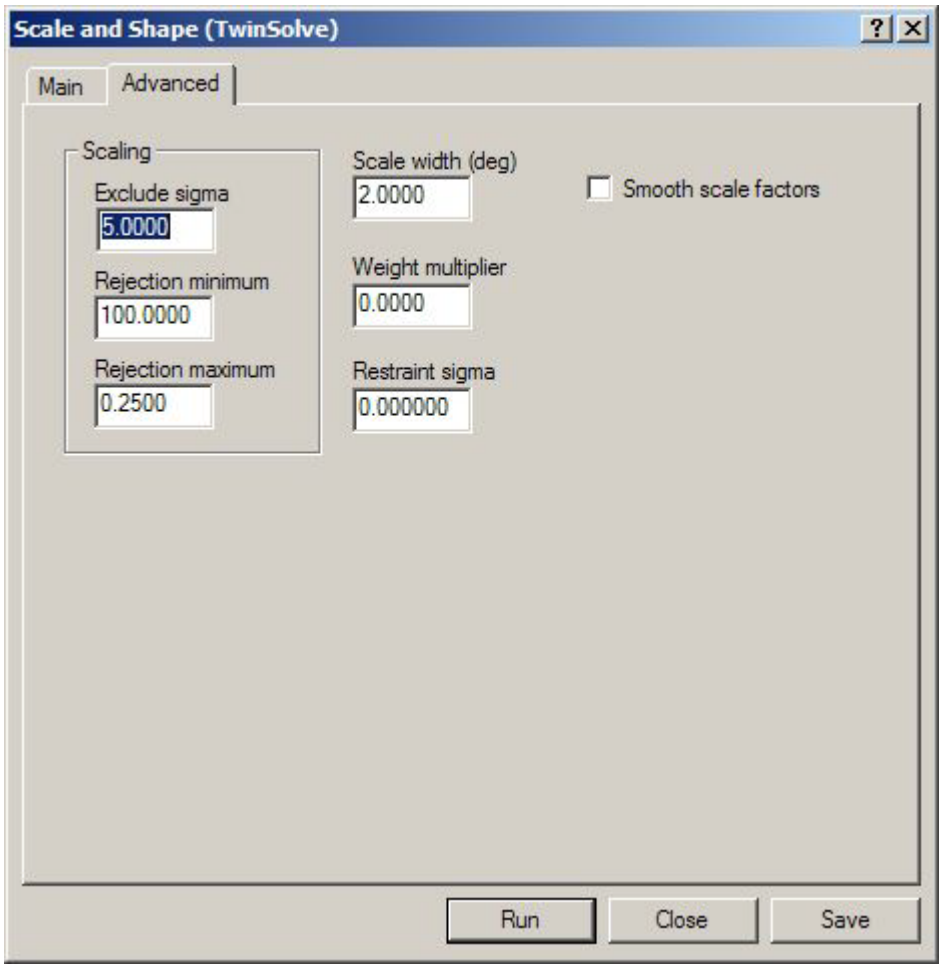
Scale and Shape Main Tab



Parameter	Description
Input *.raw reflection file(s)	For this step you need to select one or more *.raw files for input.
Number of cycles	The number of iterations in the scale and shape procedure.
Equivalent reflections based on	Scaling is accomplished by looking at equivalent reflection. These can be selected **by Laue group** or **Point group**.

Parameter	Description
Twin correction functions	You can specify the twin components that are used to calculate the scale and shape corrections. 0 means use all the available twins. Otherwise list them in a comma separated string (e.g., 1,3,4)
Twin overlap combinations	When comparing twin overlaps, the options are to: • **Calculate for all twin combinations** • **No twin overlaps** • **Only specified twin combinations.** The twin specification is taken from the edit box under **Twin correction functions**.
Spherical harmonics	These are defaulted to Even = 4 and Odd =1. They seldom need to be changed.
Weighting	The weighting options are: • **Use unit weights** • **Use actual weights**
Shape factor model	There are several options for the Shape factor model: • **Use diffracted beam direction** • **Use incident beam direction** • **Use vector perpendicular to scattering plan** • **Use scattering direction vector as reference direction** • **Average transmission for primary beam direction and diffracted beam direction**

Scale and Shape Advanced Tab

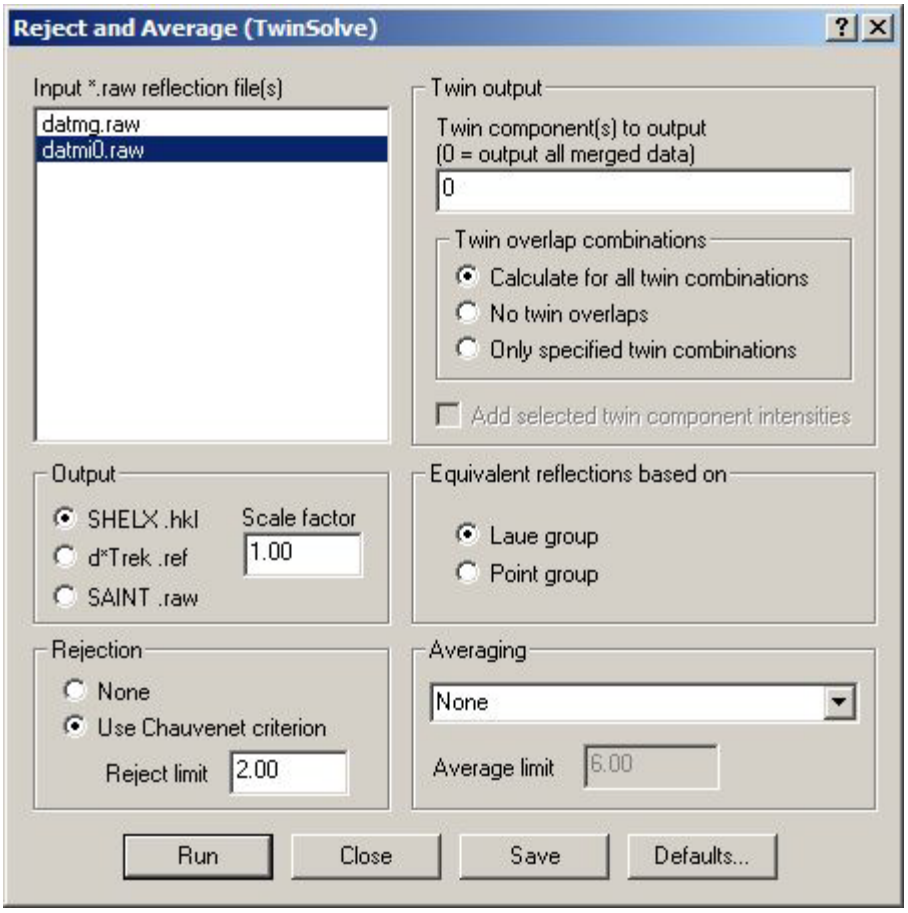


Parameter	Description
Scaling	The parameters that control scaling include: • **Exclude sigma**. The I/Sigma value below which reflections are not used to determine scaling. • **Rejection minimum.** • **Rejection maximum**.
Scale width	Set the **Scale Width** (in degrees) for batch scale factors.
Weight multiplier	The variance of I is increased by (multiplier * I)**2 upon output of each reflection.
Restraint sigma	Sigma of the least-squares restraint that the absorption anisotropy should be close to zero, that is the absorption factors should be approximately = 1.0. The default is 0.0001 (a value of 0.000 implies that the default is used). This restraint effectively constrains most of the correction to occur in the scaling rather than in the absorption correction.
Smooth scale factors	Constrains the scale factors to vary smoothly from group to group.

Reject and Average

Reject and Average is the penultimate step in TwinSolve processing which does the final averaging and writes the reflection files for use in structure solution.

Reject and Average Dialog



Parameter	Description
Input *.raw reflection file(s)	Choose one or more reflection files to **Reject and Average**.
Output	The output file formats supported include: • **SHELX.hkl** • **d*TREK.ref** • **Saint.raw** In addition you can specify a **Scale factor** which is applied to all reflections.
Rejections	There rejection options are: • **None** No reflections are rejected at this stage. • **Use Chauvenet criterion.** For this option you need to specify a rejection limit.
Twin Output	This allows you to select which twin components to include in the output file. 0 means all merged data, but you can also specify a list of twin components separated by commas.

Parameter	Description
Twin overlap combinations	When averaging reflections the following options are provided for handling overlapped reflections between combinations of twins: • **Calculate for all twin combinations** • **No twin overlaps** • **Only specified twin combinations** (taken from the Twin output list).
Add selected twin component intensities	Under certain conditions you can add intensities from overlapping twin compoenents.
Averaging	The options are: • **None** • **Use unit weights** • **Use 1.0/sigma(F2obs)**2** • **Use robust-resistant weights** • **Use robust-res.weights/sigma(F2obs)**2**
Average Limit	This is the limit to the number of reflections that can be averaged.

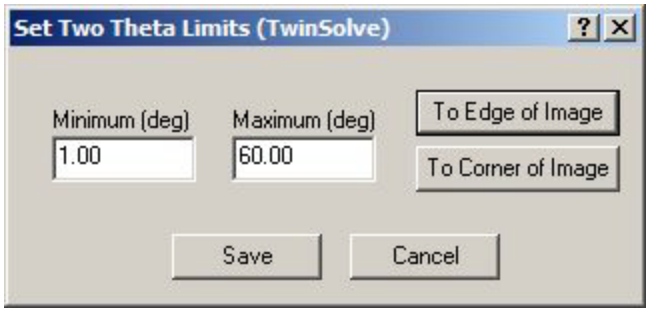
Menu Operations

The operations included in this section are accessible through the menu, but do not have corresponding steps in the flow bars so they have not been documented elsewhere.

Sample>Set Resolution

TwinSolve resolution is normally specified in degrees. If you are in TwinSolve mode, this function brings up a different dialog than is used by d*TREK. This is also true if you click the  icon in the main CrystalClear toolbar.

Set Two Theta Limits Dialog

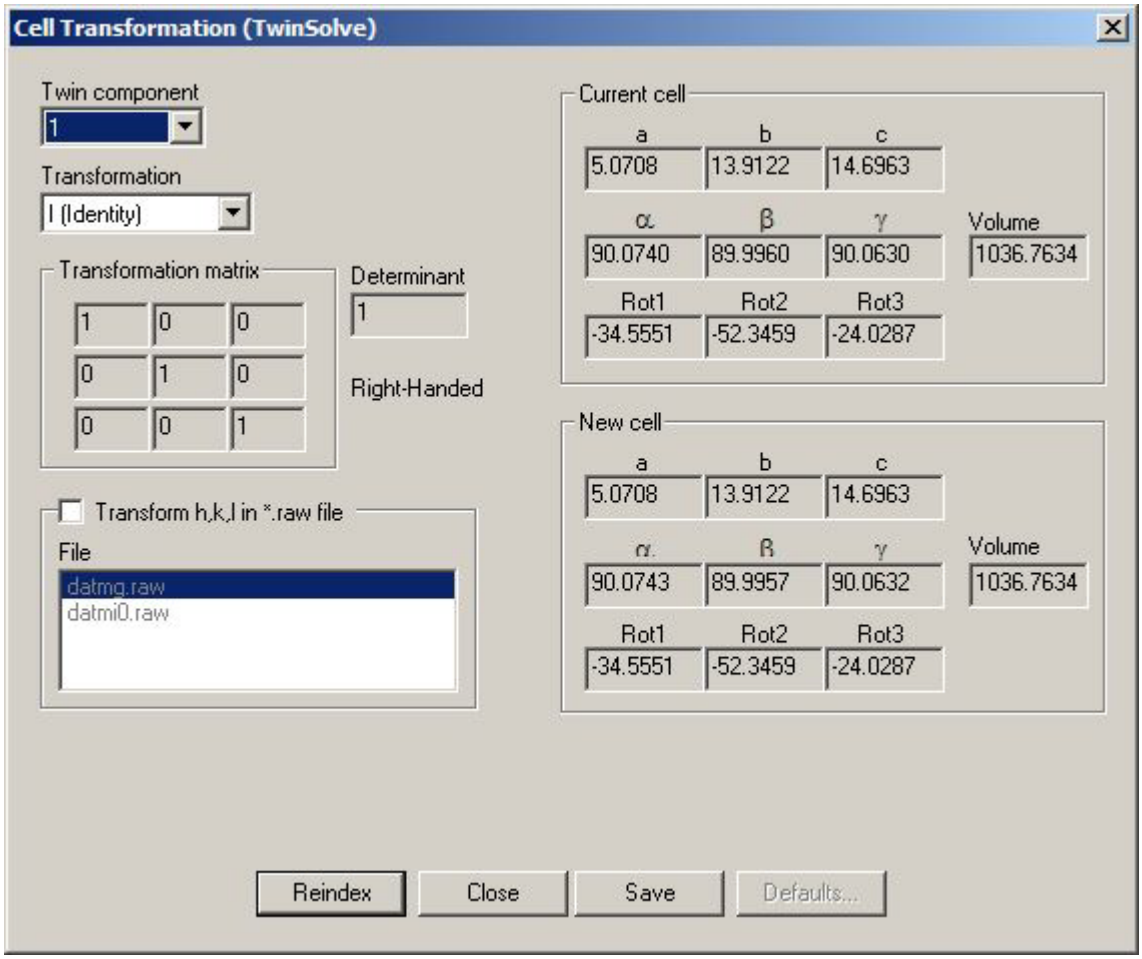


Parameter	Description
Minimum	Minimum resolution in 2 Theta degrees.
Maximum	Maximum resolution in 2 Theta degrees.
To Edge of Image	Set the Maximum resolution to the edge of the current image.
To Corner of Image	Set the Maximum resolution to the corner of the current image.

Cell Tools|Transform

The TwinSolve cell transformation has a very similar interface to the d*TREK command.

Cell Transform Dialog



Parameter	Description
Twin component	Each twin component must be transformed separately. Select the component you want to transform.
Transformation	Select the transformation you want to perform from the drop down list.
Transformation matrix	This is a read only display of the transformation matrix unless you choose **Custom Matrix** as your **Transformation** option. The **Determinant** and Right-Handed or Left-Handed are displayed but not editable.
Find Transformations	Click this button to compute the transform that would result in the lowest R-merge for the **Reflection** and the **Reference reflection** files. The results are show above this button and you can use the <> buttons to examine the ranked solutions.
Transform h,k,l in *raw file	If you want to transform the hkl's as well as the cell parameters, check this box and select the *.raw file that you would like to transform.
Current Cell	This is a read only display of the current cell.
New Cell	Unless you select **Custom Cell** as the option for the Transformation, this is just a read only display of the new cell. If you do select **Custom Cell**, you can enter the basic cell parameters.
Reindex	This applies the specified transformation to the cell and updates the reflection

Parameter	Description
	(*.raw) file if that option was selected.

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11. Reciprocal Lattice Viewer

The Reciprocal Lattice Viewer can be used view reflections in reciprocal space. This can help you decide if the crystal is twinned, whether your coverage is complete, and whether the indexing looks reasonable. For small molecules with twinning, the Reciprocal Lattice Viewer can be used in conjunction with TwinSolve to display the indexing for the various twin components. It is possible to index manually with the Reciprocal Lattice Viewer if automatic indexing fails.

Getting Started

The **Reciprocal Lattice Viewer** is When activated via the CrystalClear main toolbar icon  , you are immediately prompted to select a Reflection File Name (e.g., `dtfine.ref`). The **Reciprocal Lattice Viewer** uses the current cell parameters (including any twin information found by **TWINSOLVE**) when displaying lattices.

NOTE: Since **Reciprocal Lattice Viewer** uses **d*TREK** for its crystallographic functions, it is not able to process a reflection file that has more than one scan (e.g., from **TWINSOLVE**). However, it loads the reflection file you select and allows you the option of splitting it into a **d*TREK** compatible scans.

Toolbar

The **Reciprocal Lattice Viewer** functions are activated by clicking on the toolbar icons or by right clicking on a reflection.

Icon	Name	Description
	**Open Reflection File**	Read in a new reflection list.
	**Display Options**	Modify the rendering of reflections.
	**Color Options**	Set the color of reflections.
	**Select Reflections**	Select the reflections you want to view and optionally update the reflection file
	**Measure distances, angles, and volumes**	Measure distances angles, and volumes
	**Define Cell Planes**	Selects reflections in parallel planes for manual indexing
	**Cell Operations**	Modify the cell parameters.
	**Twin Operations**	Selecting the current twin component, creating and delete twin components, and various other operations.
	**Cell Display**	**Toggle** on or off for display and viewing control. (Default is on if cell definition is present). This button is for display and viewing control.

Icon	Name	Description
	**Viewing Transformations**	Set the viewing transform to set the view to: 1. Look down an axis 2. Look perpendicular to a plane 3. Apply a fixed rotation around an axis.
	**Rotate 90 degrees about screen X**	Rotate view 90 degrees in direction indicated by the arrow. This button is for display and viewing control.
	**Rotate 90 degrees about screen Y**	Rotate view 90 degrees in direction indicated by the arrow. This button is for display and viewing control.
	**Rotate 90 degrees about screen Z**	Rotate view 90 degrees in direction indicated by the arrow. This button is for display and viewing control.
	**Reset View**	Restores the initial view. This button is for display and viewing control.
	**Fine mode**	When this button is activated the screen controls have a finer resolution. This allows you to position the view that can be useful when trying to line up reflections. Clicking it again toggles to the off state.
	**Pick mode**	This button changes the Rotation Cursors to a Pick Cursor so you can rotate the display or select a reflection to be centered in the rotation; or to display its properties.

Cursor controls

Use the  Control Button to toggle between **Pick Mode** and **Rotation Mode.** When a normal arrow cursor is displayed, you are in a **Pick Mode**. When a special arrow cursor is displayed, you are in a **Rotation Mode**. Each mode has several options.

Pick Mode

When a normal arrow cursor is displayed, it indicates you are in **Pick Mode**.

Pick Mode Functions (Normal Cursor)	Description
Left-click reflection	Select reflection and perform operation active in current dialog. **NOTE**: Inactive unless a dialog is active.
Right-click reflection	Access menu with two options: **Set Center of Rotation:** Set reflection to be the center of rotation. **Report Properties:** Displays resolution, HKL, and other properties associated with the reflection.
Right click / drag.	Selects multiple reflections by dragging a selection rectangle over them (Only active when the interactive selection option is selected in an appropriate dialog.)
Left click blank screen / drag.	If you do not right click any object, the system reverts to transformation mode. All transformations active. Special cursors not displayed.

Rotation Mode

The special arrow cursors indicate you are in **Rotation Mode**. Viewing operations are activated by clicking and dragging. The operation performed depends on the area of the screen in which you first click, as shown in the table below.

NOTE: Right-clicking in **Rotation Mode** has the same effect as **Pick Mode**.

Special Cursors	Screen Location
	**XY Rotation** - Comprises middle 80% of display.
	**Zoom**- Comprises left 10% of display.
	**Y Translate** - Comprises right 10% of display.
	**Z Rotation**- Comprises top 10% of display.
	**X Translate**- Comprises bottom 10% of display.

How to Use the Reciprocal Lattice Viewer

This section gives short instructions for using the Reciprocal Lattice Viewer for several common tasks including:

- Edit a reflection file
- Edit cell parameters
- Manually indexing of a non-twinned sample
- Manual indexing of a twinned sample
- Examine HKL planes
- Use as a reflection viewer

NOTE: If using d*TREK for **Find Spots**, usually a **3D Find** is used for the **Lattice Viewer**. Perform **Find** on several images to provide enough three-dimensional information for automatic plane detection to be accurate.

Edit a Reflection File

Periodically indexing fails in the **Index Spots** operation of CrystalClear because of certain spots which appear on the image, but are not really valid reflections. While it is possible to edit reflections in the CrystalClear image viewer, it may be easier to see the problem in reciprocal space. Using the **Lattice Viewer**, you can edit a reflection file (*i.e.* dtfind.ref). This reflection file is created even when using TwinSolve.

1. Start the Reciprocal Lattice Viewer**.** The **Open Reflection List** dialog appears.
2. At the prompt to select a **Reflection File Name**, for this example select the dtfind.ref file.
3. Access the **Select Reflections** dialog using the  icon.
4. Edit the reflection list. The detailed instructions for doing this are found in the description of this dialog. It is often useful to **Delete Excluded** reflections as you go along.
5. Use **Write Selected** to write an edited file.
6. Exit **Lattice Viewer** by accessing the **Twin Operations** dialog using the  icon.

7. **DO NOT SAVE** when asked to save. Your reflection file has already been update and you do not want to pull any cell information from the Reciprocal Lattice Viewer.
8. Now try to index using the edited reflection file.

Edit Cell Parameters

If you suspect the cell parameters are wrong (e.g., one axis is twice as long as it should be), you can examine the quality of the indexing and adjust the cell parameters as needed.

1. Access the **Twin Operations** dialog with the  Control Button.
2. Set the **Indexing Tolerance** (Remember to **Index** again). If there is more than one **Component**, select the **Component** you want to look at.
3. Access the **Cell Operations** dialog with the  Control Button.
4. Adjust the Cell parameters. The Reciprocal Lattice Viewer re-indexes at each update so you can see how well the current parameters index.
5. Go back to the **Twin Operations** dialog.
6. Click **Save All** to send the **Current Component** cell information back to CrystalClear.
7. Continue working in CrystalClear with the new cell.

Manual Indexing of a non-twinned sample

If normal **Index Spots** with CrystalClear has failed, or has produced questionable results, you may try manual indexing using Reciprocal Lattice Viewer. If the crystal appears to be twinned, use the procedure for handling twinned crystals. If the crystal does not appear to be twinned, use the procedure as follows:

1. Access the **Define Cell Planes** dialog by using the  Control Button.
2. Click **Best Views** button. (This process may take some time.)
3. Select planes by stepping through the **Views** in the drop-down list until you find one that has parallel planes that you think may bound the cell.

NOTE: Planes may be found through two different methods: **(1) Best View/Best Plane** and **(2) Interactive Select**. You can use either method or mix methods.

4. Click **Best Planes** button. (This process also takes some time.)
5. Step through the **Best Planes** in the drop-down list. When you find the first plane that looks like it might bound the cell, click **Set A**.
6. Continue stepping through the planes until you find another set that looks promising, click **Set B**. You may have to go to another view to find Set B, but often you can find **A** and **B** from a single view.
7. Go back to **Best View** selection and continue stepping through the **Best Views** until you find another good view.
8. Click **Best Planes** again for this view and step through the planes until you find the final set of bounding planes and then click **Set C.**
9. Click **Cell From Planes** to do a **Cell Reduction**. Select the appropriate cell from the drop down list.
10. Access the **Twin Operations** dialog by using the  Control Button. Click **Save All** and then click **Exit**.
11. Continue working in CrystalClear with the new cell.

Manual Indexing of a twinned sample

If you suspect twinning, you can use the following procedure to manually index the twin components. This procedure is basically the same as for the non-twinned case, but it is repeated for each twin.

1. Access the **Define Cell Planes** dialog by using the  Control Button.
2. Click **Best Views** button. (This process may take some time.)
3. Select planes by stepping through the **Views** in the drop-down list until you find one which has apparent parallel planes.

NOTE: Planes may be found through two different methods: **(1) Best View/Best Plane** and **(2) Interactive Select**. You can use either method or mix methods.

4. Click **Best Planes** button. (This process may take some time.)
5. Step through the **Best Planes** in the drop-down list. When you find the first plane that looks like it might bound the cell, click **Set A**.
6. Continue stepping through the planes until you find another set that looks promising, click **Set B**. You may have to go to another view to find Set B, but often you can find **A** and **B** from a single view.
7. Go back to **Best View** selection and continue stepping through the **Best Views** until you find another good view.
8. Click **Best Planes** again for this view and step through the planes until you find the final set of bounding planes and then click **Set C.**
9. Click **Cell From Planes** to do a **Cell Reduction**. Select the appropriate cell from the drop down list.
10. Access the **Twin Operations** dialog by using the  Control Button.
11. Click **Remove** to remove the reflections that are indexed by the current component.
12. If there are still a significant number of reflections left, Click **Create New** to create a new component and go to step 3. Repeat for each twin component until there are no longer enough un-indexed reflections left to justify continuing.
13. When you are done, click **Save All** and then click **Exit**.
14. Continue working in CrystalClear (using the TwinSolve processing suite) with the new twinned components.

Examine HKL Planes

You can use **Lattice Viewer** to examine the **HKL** planes in your integrated data.

1. Access the **Select Reflections** dialog by using the  Control Button.
2. At **Property**, select **HKL.** Select **Include Only** as the **Property** function.
3. Enter the plane you want to view into the **HKL** parameters (eg. 0 * * for 0KL.) Click **Update Selection**.
4. Click **Delete Excluded**. This removes everything else so you can see the plane clearly.
5. If you want to look at another plane, click **Restore All**. This brings the reflections back. Now go to step 5.
6. When you are finished, click **Cancel** to exit **Lattice Viewer** without saving.

Use as a Reflection Viewer

The Reciprocal Lattice Viewer can be used as a diagnostic tool to view any reflection file (i.e. *.ref file) as long as there is some sample information (or a .head or image file) available. Sometimes it is just useful to look at a reflection file in reciprocal space to see what is going on. For example, you can look at dtintegrate.ref to determine your coverage of reciprocal space. Or you may look at a dtfind.ref to see if you can figure out why index is failing.

Operations

The Reciprocal Lattice Viewer is run through a series of dialogs that are activated through the toolbar at the top of the display

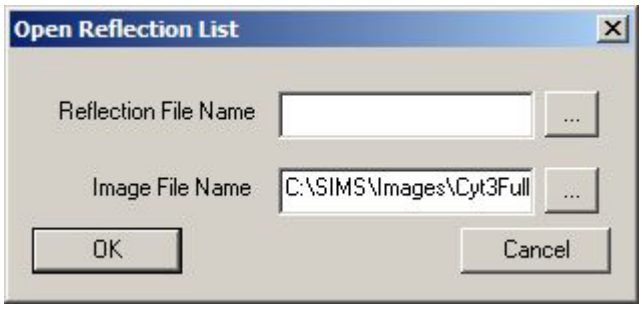
Open Reflection List


When you start up the Reciprocal Lattice Viewer you are prompted to select a reflection list. However, during the operation of the Reciprocal Lattice Viewer you can open a different reflection list and image file by clicking on this icon and selecting the files.

When you use the **Open Reflection List** dialog, you are no longer be able to save the results back into CrystalClear since they may not be compatible with the current sample.

The image file (you can, and usually should, use a header file) contains the current cell information for this sample. The Reciprocal Lattice Viewer displays this cell if there is crystal information the header. Otherwise the Reciprocal Lattice Viewer creates a default cell and turns the cell display off (10, 10, 10, 90, 90, 90).

Open Reflection List Dialog



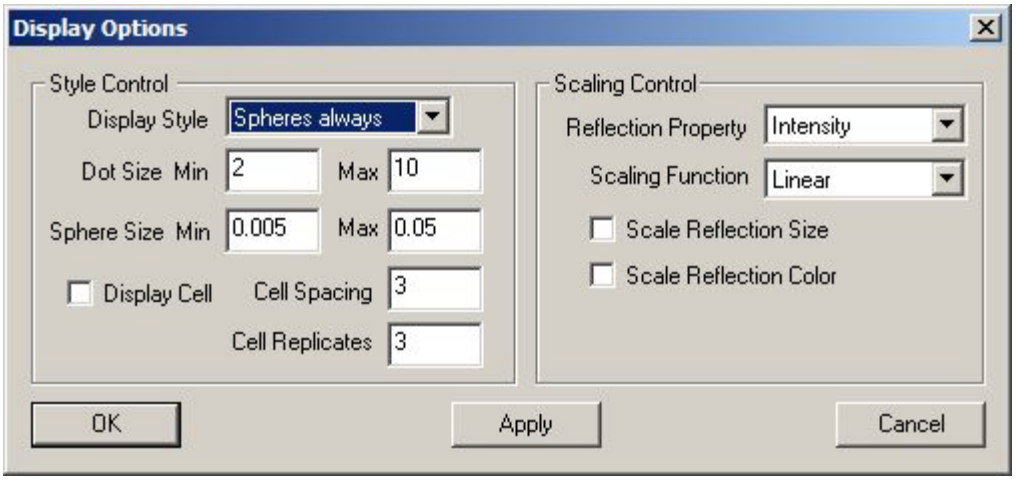
Parameters	Description
Reflection File Name	The reflection list (*.ref) to be displayed. Click the file browser button ... to locate the file. This file need not be in the existing sample.
Image File Name	Specifies a file from which to extract header information (*.head, *.img, or *.osc) Click the file browser button ... to locate the file. This should be from the same sample as the reflection file, but need not be the current sample.
OK Button	Accept new file and close dialog.

Display Options


The **Display Options** dialog allows you to change the display style of the reflections.

The axes are not transformed by any detector or cell transformations. The cell positions are calculated by multiplying a unit cell by the orientation matrix and then drawing them at various offsets according to the spacing indicated in the dialog.

Display Options Dialog



Control Groups		Description
Style Control		
	Display Style	Controls display of reflections as dots or spheres. • Dots on update • Dot Always • Spheres always If there are fewer than 50 reflections, the default is **Dots on Update**. If there are more than 50 reflections, the default is **Dots Always**. If you are drawing lots of spheres with slow redraw times, it is possible to lose mouse events. If you can't rotate for a while, just wait and let all the redraws process and it re-activates. If you have a fast computer with a good graphics card, you may want to select Sphere always since it is easier to see the reflections as spheres.
	Dot Size	Draw bigger dots if single pixel dots are hard to see. Dots are drawn at the minimum size if **Scale Reflection Size** is unchecked. **NOTE**: Large dots are not centered because the lower-left corner of the dot is at reflection position.
	Sphere Size	**Sphere Sizes** are in reciprocal space coordinates (*i.e.* small -- 0.005 is the default)
	Display Cell	Check to display cells, uncheck to remove cells. This can also be toggled by the **Cell Display** icon in the toolbar.
	Cell Spacing	Controls the spacing between the cells. There is an NxNxN lattice of cells.
	Cell Replicates	Controls the number of replicates (N above) for the cell display.
Scaling Control		
	Reflection Property	Scale the reflections on: • Intensity • I/Sigma

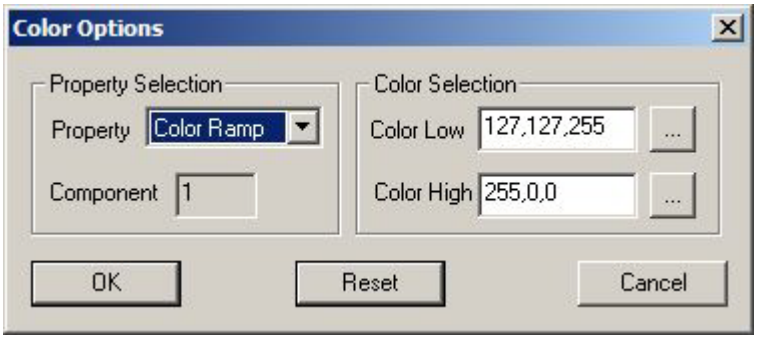
Control Groups		Description
	Scaling Function	The color and size scaling can be: • Linear • Square Root • Log 10
	Scale Reflection Size	**Scale Reflection Size** is useful for impressive pictures (in sphere mode), but sometimes makes alignments and other interactions more difficult.
	Scale Reflection Color	**Scale Reflection Color** applies both to spheres and dots. Default is neither size nor color used. This makes it easier to see what has been indexed (blue).
OK Button		Accept the new values and close dialog.
Apply Button		**Apply** the current selection and redraw the picture. It is best to use **Apply** rather than OK to experiment with settings until you get something you want to work with.
Cancel Button		Close the dialog. Any changes that have not been "applied" are lost.

Color Options

 The Color Options dialog allows you to change the colors in the Reciprocal Lattice Viewer. This is mostly used for presentation. The reflection colors carry meaning, so they should not normally be changed for day to day work.

The **Color Ramp** is the color that is assigned to selected reflections. This color may be scaled by the intensity, so a ramp (with a low value and a high value) must be specified.

Color Options Dialog



Control Groups	Description
Property Selection	

Control Groups		Description
	Property	Select the **Property** that is changed. The drop-down menu choices are: • Color Ramp (blue for the first component) • Rejected (fuchsia) • Ignored (green) • Unselected (dull yellow) • Unindexed (pink) **NOTE: Color Ramp** is used to show the select reflection property (e.g., I/Sigma) if **Scale Reflection Color** is the current display option. It only applies to the **Current Component**. When Scale Reflection Color is not checked, the **Color Low** value is used.
	Component	This read-only-control shows current **Component**. The Color Ramp applies to this component. **NOTE**: **Color Ramp** is used only for the **Current Component**. All other **Components** are drawn at their **Color Low** values.
Color Selection		
	Color Low **Color High**	RGB values (0-256 and comma separated) for the color. The browse button  brings up the Windows color dialog, which allows you to pick colors interactively. The display is redrawn when these values are changed.
OK Button		Accept the displayed values and close the dialog.
Reset Button		Restore the default values built into the program and redraw the display.
Cancel Button		Close the dialog with displayed values.

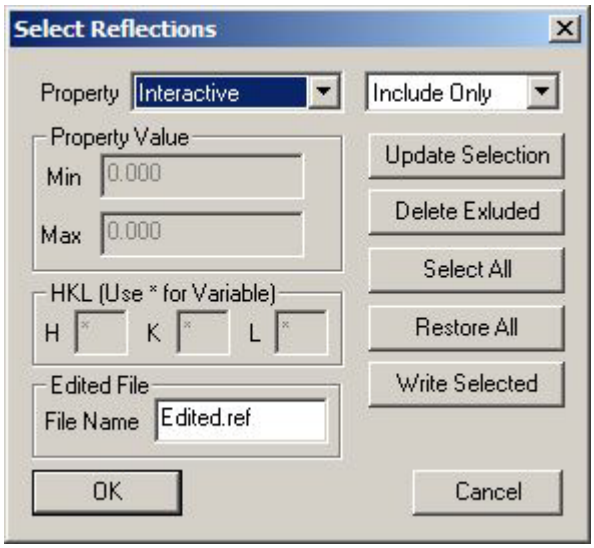
Select Reflections

 Many operations in the Reciprocal Lattice Viewer work on selected reflections. This dialog provides a very powerful set of tools for selecting reflections based on various criteria. This power is at the cost of some abstraction in the operations, so it is useful to read this description carefully and experiment with various selections to understand how this works. Unselected reflections are normally displayed in a dull yellow. Selected reflections are displayed depending on their status (indexed, rejected, etc.) Since the reflection status is color coded, it possible to experiment without actually performing the operations just by looking at what has been selected visually.

As an example if you want to exclude reflections within the resolution range from 2.5 to 2.4, you would:

1. In **Property** drop-down list, select **Resolution**.
2. Set **Min** to 2.5 and **Max** to 2.4.
3. In **Property function** drop-down list, select **Exclude**.
4. Click **Update Selection**.

Select Reflections Dialog



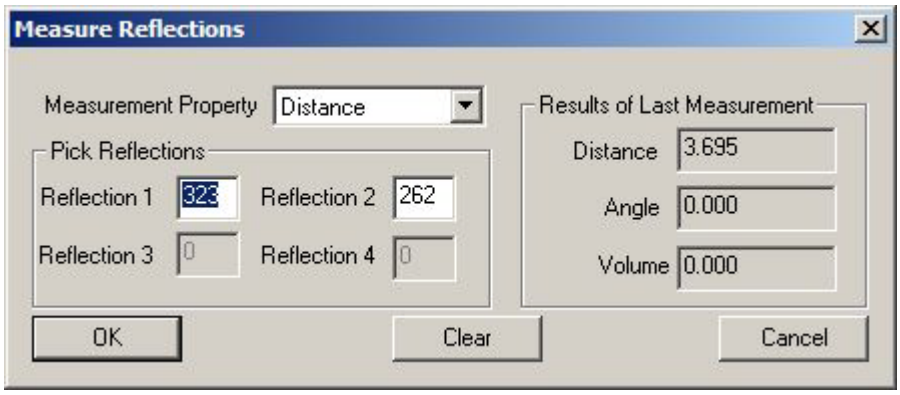
Control Groups		Description
Property		This specifies the property that is used to select the reflections to go into the selected reflections list. • **Intensity**. Absolute intensity of the reflection • **I/Sigma**. This allows you to remove weak reflections • **Resolution**. This allows you to remove high resolution reflections if they are causing problems. • **Phi**. This allows you remove a range of reflections based on the scan angle. • **Interactive**. When you select this option you should select the reflections you want by right clicking on the display and dragging out the reflections you want to select. You are dragging out a rectangular box, so it is best to orient the display so that the reflections you want to choose are in a horizontal or vertical line. • **HKL**. The most frequent use of this option is to view a plane in hkl space.
Property function		Each reflection has a status of selected or unselected. The unselected reflections are shown in dull yellow. The selected reflections are color coded depending on their status (indexed, rejected, etc). These functions specify how the currently selected reflections relate to the current status. When you start all reflections are selected.
	Include	Any reflections that meet the criterion are selected. Conversely, all those that do not match are unselected so they show up yellow. This does not change the state of any of the reflections which are not selected, so the effect is to add to currently selected reflections to the total list of selected reflections.
	Exclude	Change the state of selected reflections to unselected. This removes reflections from the currently selected list by marking them as unselected so that they show up yellow. In some cases, it is useful to use **Delete Excluded** button to remove the excluded reflection. In you need to you restore them later with **Restore All**
	Include Only	This first marks all reflections as unselected and then just selects those that meet this criterion. So this essentially starts from scratch.

Control Groups		Description
	Property Value	
	Mininum **Maximum**	Set the **Min** and **Max Property Value**. These are defaulted to the actual **Min** and **Max** values found when reading in the reflection list.
HKL (Use * for Variable)		
	H **K** **L**	These edit boxes are only enabled if the **Property** is **HKL**. Each box should have a valid index or an * to indicate that it is variable. For example you can select a plate by using 0,*,*.
Edited File		
	File Name	The **File Name** for writing the edited reflections. This file is stored in the current sample directory.
Update Selection		Updates the selection based on the value in the **Property function** drop-down. This is normal button to use as you build up the selection list by selected reflection operations.
Delete Unselected		Deletes unselected (dull yellow) reflections from the reflection list in memory. They are no longer displayed. If you need to restore them you can click the **Restore All** button.
Select All		Select all reflections (i.e., marks all reflections as selected).
Restore All		Return to original state of list when this dialog was opened. **NOTE**: The original reflections are lost if you close dialog using **OK,** then re-enter it.
Write Selected Button		Write selected (displayed) reflections into a file. **Edited File Name** is used. It is stored in the current Sample directory. **NOTE**: If the primary purpose is to edit the reflection list using **Lattice Viewer**, be sure to use **Write Selected** button to write the edited list.
Cancel Button		The **Cancel** button performs a **Restore All** before closing the dialog.
OK Button		**OK** keeps the current selection status of the reflections. **NOTE**: The original reflection list is lost if you close the dialog using **OK,** and then re-enter it.

Measure Reflections

 The dialog that pops up when you click **Measure Reflections** provides various tools for measuring distance, angle, and volume. The reflections used in the measurement are picked from the display by click them with the mouse. The distance and volume measurements are in real space in Angstroms. The angle measurements are in real space degrees.

Measure Reflections Dialog



Control Groups	Description
Measurement Property	The selected property is automatically calculated when all the reflections are defined. • **Distance** • **Angle** • **Volume**
Pick Reflections	
Reflection 1 **Reflection 2** **Reflection 3** **Reflection 4**	These values are the current indices of the reflections in the reflection list. They are useful as a visual feedback that a reflection has actually been picked.
Results of Last Measurement	
Distance **Angle** **Volume**	**Results of Last Measurement** of **Distance**, **Angle** and **Volume** are displayed here. The **Distance** and **Volume** are in real space Angstroms and the **Angle** is in real space degrees. The results are also displayed on the screen.
Function Buttons	
OK	Close the dialog.
Clear	Clears the values. Allows you to clean up the clutter from measurements.
Cancel	Close the dialog.

Define Cell Planes

 Visual or manual indexing is a process of finding bounding planes in reciprocal space. This is a two stage process:

1. Find a view where the reflections line up. Since the selection mechanism is a bounding box, the reflections should line up horizontally or vertically rather than at an angle.
2. Select reflections that form a bounding plane.

The Reciprocal Lattice Viewer provides two methods for manual indexing.

Method (1) - Best View / Best Plane

The first method for defining planes is a two step process. The first step is started by clicking on **Best Views.** This runs an algorithm which attempts to align reflections so that parallel planes are

clearly visible (i.e. perpendicular to the view). This process may take a few minutes for large reflection lists.

Select one of these views from the drop down list and, if the view looks promising, click **Best Planes.** This attempts to find sets of parallel planes in the selected best view. This process may also take a few seconds to run.

Then you can go through the **Best Planes** in the drop down list. They show up as red lines on the screen. If one of these planes appears to be a good candidate for a cell boundary, click **Set A** for the first set, **Set B** for the next set, etc. The current planes are drawn as green planes if only one set is defined (they look like lines straight on) or a parallelpiped which is either extend for two planes defined or the shape of the cell if all three planes are defined.

NOTE: You have to use at least two best views in order to get all three planes and not all views nor all planes are good candidates for indexing.

Method (2) -Interactive Select

The second method for defining planes is to select them interactively by dragging the right button of the mouse over a set of reflections that you think form a plane. Note that you don't have to select the whole plane all at once. You can select a few reflection and then add a few more. The selection accumulates until a **Set** button or the **Clear Selection** button is used.
Once a **Set** button is used, the Reciprocal Lattice Viewer attempts to find a plane that fits the selected reflections and a plane which is parallel to it.

NOTE: If only one or two images are selected for indexing, there is very little 3D information for finding these planes. As a result, the fit is sometimes not very good.

Mixing Methods for Defining Planes

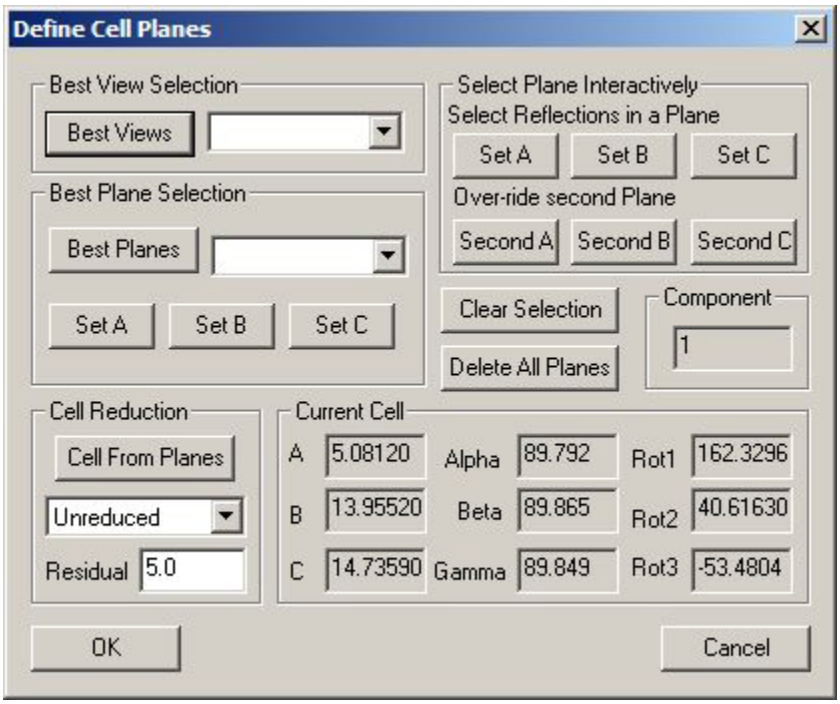
The two methods for defining planes can be mixed. For example you may define planes **A** and **B** with the **Best View / Best Plane**, and define **C** with **Interactive Select.**

Click the **Cell From Planes** button once the three planes are defined (green box on the display screen and distances between the planes are listed in the **A**, **B**, and **C** parameters for the cell). This runs a **Cell Reduction** algorithm and puts a list of possible cells into the drop down list. You can enter a different residual for the cell reduction if desired.

Once the desired cell has been selected, go to the **Twin Operations** dialog (rotated boxes) to either

- Create a new twin component, or
- Save your results

Define Cell Planes Dialog


Define Cell Planes
Best View Selection
Best Views
Best Plane Selection
Best Planes
Set A
Set B
Set C
Select Plane Interactively
Select Reflections in a Plane
Set A
Set B
Set C
Over-ride second Plane
Second A
Second B
Second C
Clear Selection
Delete All Planes
Component
1
Cell Reduction
Cell From Planes
Unreduced
Residual
5.0
Current Cell
A
5.08120
Alpha
89.792
Rot1
162.3296
B
13.95520
Beta
89.865
Rot2
40.61630
C
14.73590
Gamma
89.849
Rot3
-53.4804
OK
Cancel

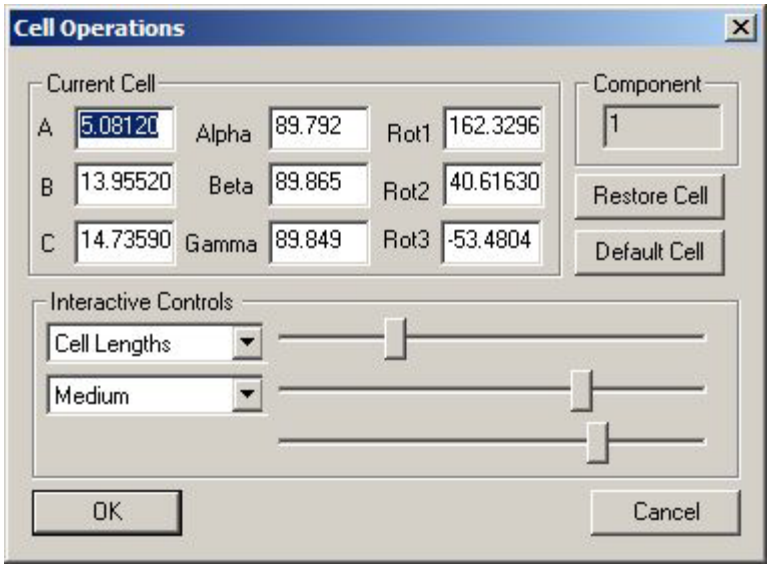
Control Groups		Description
Best View Selection		
	Best Views	**Best View Selection** attempts to compute ten **Best Views** for recognizing parallel planes in the reflection list. When these views have been calculated they appear in the drop-down list. The selected **Best View** from this list appears in the display.
Best Plane Selection		
	Best Planes	**Best Plane Selection** attempts to find ten **Best Planes** in the current view. The selected **Best Plane** appears in the display as red lines.
	Set A **Set B** **Set C**	Used to **Set A**, **B**, and **C** planes (respectively) from the current **Best Plane**. **NOTE:** You must define all three planes before using **Cell From Planes.**
Cell Reduction		
	Cell From Planes	**Cell From Planes** produces a list in a drop-down menu similar to the following: • Unreduced (blank) • Triclinic 1 • Monoclinic 2 • Orthorhombic 4 • Orthorhombic 5 • Orthorhombic 6 • Trigonal 7 • Tetragonal 8 • Tetragonal 9 • Trigonal 10 • Monoclinic 11 The selected **Cell Reduction** appears in the display as green lines. **Unreduced** displays blank.
	Residual	The **Residual** value is displayed.
Select Plane Interactively		
	Select Reflections in a Plane **Set A** **Set B** **Set C**	Used to **Set A**, **B**, and **C** planes (respectively) to define the selected plane.
Component		
	Component	Displays the current **Component** being indexed **NOTE:** This field is Read-Only. Refer to Lattice Viewer **Twin Operations** dialog to change to a new component.
Current Cell		
	A **B** **C** **Alpha** **Beta** **Gamma** **Rot1**	**NOTE:** These parameters are Read-Only. Go to the **Cell Operations** dialog to change controls. These controls show: • the **Current Cell**, • the **A**, **B**, and **C** values are the distances between the parallel planes if you are indexing Orientation Angles

Control Groups		Description
	Rot2 **Rot3**	
Clear Selection Button		Clears the reflections that have been selected interactively.
Delete All Planes Button		Deletes the current planes so that you can start over again.
OK Button		Close the dialog.
Cancel Button		Close the dialog. **Cancel** does **not** undo the cell definition.

Cell Operations

 The main purpose of the **Cell Operations** dialog is to review and make adjustments to the cell parameters. If you know cell parameters from other sources, you can just enter them directly. Or you can make minor changes to various parameters through interactive controls. For most manual indexing the **Define Cell Planes** dialog is much more powerful and easier to use.

Cell Operations Dialog



Control Groups		Description
Current Cell		
	A **B** **C** **Alpha** **Beta** **Gamma** **Rot1** **Rot2** **Rot3**	**Current Cell** values can be adjusted with the interactive controls or you can enter these values. In Windows fashion, these edits only "take" when the focus moves to some other control (*e.g.*, Tab, but not just Enter.). Orientation Angles
Component		
	Component	Displays the current **Component** being indexed

Control Groups		Description
		NOTE: This field is Read-Only. Go to the **Twin Operations** dialog to change to a new component.
Interactive Controls		
	Cell Lengths **Cell Angles** **Rotation**	Adjust the selected property values with sliders: Each adjustment has a **Medium**, **Coarse**, or **Fine** mode. **Fine** mode always centers each slider. **Medium** and **Coarse** modes use a constant scale for all three controls.
Restore Cell Button		Return to the cell which was defined when you opened this dialog.
Default Cell Button		Resets a 10, 10, 10, 90, 90, 90 cell.
OK Button		Close the dialog and update the cell with any changes that have been made.
Cancel Button		Close the dialog and do not update.

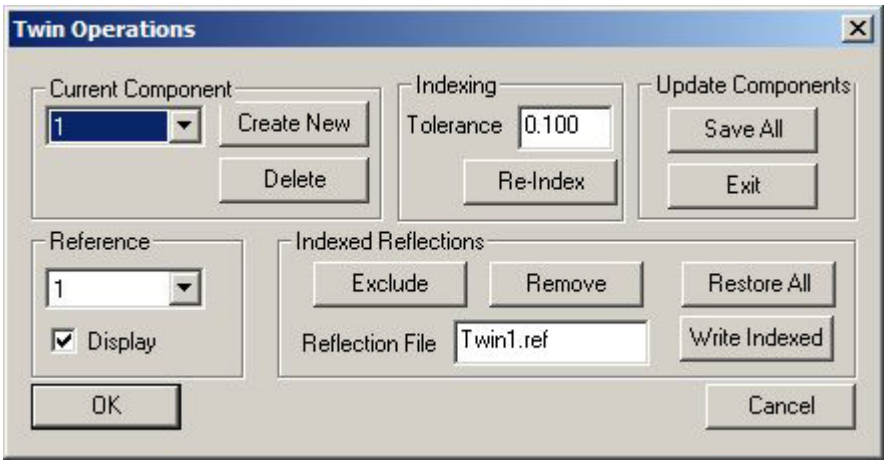
Twin Operations

 The **Twin Operations** dialog is used for selecting the **Current Twin Component**, creating and deleting twin components, and various other operations.

Twin Operations and **Cell Planes** are usually used in conjunction to index a set of twin components. The normal flow of interaction is:

1. Start up the **Lattice Viewer** from CrystalClear Toolbar.
2. Use the **Cell Planes** dialog to index some of the reflections.
3. Use the **Twin Components** dialog to remove the reflections that have been indexed.
4. If there are still significant numbers of un-indexed reflections, create a new component with **Create New** and go back to step 2 for the new component.
5. Otherwise use **Save All** to save the twin components definitions and refection lists back into CrystalClear and **Exit**.

Twin Operations Dialog



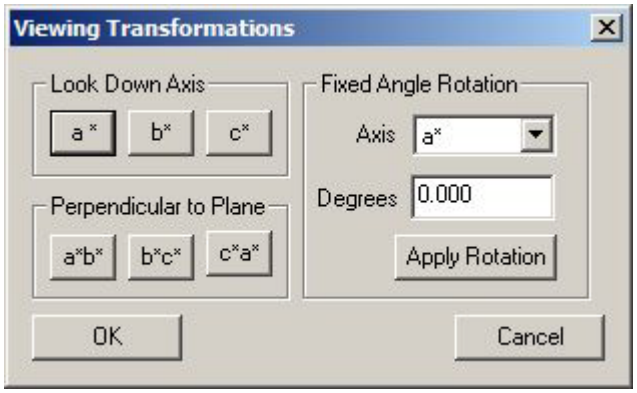
Control Groups		Description
Current Component		
	Current Component	Select the desired **Component** from the drop down list. The cells for the selected **Component** are displayed as soon as the **Component** is selected.
	Create New	Create a new **Component**.
	Delete	Delete the current **Component.** This may result in the renumbering of other **Components**.
Reference		
	Reference	The cell is displayed if it is different from the **Current Component** *and* the **Display** box is checked. For example you can use this to see the relationship between the current component and the first component.
	Display	**Display** check-box is used to display a different **Current Component**.
Indexing		
	Tolerance	Sets the **Tolerance** for indexing. Each reflection is **Indexed** to a floating point number. If the number is within an integer of this **Tolerance**, the reflection is considered to be **Indexed**. This value is global for all **Components**.
	Re-Index	This **Re-index**es the reflections for the **Current Component** using the new **Tolerance**. This is done using d*TREK indexing.
Update Components		
	Save All	Send the **Current Component** cell information back to CrystalClear and write a full reflection file with all reflections marked with the component which indexes them. The reflection file is called TwinAll.ref and is stored in the open sample. If you have used the **Open Reflection List** dialog to look at a different reflection file, you should not attempt to do a **Save All** to CrystalClear since this may not match the current sample.
	Exit	Exit **Lattice Viewer**. You can also exit by clicking on the X in the corner of the view. . If **Lattice Viewer** detects changes that may have been made, you are asked if you would like to **Save**. Only reply "**Yes**" if you have made changes to the **Twin Component** cells that you would like saved into the CrystalClear state for the current sample.
Indexed Reflections		
	Exclude	The **Indexed** reflection is drawn in the unselected color (yellow by default). This is useful if the current color selections are such that it is hard to see what has been **Indexed**. This is simply a display function and does not update any files.
	Remove	Currently, **d*TREK** algorithms work on the whole reflection list that is displayed (selected or not). Before you go on to the next **Component,** you should remove the reflections indexed by the **Current Component**. This gradually diminishes your reflection list, but when you update CrystalClear with the **Save All** button, the full reflection list with all components is sent back to CrystalClear.
	Restore All	Restore any reflections that have been removed, but only if the **Twin Operations** dialog has not been closed and re-opened. Once the dialog is closed, the reflections cannot be restored, but they are saved.
	Reflection File	Displays the **Reflection File** into which the indexed reflections are written.

Control Groups		Description
	Write Indexed	Write a reflection file with only those reflections that are indexed by the **Current Component**. The file name is given by **Reflection File** and it is stored in the currently open sample. It is not usually necessary to do this since the full reflection file is normally written by **Save All**, but it may be useful in some circumstances.
OK Button		Close the dialog with displayed values.
Cancel Button		Close the dialog.

Viewing Transform

 It is often useful to be able to view reciprocal space along an axis or perpendicular to a plane. This dialog gives you tools for setting the view. This just sets the current view to the orientation chosen. The view can still be manipulated interactively.

Viewing Transform Dialog



Control Groups		Description
Look Down Axis		
	a* **b*** **c***	Orients the view looking down the selected axis.
Perpendicular to Plane		
	a*b* **b*c*** **c*a***	Orients the view looking perpendicular to the plane.
Fixed Angle Rotation		
	Axis	Rotates the view a specified amount about the selected axis. • a* • b* • c*
	Degrees **Apply Rotation**	Enter the desired value in **Degrees**, then click **Apply Rotation**. The display refreshes.
OK Button		Closes the dialog.
Cancel Button		Closes the dialog.

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12. Tools

The tools described in this section are accessed through the menu. For this reason the descriptions of these tools are organized by their location in the menus. Functions and dialogs that have already been described previously (e.g., in the chapters on Data Collection, d*TREK Processing, etc) are not included in this section. The tools that are included in this chapter include:

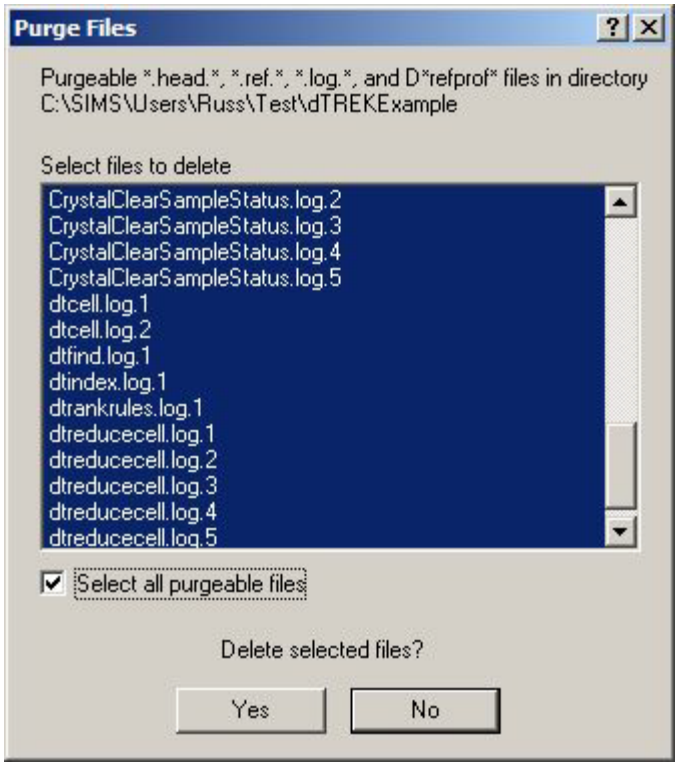
- **File Menu**
  - **Purge Files**
  - **Utilities**
    - **Write Sample**
    - **Write d*TREK header**
    - **Read d*TREK header**
    - **Write PROCESS Resource**
    - **Read PROCESS Resource**
    - **Change Image Directory**
    - **Prepare Bug Report**
    - **Write CIF**
    - **Determine Structure**
    - **Move User Data Directories…**
    - **Prepare Structure Determination Area**
  - **Info**
  - **Print**
- **Edit Menu**
  - **Header/Database editor**
- **Sample Menu**
  - **Search NIST database**
  - **Search Cambridge Structural Database**
- **Instrument Menu**
  - **Compute Axial Photographs**
  - **RAXVideo Version**
- **Tools**
  - **Preferences**

File Menu

File>Purge Files

Various files kept in the CrystalClear sample directory are versioned either with a *.1, *.2, etc or with an *_1.*, *_2.* etc. For example if you have run index 3 times, you have dtindex.log, dtindex.log.1 and dtindex.log.2. The most recent file is dtindex.log. The second most recent is dtindex.log.1, etc. If you do a lot of work in a sample, these files can mount up. This function helps you purge them. This applies only to the currently open sample.

Purge Files Dialog



Parameter	Description
Select files to delete	The files in this list is all the files that can be deleted without losing any current information. If **Select all purgeable files** is checked then all the files are selected. Otherwise you can select the file(s) to be deleted using standard Windows file selection methods.
Select all purgeable files	If this is checked all files in the above table are selected.

File>Utilities>Write Sample

The sample database files (e.g., <sample>_20.ho and <sample>_20.xml) are updated after each step, but certain information (e.g., any updates to the current dialog) are kept in memory. You are asked if you want to save the sample whenever you exit CrystalClear or start a new sample. You can also save the sample information by using the Save Sample icon or through the Write Sample function in the Utilities sub menu.

The sample information is written in a <sample>_version.ho file and a <sample>_version.xml file. Version is the CrystalClear version (e.g., 14, 20, etc). For example if your sample were MyTest, the file names would be MyTest_20.ho and MyTest_20.xml. If at some time you upgrade to version 2.1 of CC, then the first time you write a new sample it would be MyTest_21.ho and MyTest_21.xml. Since the format of these files is dependent on the CrystalClear version, this allows you to revert to an earlier version of CrystalClear without losing all the sample information. You will, however, lose any changes made in the new version if you revert to a previous version.

File>Utilities>Write d*TREK Header

d*TREK keeps a good deal of state information (e.g., the current cell) in header files. These are normally updated automatically as needed by CrystalClear, however, you can write the current header information into an *.head file with this function. You are provided a full file browser dialog, so this file can be stored anywhere.

File>Utilities>Read d*TREK Header

It is also possible to read a header file and update the state information from its contents. This is primarily used for diagnosing problems and is only recommended if you know a lot about both d*TREK headers and CrystalClear.

File>Utilities>Write PROCESS Resource

Fine Slice PROCESS (FS_PROCESS) keeps its state information in a file called resource.dat. Again CrystalClear normally creates, updates, and reads this file as needed, but you can store the file explicitly using this function.

File>Utilities>Read PROCESS Resource

Reading an external resource.dat updates the state information and is recommended only if you are in expert in FS_PROCESS and CrystalClear.

File>Utilities>Change Image Directory

The sample keeps an absolute link to the directory where the images are stored. This is by default ./Images, but can be anywhere else. If you need to move the images for some reason, you can either create an entirely new sample (usually the best option) or change the image directory in the sample that references these images. Changing the image directory removes all the state information, so you have to reprocess (Find, Index, Refine, etc) the sample once you have made the change.

File>Utilities>Prepare Bug Report

This function gathers up the log files that are needed for diagnosing a bug so that you can email it to your Rigaku support person. Many organizations prevent you sending emails outside the firewall with an smpt based email server like the RMailer server that CrystalClear uses, so you may have email this information to yourself and then forward it to Rigaku from your account.

File>Utilities>Write CIF

The flow bars for small molecule processing automatically write CIF files, but it may be useful to write it explicitly at some other stage. CrystalClear first checks to see that it has all the information it needs to write the CIF file.

File>Utilities>Determine Structure

This is a small molecule feature only. It starts up CrystalStructure and passes it information from the current sample. It is the equivalent of the **Solve** button in the small molecule flow bars. Once CrystalStructure has been started, you should switch to that interface to continue your processing.

File>Utilities>Move User Data Directory

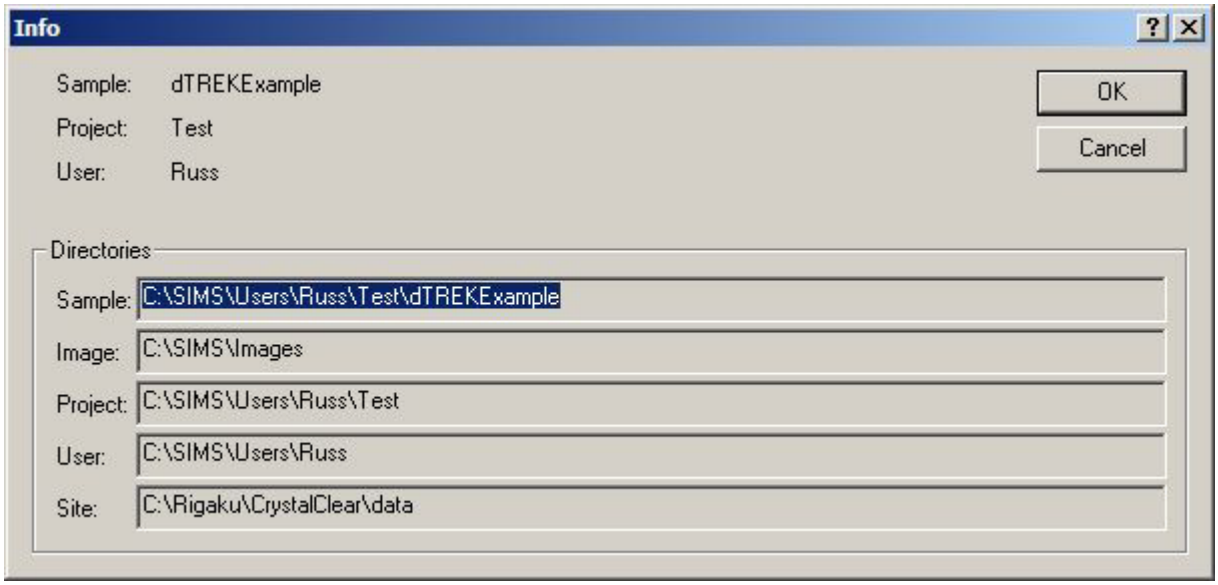
This is documented in Chapter 2 under User Administration.

File>Utilities>Prepare Structure Determination Area

For small molecule work, the flow bars normally create a .\Structure directory where the CIF and other necessary files are stored. The same files are created in this Structure directory regardless of the processing suite used to process the data. This function allows you to create the Structure directory and copy the files into it manual. This is not normally needed, but could be useful at times.

File>Info

This creates a table which shows the current sample name and the directories where information is kept.



FilePrint

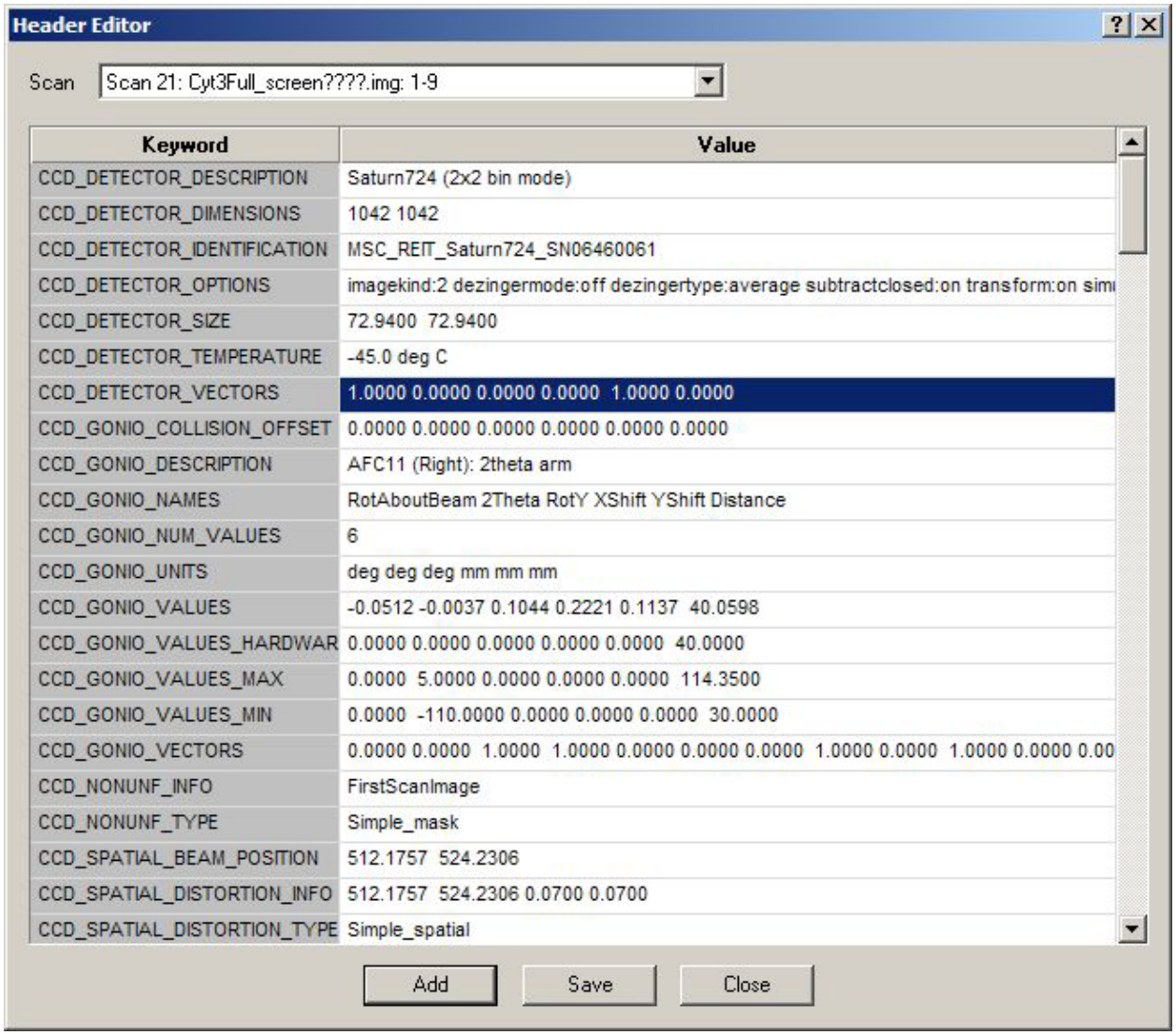
This prints the current image from the **Image Display**.

Edit Menu

Edit>Header/Database Editor

This function creates a new input.head based on values extracted from the images and from the existing input.head. It is normally used to fix problems found in the image headers or other processing problems. It does not update the images themselves, but by updating these values in input.head it can override the values in the image headers and thus correct a problem that would prevent processing the data. This is only used in d*TREK mode and is intended for expert users.

Header Editor Dialog



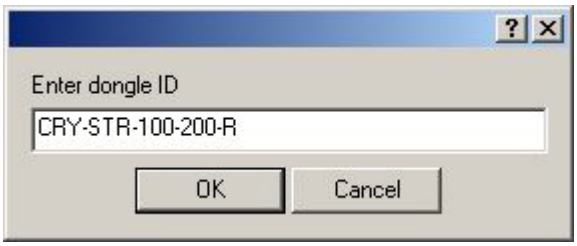
Parameter	Description
Scan	The first image from the scan is used to populate the fields of the header that come from the images.
Header Table	A d*TREK header is made up of **Keyword Value pairs**. Some of these describe the instrument, the data collection conditions, and other properties of the image itself. But this header is also used to pass state information from one process to the next within d*TREK. For example the CRYSTAL_UNIT_CELL might be added by Index Spots (dtIndex) and used by Refine Cell (dtrefine). You can double click a value to edit it. You cannot edit **Keywords**, but you can **Add** a new **Keyword** and then give it a **Value**. Extreme care should be taken in editing headers and you should only attempt it if you an expert in both d*TREK and CrystalClear.
Add	Add a new **Keyword Value** pair. This brings up a simple dialog to allow you to enter these text strings. The **Keyword** should be in all capitals with connecting underscores. The value can be any space separated string.

Parameter	Description
Save	This writes the current file to input.head.
Close	If changes have not been saved, you are reminded to do this before you close.

File>Edit Dongle ID

CrystalClear is currently licensed through a dongle. You normally enter the dongle ID the first time you run CrystalClear after it has been installed. But if you change dongles, or need to convert a demo version to a licensed version, you can use this function to enter the new dongle number.

Enter Dongle ID Dialog



Parameter	Description
Enter dongle ID	The dongle ID is usually printed on the dongle or in documentation sent to you with the dongle.

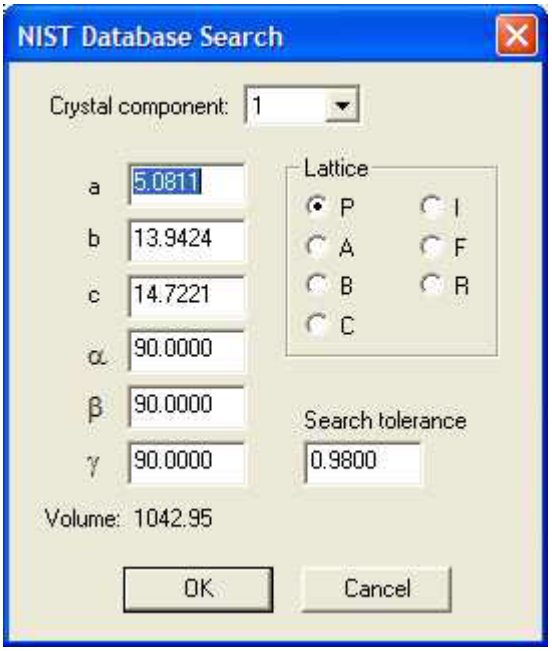
Sample Menu

Most of the items in the sample menu have been dealt with elsewhere since they appear in the flow bars. However, there are two commands for searching small molecule databases that have not been covered.

Sample>Search NIST Database

This function displays the **NIST** dialog, which allows you to search the database. This is sensitive only when the database is available (i.e. mounted in the CD ROM drive.) The **NIST** environment variable must also be set to the directory in which the database files are contained. You must also have the **nbsreduc.exe** which is distributed with CrystalClear, but is not useful without the database itself.

NIST Database Search Dialog

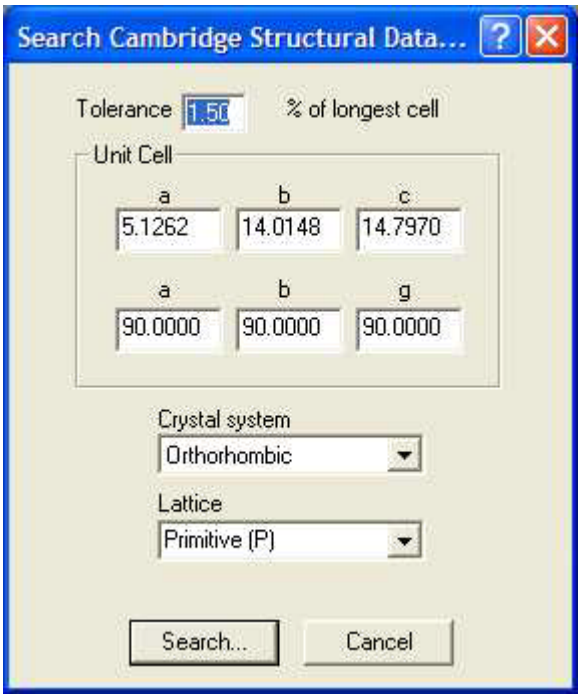


Parameter	Description
Crystal component	For d*TREK and FS_PROCESS, this is always 1, but for TwinSolve may be something else.
{Cell Parameters}	The current cell parameters are taken from the sample. You can modify them if you need to.
Lattice	This defaults to the current lattice from the sample, but other selections are possible.
Search tolerance	This is how close to the cell in the database you need to get before a match is declared. It is a fraction of 1.0 and should normally be close to 1.0. The default is 0.98.
OK	When you click OK this starts up nbsreduce.exe and performs the search, but only if you have the NIST database properly installed.

Sample>Search Cambridge Structural Database

This function brings up a dialog to allow you to enter parameters for a search of the Cambridge Structural database. The sample must have at least been indexed before you can run this command.

Search Cambridge Structural Database Dialog



Parameter	Description
Tolerance	This is the percentage that you are allowed to deviate from the cell parameters and still be considered a match. It is relative to the longest cell dimension.
Unit Cell	This is automatically filled in from the current cell parameters, but you can modify these values if you need to.
Crystal system	This is the current **Crystal system** for this sample. You can select a different crystal system to search for.
Lattice	The current **Lattice** which can also be changed.
Search.	When you click search, CrystalClear starts up an application from the Cambridge Crystallographic Structural Database group called ConQuest. Conquest must be installed on your computer. The ConQuest is not included in CrystalClear and must be obtained from the CCSD group.

Instrument Menu

The more common functions in this menu have icons in the main toolbar and have been described in chapter 4. The remaining functions are used less often and accessible only through the **Instrument** menu.

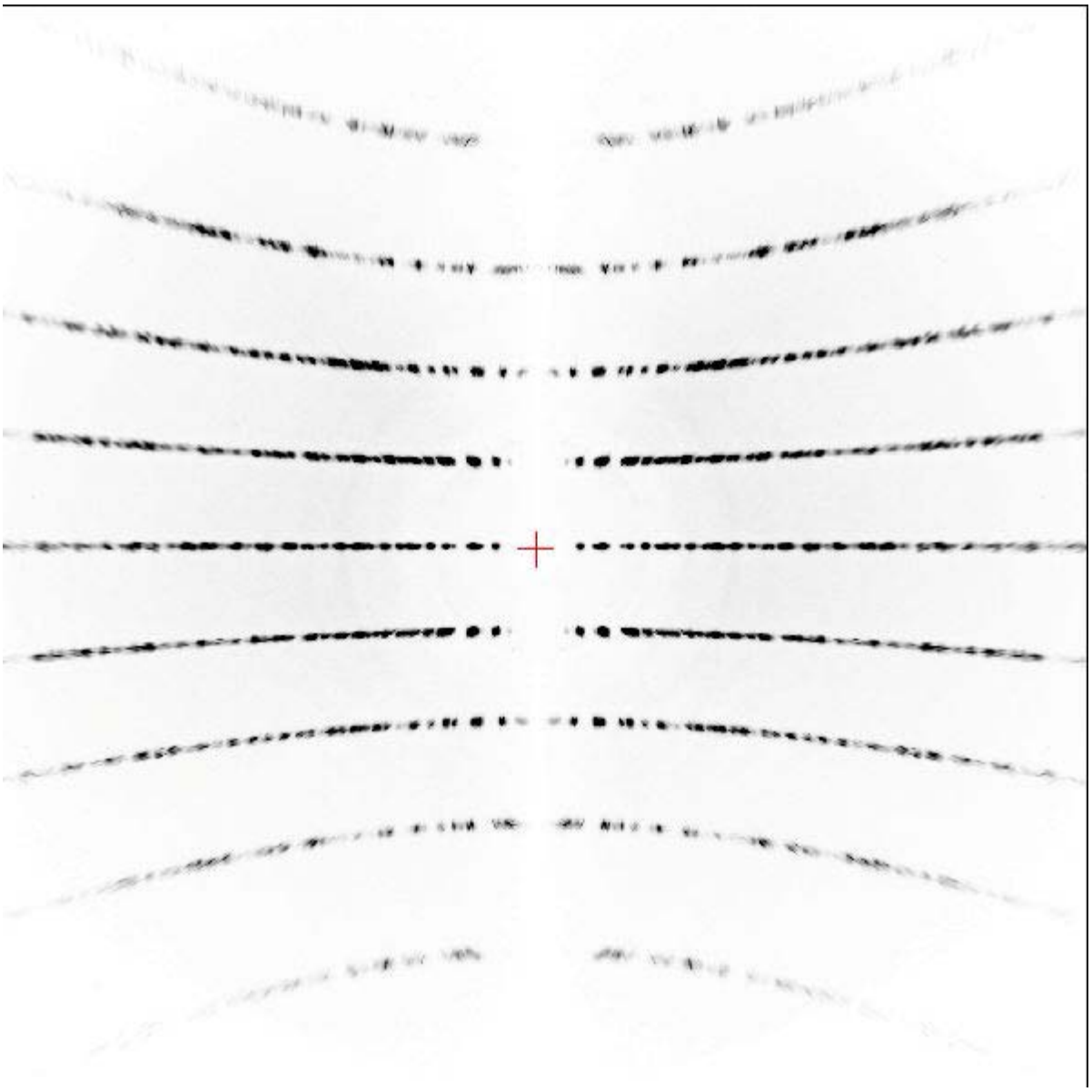
Instrument>Evaluate Exposure Time

This feature is still under development and should not be used at this time.

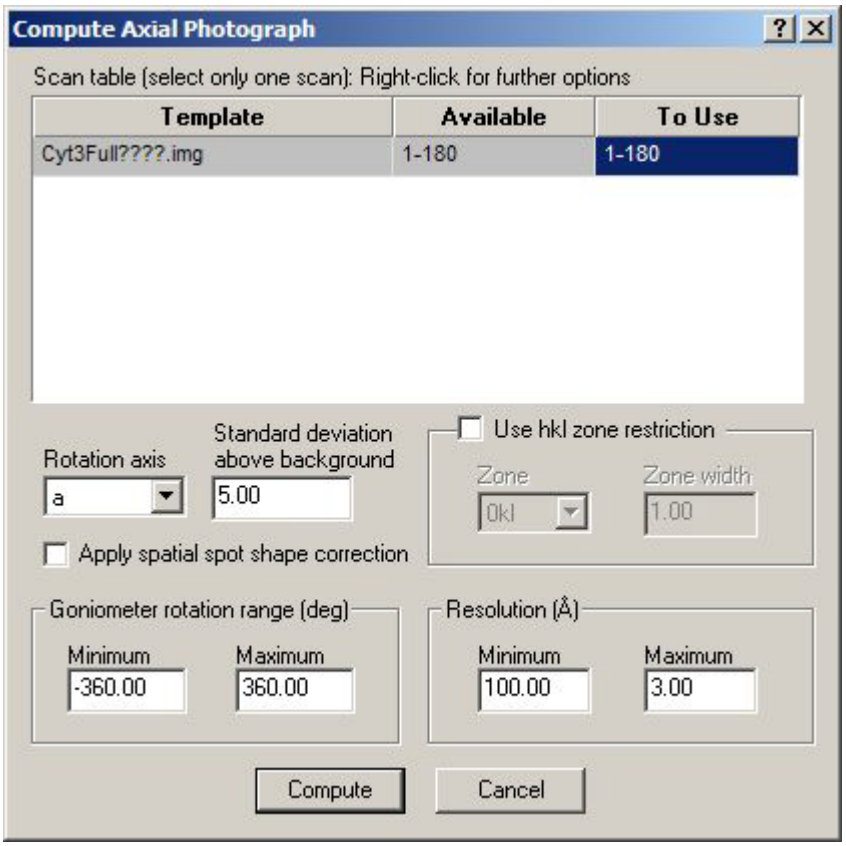
Instrument>Compute Axial Photograph

Once the cell constants for a crystal are known, the **Compute Axial Photograph** menu function should be used to collect an axial photograph to (1) confirm lattice spacing and cell centering, (2) help determine

the **Laue** group and (3) provide visual indication of crystal quality. A typical axial photo is shown below.



Compute Axial Photograph Dialog



Parameter	Description
Scan table	This is a standard scan table to allow you to select the images that you want to use for generating the axial photograph.
Rotation axis	The photograph is computed to view down the Rotation axis that you choose. (a ,b, or c in reciprocal space)
Standard deviation above background	Reflections with a lower I/Sigma are not shown in the axial photograph.
Use hkl zone restriction	If you check this box, you can select a specific **Zone** and **Zone width** (in hkl space) for selecting reflections.
Goniometer rotation range	You can limit the rotation range so that only reflections within the specified range are included. The range is in degrees and refers to the scan axis for the scans (e.g., omega).
Resolution	You can limit the resolution of the reflections to be included.
Compute	This generates the image which is stored in the sample directory (not the image directory) as dtaxial_x.img where x is the rotation axis. This computation can take a while for a full data set.

Analyzing the Axial Photograph

Lattice spacing

Confirmation of lattice spacing is especially helpful when perhaps one "satellite" reflection has caused the doubling of an axis, or when a weak super-lattice exists.

Confirmation of cell centering also may be useful in some cases. For example, a C-centered cell could be confirmed with a photo of [110], which (due to the equivalent point in the face center) would indicate a spacing equal to half of the length of the face diagonal.

Calculation

$d = (n\lambda)/\{\sin(\tan\text{-}1[Y/R])\}$

where d is the lattice spacing, n is the number of the layer line (center line is 0), λ^2 is the wavelength, Y is the distance from layer line n to layer line zero, and R is the crystal-to-detector distance

Symmetry:

An X-ray image contains all of the symmetry elements that the incoming X-ray beam has traversed. So, if there is a mirror plane perpendicular to the axis being photographed, the photo contains mirror symmetry. This can be used to verify the Laue group, which is especially useful for tetragonal, trigonal, hexagonal and cubic groups (for which there are two possible Laue symmetries).

Systems which, by accident, appear to have higher symmetry (monoclinic with beta = 90°, orthorhombic with a=b, etc.) are easily detected by this method.

Furthermore, observation of "impossible" combinations of mirrors -- e.g., monoclinic or orthorhombic with two mirrors -- probably indicates a twin.

NOTE: **Axial Photographs** only indicate symmetry elements that the beam has traveled along --- so you see the mirror if the beam goes along a mirror. If the axis being photographed is, say, a four-fold axis, you don't see four-fold symmetry in the photo because the x-ray is traveling perpendicular to, rather than along, the axis.

Crystal quality

This is always useful for determining whether to remove a crystal on the grounds of "bad crystal quality." When crystallographers used diffractometers with point detectors, the observation of splitting and elongation of spots was useful for guiding the operator in the choice of scan parameters or special geometry. Today, in the age of two-dimensional detectors, this sort of visual indicator gives the user an idea of whether to expect some problem from the mosaic-determining routine and/or the integration of the data. And yes, it might lead one to get rid of a crystal on the grounds of poor quality.

RAXVideo Version

RAXVideo is an auxiliary program for displaying a video capture of the crystal. It requires a video camera and a supported video capture card. Since not all video capture cards support the same software, there are two versions of this program. Your system should normally be setup with the right version, but if you are not able to run RAXVideo, you can try another version. The options are:

- **DirectX**. This is the most advanced version and should be selected if you hardware supports it. Try it first and then if it doesn't work, revert to Video For Windows.
- **Video For Windows**. For some capture cards you need to revert to this version.

RAXShape, the auxiliary program for interactively outlining the shape of the crystal, depends on **RAXVideo**.

Tools Menu

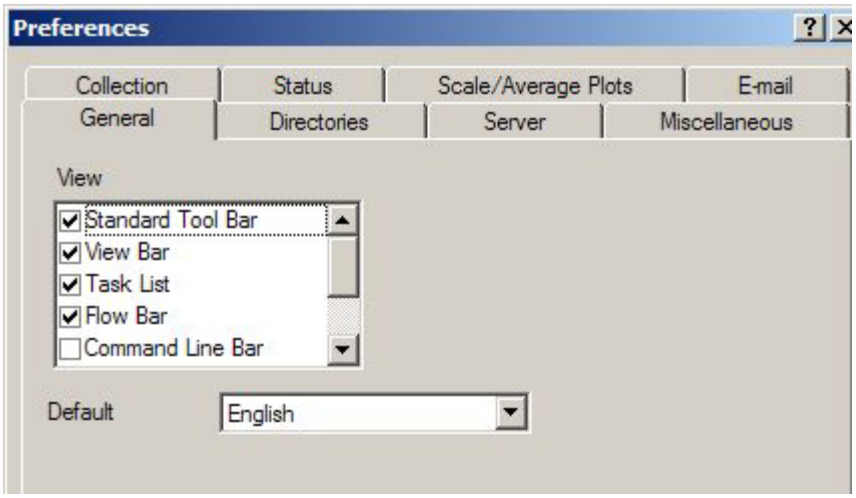
Tools>Administration

The Administration tool is mainly used to add and edit users. It is described in Chapter 2.

Tools>Preferences

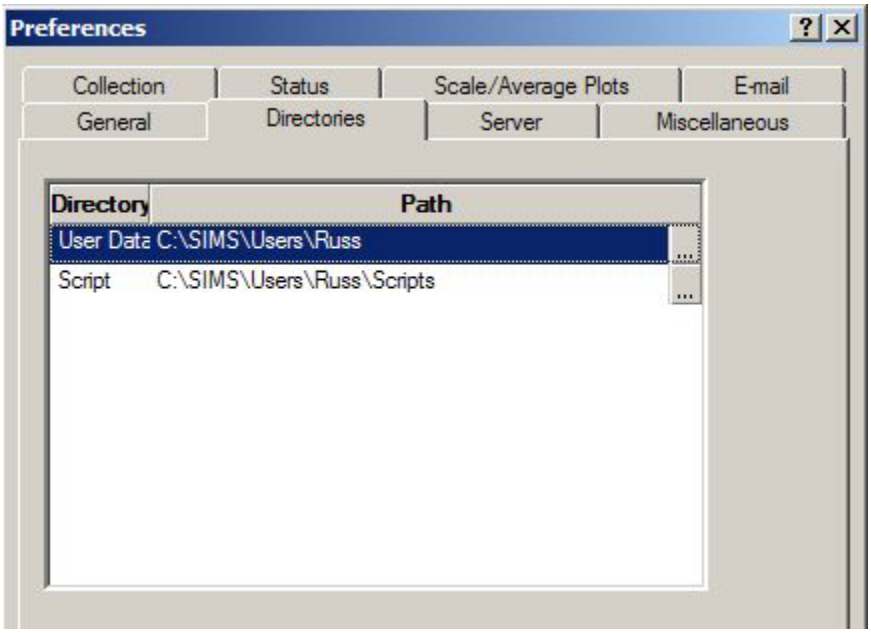
Preferences are used to set display, processing and other user preferences. Each user has their own set of preferences which is store in their User Data Directory in a binary file called UserPrefs_20.ho and an xml version called UserPrefs_20.xml. These preferences are set by going to the **Tools** menu and selecting **Preferences**.

User Preferences General Tab



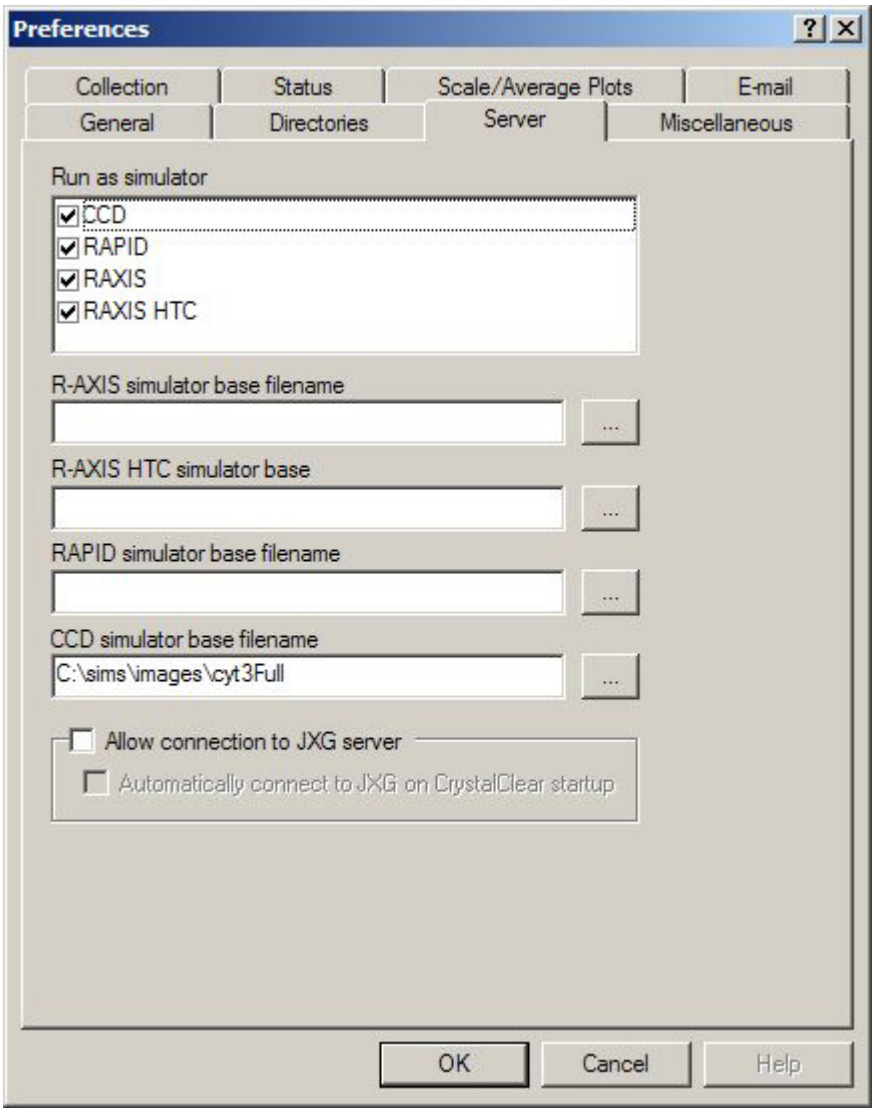
Parameter	Description
View	You can turn off parts of the standard display if you don't want to see them. This is not recommended. Simply uncheck the view that you do not want to see.
Default	English is the only supported language for CrystalClear at this time.

User Preferences Directories Tab



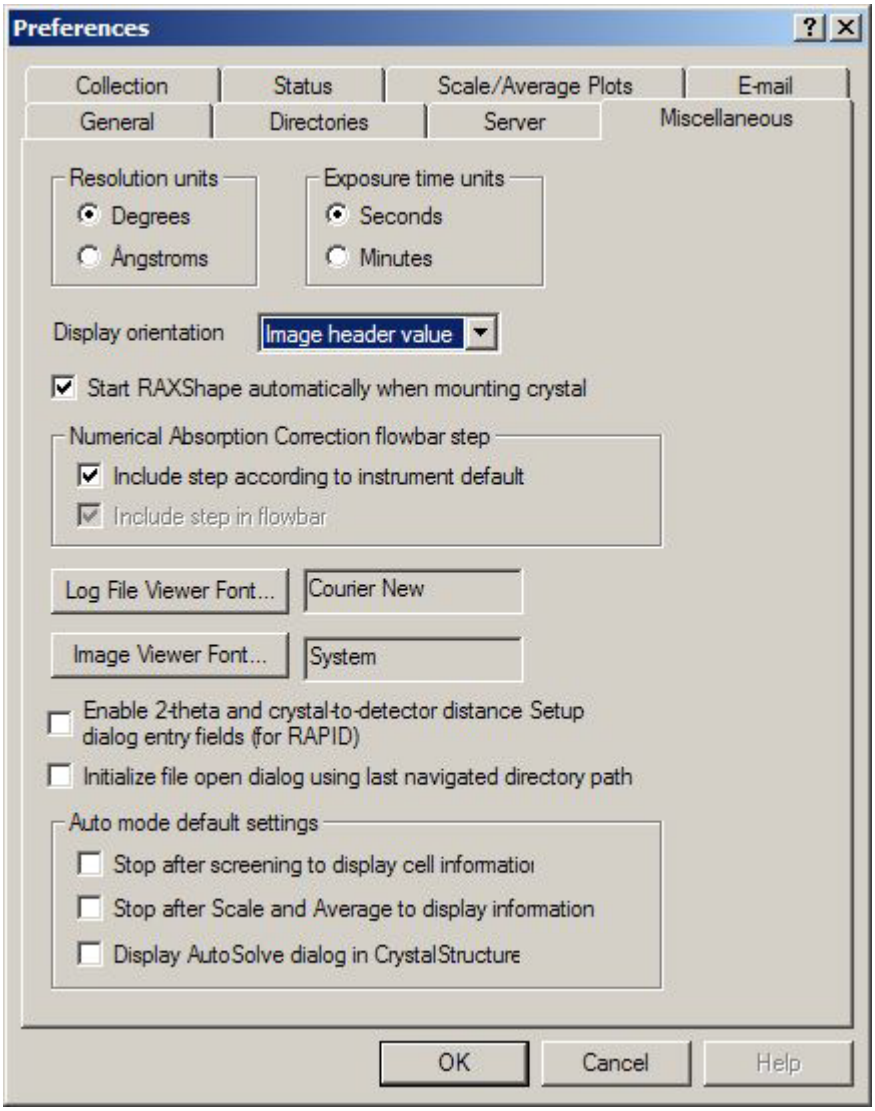
Parameter	Description
Directory Table	This is an alternate way for changing the **User Data Directory**. However, it is recommended that you do this through the **Administration** dialog on the **User** tab. The Script directory is not currently used.

User Preferences Server Tab



Parameter	Description
Run as simulator	If this box is checked CrystalClear runs an xxx_Simulator version of the instrument servers. This is mainly used for testing, but can also be useful if you are running CrystalClear off line on a computer intended primarily for processing data.
XXX-simulator base filename	For each class of instruments you can specify a base filename to be used if you do a simulated data collection. This is only really useful for testing CrystalClear.
Allow connection to JXG Server	At some sites the X-Ray generator is controlled by a program called JXG which acts as a server to CrystalClear. If you have the hardware and software setup to run JXG, you should check this box. You may also check **Automatically connect to JXG on CrystalClear startup** if you want to do this.

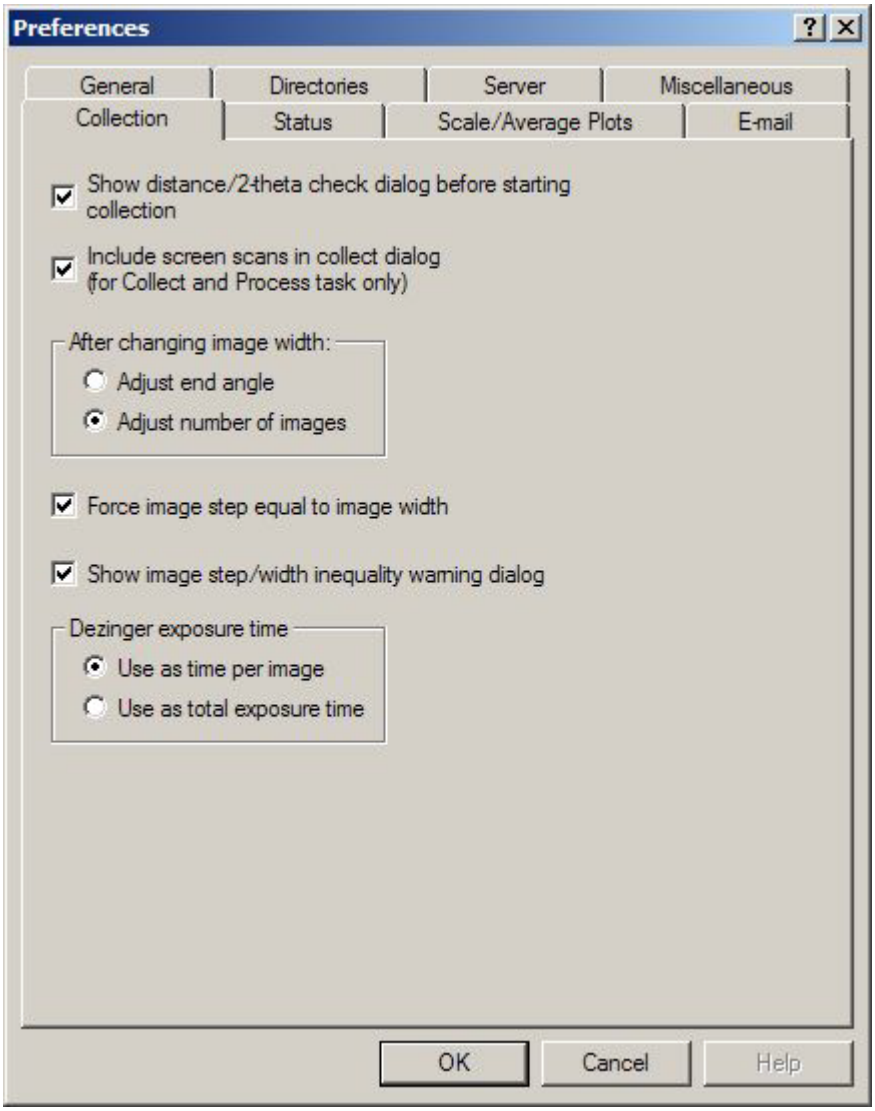
User Preferences Miscellaneous Tab



Parameter	Description
Resolution Limits	Some small molecule crystallographers prefer to work in Degrees. Protein crystallographers tend to work in Angstroms.
Exposure time units	If you have an R-AXIS instrument with a low power X-Ray generator you may prefer to specify exposure time in Minutes.
Display Orientation	The recommended practice is to take the display orientation from the image header, but you prefer you can specify an explicit orientation by selecting it in this drop down list.
Start RAXShape automatically when mounting crytsal	If you have RAXVideo and RAXShape installed you can have RAXShape start up automatically when you mount the crystal so that you can interactively outline the crystal shape. This allows you to do numerical absorption correction later in the process.
Numerical Absorption Correction flow bar step	If nothing is checked, this step never appears in the flow bar. If **Include step according to instrument default** is checked, then the decision is made based on whether your instrument type is most often used for small molecule or

Parameter	Description
	macromolecule work.
Log File Viewer font	You can select the character font for the log files. Columns line up better if you choose a fixed spacing font. But care must be taken if you are not using an English version of Windows. The same fonts may not be fixed spacing in another language version of Windows.
Image Viewer font	This is more a matter of personal preferences.
Enable 2 theta and crystal-to-detector…	The Rapid normally has a fixed 2 theta and crystal-to-detector distance setting. Checking this allows you to enter your own values.
Initialize the file open dialog…	If you want the file open dialog to remember the last path each time it opens, check this box.
Auto mode default settings.	These settings only apply in Small molecule Auto mode. • **Stop after screening to display cell information**. Check this if you want to review the cell information or the screen images before you go on to data collection • Stop after Scale and Average to display information. This stops before an attempt is made to solve the structure so that you can look at the results of integration. • Display AutoSolve dialog in CrystalStructure. In CrystalClear 2.0 the Solve step starts up CrystalStructure and passes it information about the sample. If you do not check this box, CrystalStructure immediate runs AutoSolve on the sample. However, there are some options for AutoSolve, so if you would like to set them in the AutoSolve dialog you should check this box.

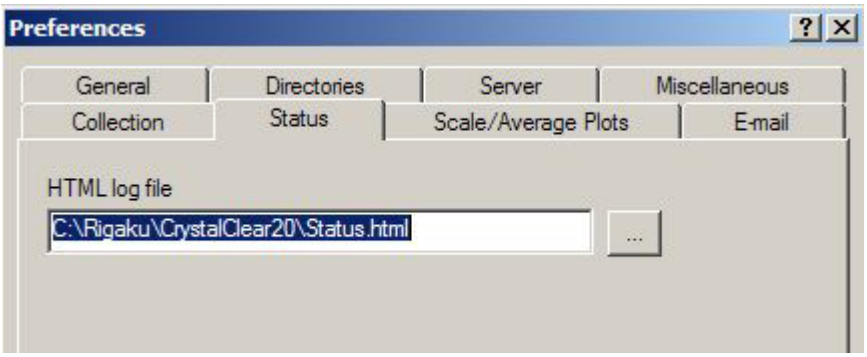
User Preferences Miscellaneous Tab



Parameter	Description
Show distance/2-theta check dialog before starting collection	This activates a reminder dialog that is brought up before each data collection to remind you to make sure that the distance and 2 theta specified do not cause a collision. CrystalClear does its own collision check that is normally very reliable, but for a new installation this might be wise.
Include screen scans in collect dialog…	This is mostly for informational purposes. The screens scans are marked as already collected, so you would not normally recollect them. However, they can remind you what the collection parameters were for screening.
After changing image width	The scan angle range, image width, and number of images are all inter-dependent. When you change the image width, you could either **Adjust the end angle** or **Adjust** the **number of images** to keep them consistent.
Force image step equal to image width	If you never want to use a different step (angular distance between images) and image width, then you should check this. This automatically updates image step when you update image width. But you can always go back in an override with a different step size.

Parameter	Description
Show image step/width inequality warning dialog	The goal of this option is to prevent you from accidentally creating a scan with a different image step and width by warning you whenever they are different. It applies only to the Collect dialog and not the Initial Images.
Dezinger exposure time	The two options here are to use the specified Dezinger exposure time (only available for certain instruments) as • Use as time per image • User as total exposure time

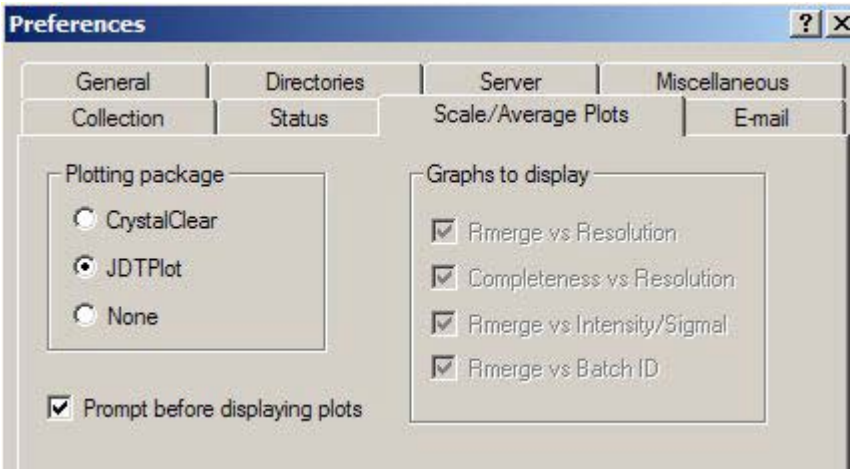
User Preferences Miscellaneous Tab



Parameter	Description
HTML log file	In order to support viewing CrystalClear status through a web browser, you may specify a path for the HTML version of the log file. This allows you to store and update this file in a location accessible to your web server, but requires that your IT group has configured a web server to link to this location.

User Preferences Scale/Average Plots Tab

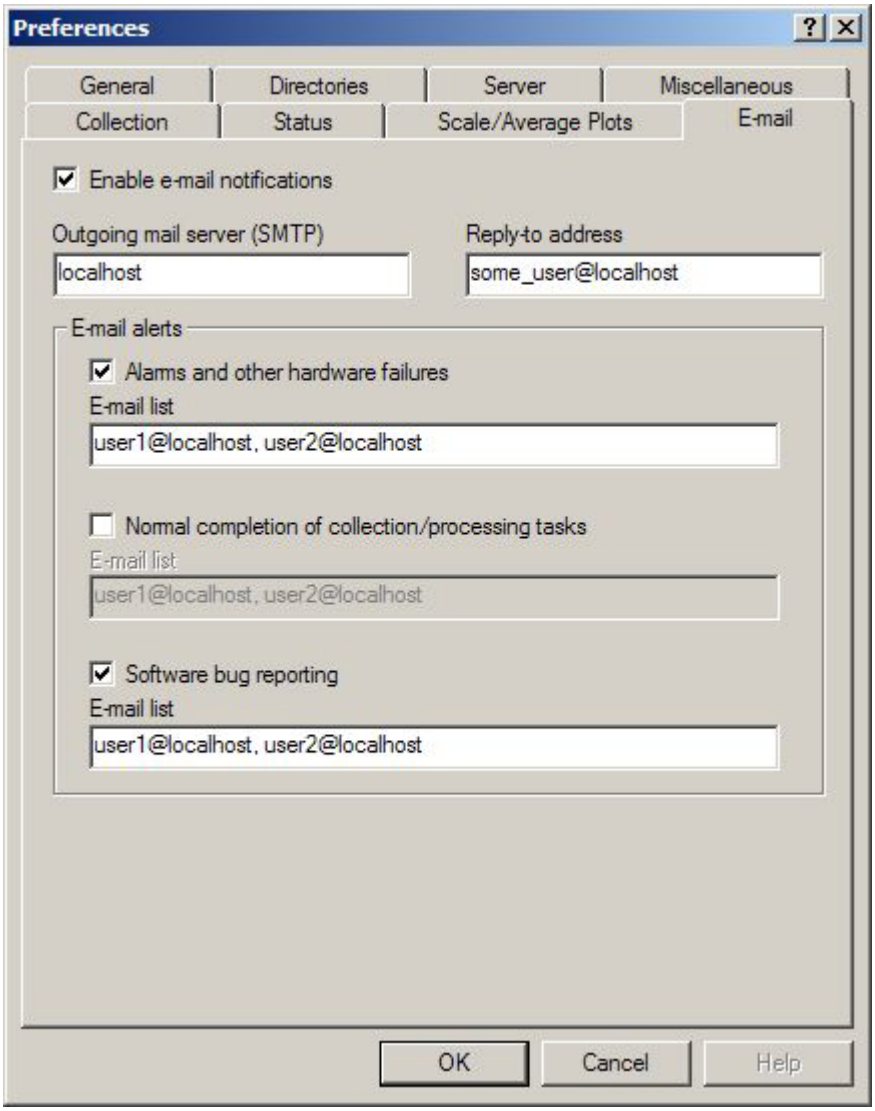
Scale and Average generates a lot of statistics that you should examine to determine the quality of the data. You can look at the tables in the log files or use JDTPlot to show various graphs with this information displayed in a convenient form.



Parameter	Description
Plot package	The CrystalClear plotting package option is no longer fully supported in version 2.0. So we recommend you select either JDTPlot or None.
Prompt before displaying plots	If you don't always want CrystalClear to start up JDTPlot (which is a separate program) check this box and a prompt is displayed before displaying the plot.
Graphs to display	This only applied to CrystalClear plots so is not longer relevant.

User Preferences Email Tab

CrystalClear uses an email server called RMailer to send emails at various times. This can be useful. For example if you start a data collection before you leave the lab on Friday night and there is a problem with the X-Ray generator at 8:00 PM that night, you might want to go in an fix it so you don't lose the whole weekend to data collection. Using this tab you can configure RMailer to send you an email when some event happens that you want to know about.



Parameter	Description

Parameter	Description
Enable e-mail notifications	In order to activate emails you should check this box. But you should also make sure that RMailer is installed and working. This is part of a **Utilities** package that contains RMailer and JXG which must be downloaded and installed separately from CrystalClear. It also requires that your control PC has network access to an email server.
Outgoing mail server	See your IT department to get access to an email smtp server that can be used to send emails from a program.
Reply-to Address	Some systems require a real user here and for others you can just use a fake address that tells you that it came from your instrument.
Alarms and other hardware failures	If you check this box, an email is sent to the addressed in the **E-mail list** whenever there is an alarm such as an X-Ray generator failure.
Normal completion of collection/processing tasks	This sends an email with a data collection step has finished. Many smtp servers are restricted so that they cannot send an email outside the firewall. So you may be able to use your email from the organization that owns the instrument, but not your private email account in this **E-mail list**.
Software bug reporting	If you have this checked when you select **Files>Untilities>Prepare bug report**, the report is automatically emailed to the **E-mail list**.

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13. FAQs

This chapter has a list of Frequently Asked Questions (FAQs) and short answers. These answers often refer you to other chapters in this manual.

Installation and Administration

Why don't I see a full CrystalClear window at startup?
CrystalClear was probably closed while it was minimized. After logging into CrystalClear and selecting the project / sample, right-click CrystalClear on the task bar and select **Maximize**. Close CrystalClear while it is maximized. Open CrystalClear and log in. You should be able to size the CrystalClear window.

Why does CrystalClear take a long time to respond, then an error message occurs saying that it cannot get a response from the R-AXIS?
If you are not actually connected to an **X-ray detector**, you should make sure CrystalClear is set in Simulator mode. Click **Tools > Preferences**, then click the Server tab. Check the detector in the list to force CrystalClear to run in Simulator mode. If you are connected to a real instrument make sure it is powered up before you boot the Control PC on which CrystalClear is running.

Why does CrystalClear "hang" when it tries to communicate with the CCD Camera?
If the MSCServDetCCD.exe program starts up but cannot connect to the Camera Server (MSCServCCDCamera.exe) open CrystalClear.configuration, which is found in the C:\Rigaku\Config directory. Change the value of "DaemonIP" to be the IP address of the computer on which the Rigaku Daemon is running. Make sure the Daemon is actually running. The instrument server starts up the camera server which is often on another FrameGrabber computer though the Daemon. So if it can't find this Daemon or can't start the server, it fails.

What should I do if my user list is empty after I installed a newer version of CrystalClear over an older version?
When updating CrystalClear you should almost always do a **Repair**. If you need to remove CrystalClear entirely, you should copy the **CrystalClear** (not CrystalClear140 etc) directory to a save location so that if it is removed when you remove CrystalClear you can replace it after installing the new version. The CrystalClear Directory contains the Administration directory which contains the list of users and sometimes the Administrator directory.

What does the Crypto-Box Error mean?
The software key or "dongle" is not recognized or is not properly installed. Remove the dongle. Run CBSetup.exe and then install the dongle on a different USB port. If you need to use the same port, you may have to go into the Device Manager and remove the device, unplug it, run CBSetup, and then plug it back in.

Crypto-Box Error 8: no dongle found.

The CrystalClear Crypto-Box dongle was not found. Install the Crypto-Box Dongle and try again.

Crypto-Box Error 30: Error, the attached Crypto-Box did not come from Rigaku.

A Crypto-Box was found but it did not have the Rigaku identifier. Install the Crypto-Box Dongle that came with CrystalClear on the PC where CrystalClear is installed and try again. If you continue to have problems contact your support person to get a replacement dongle.

Crypto-Box Error 31: Error, registration key in the registry and registration key in the Crypto-Box do not match.

Normally this is caused when CrystalClear was installed with an incorrect Dongle ID, or the user changed the Crypto-Box dongle ID. In this case, reinstall CrystalClear and input the correct license number during installation. If you still have demo time left, you can also use Edit|Set Dongle ID.

This error may also occur if a non-CrystalClear Crypto-Box dongle is installed on the PC, but the CrystalClear dongle was not. In this case, install the CrystalClear Crypto-Box Dongle and CBSetup software which came with CrystalClear on the PC where CrystalClear is installed and try again.

How can I change the font on the Log File Viewer? The table columns do not align properly in the log file?
This often occurs with CrystalClear installations on Japanese Windows systems. When table columns do not align properly, the font setting in the log file view is not a fixed width. Change the font using **Preferences** dialog using the following procedure. On the CrystalClear Menu Bar, click **Tools** > **Preferences** > **Miscellaneous** tab > **Log file viewer font...**button. Select the desired font. **NOTE**: We recommend Courier New for English versions of Windows, and MS Gothic for Japanese Windows.

When I save the **Setup** dialog to the **Site-Level Defaults**, Wavelength, Polarization values and Collimation type (among other settings) are not being saved. Why don't these values appear in new collection samples?
To save these in newer versions of CrystalClear click **Save to Config** in the X-Ray Source tab. This updates the configuration file that controls these values.

RAXShape and **RAXVideo** start everytime I click **Mount Crystal.** How can I turn off this feature?
Turn off **RAXShape** and/or **RAXVideo** using the **Preferences** dialog as follows: On the *CrystalClear* Menu Bar, click **Tools** > **Preferences** > **Miscellaneous** tab > Uncheck **Start RAXShape automatically when mounting crystal.** If RAXVideo doesn't function properly, try clicking Instrument **> RAXVideo > DirectX.** If this doesn't work, then click **Instrument > RAXVideo > VideoForWindows version.**

How can I add a user?
Only users with administrative privileges may add new CrystalClear users. On the **CrystalClear** menu bar, click **Tools > Administration.** The **Administration** dialog appears Click the **Users** tab, then click **Add**. Refer to the Chapter 2**, User Management** for additional information.
NOTE: If the **Administration** option is grayed-out, exit CrystalClear and log on using an account that has administrator privileges.

RAXVideo doesn't start up. An error message says it is not able to initialize the video capture device.
If **RAXVideo** doesn't function properly, click **Instrument > RAXVideo Version> DirectX** on the CrystalClear menu bar**.** If this doesn't work, then click **Instrument > RAXVideo Version>VideoForWindows.**

Crystallographic Questions

Why does Crystal to Detector Distance show "Per Scan?"
In this sample there are images with different crystal to detector distances. Look in the **Scan State Display** for information relative to each scan.

How can I hide the columns on the Scan State Display which contain information in which I am not interested?
Right-click any column heading in the **Scan Table**. An **All Columns** menu appears. Check (select) all the columns you wish to display and uncheck those you want to hide.

Why is the TWINSOLVE button now grayed out?
CrystalClear requires a license file to run the TwinSolve processing software. Contact Rigaku/MSC technical support for information about obtaining a license file. Note that TwinSolve is most effective for work with small molecules.

If I aborted a collection before it was complete, should I modify the Collect Images dialog to resume collection?
CrystalClear tracks the progress of data collection and can normally be restarted and it just finishes the collection. To resume the collection, access the **Collect Images** dialog and click the "**Run**" button. You can visually confirm the collection by looking in the "**Scheduled**" column which displays the images scheduled for collection.

How do I take axial photos?
Select **Instrument>Axial Photo** on the menu bar. The **Axial Photo** dialog appears. This is documented in Chapter 12

How do I perform Cell Transformation?
Access the **Cell Transformation** dialog by clicking **Processing** > **Cell Tools** > **Transformation** on the CrystalClear Menu Bar. The **Cell Transformation** dialog appears. This dialog varies depending on the processing suite (d*TREK, FS_PROCESS, or TwinSolve). The version for each processing suite is described in the suite's chapter.

How can I do a Cell Reduction?
Access the **Cell Reduction** dialog by clicking **Processing** > **Cell Tools** > **Reduction** on the CrystalClear Menu Bar. This operation is defined for d*TREK in Chapter 8 and FS_PROCESS in Chapter 9. It is not available for TwinSolve.

Why doesn't the Flow Bar include the processing options I want?
You may have selected a **Collection** task. All the other options in the **Tasks** drop down list include processing steps.

Why have my dialog settings reverted back to the system defaults, instead of to settings I specified in a previous session?
Your settings are saved during a session, but are not saved between sessions unless you click the **Save** button on the dialog. Read about Defaults in Chapter 3.

Why can't I select a processing suite? (i.e. d*TREK , TwinSolve or FS_PROCESS.)
You must have a sample open before you can select a processing suite. Click **File > Open Sample** from the menu to select a sample, or **File > New Sample** to create a new sample. You must specify the processing suite when you open this sample. Note that the processing suite is associated with the sample, so you are not automatically in that processing suite if you open an old sample that had a different processing suite.

Why does the Crystal-to-Detector Distance show "0" when I start a Sample as a Process task?
You may have collected the data without setting the **Crystal-to-Detector Distance** in the software for addition into the header or be using images from a 3rd party detector that do not carry this information.

- If you know the Crystal-to-Detector Distance, go the the **Setup** dialog, edit the **Crystal-to-Detector Distance**; then continue.
- If you do not know the true **Crystal-to-Detector Distance**, try the following:
    - In an image display window, turn on the arcs tool and play with the crystal to detector distance in the image information area. The arcs change size and shape relative to this (and the 2-theta angle). See if you can line up a ring (ice, salt, etc.). Then take these numbers to the Setup dialog and edit the fields.
    - Set a best guess in the crystal to detector distance field in Setup and try to index. The closer you are to the crystal-to-detector distance (or any geometric parameter), the lower the residual of the correct lattice. This may be quite a bit of work. If no solutions appear after many attempts, Find Peaks off two (or more) images in the same scan separated by nearly 90 degrees and use difference vectors in indexing.
    - If the lattice is uncertain, but the detector parameters are remembered vaguely, try indexing and using the triclinic cell for refinement then re-index to see if the higher symmetry cell is determined.

How do I change the Resolution ranges for all steps in processing?
Anywhere there are Resolution Limits fields and a "Set" box, select the box and choose the processing steps to use the resolution limits currently in the edit fields and press "OK". You can also click the  icon in the main toolbar or **Sample>Set Resolution…**

How can I get Cell Lengths? (I have no idea what the Cell Lengths may be.)
In an image display window zoom in on an area where spots are regularly spaced. Turn on the measure tool by selecting the ruler icon. Left-click one spot and drag to an adjacent spot. The inter-spot distance is displayed as you drag.

Why doesn't the red plus sign appear in the Beam Stop shadow?
You are collecting images with an improper direct beam position and should contact your administrator to redo direct beam shots

Why did I get a "Find Spots Failed" message and the log file shows "...reading image lys0001.osc... Error opening file lys0001.osc Error is: -2"?
The image is not there or is corrupted. You may have to wait for the image read to finish, especially if you are running on an R-AXIS.

How can I increase the number of reflections found in **Find Spots**?

- Decrease the I/sigma
- Widen the resolution range
- Decrease the minimum pixel value
- Decrease the peak filter
- Increase the number of images used
- Use the Add Spots button to manually pick spots

How can I decrease the number of reflections found in **Find Spots**?

- Slide the number of spots slide in the image information window to the left.
- Use the delete spots icon to delete spots.
- Set the I/sigma higher in **Find Spots** dialog.

- Increase Min Pixel Value.
- Increase the Peak Filter.

After running **Predict Spots**, why don't I see predictions on my second screening image?
Prediction cannot be run on non-contiguous images. If you wish to predict on a second screening image, run **Predict Spots** again on the second image.

How can I increase my spot-spot separation?
Move the detector back so the crystal-to-detector distance increases.

How can I get the correct indexing based on the **Measure Tool** distances?

- Ensure that the crystal to detector distance, X and Y beam positions, wavelength, and 2-theta angle are correct. If these values are wrong, the **Measure Tool** values are wrong too.
- Indexing may be correct if there is additional symmetry. Continue to **Predict Spots** for verification.
- If the peak shapes are poor, then try raising the I/sigma level to avoid the weaker peaks.
- If the cells are all much too large, including the triclinic, set the maximum cell in the indexing to a smaller value (about 2x that of the closest spot separation for starters).

How can I see all the crystal lattices in the table of indexed lattices?
Set the maximum residual in the advanced tab of index to 100.

How can I choose the correct lattice in indexing?
In the **Index Spots** dialog, select the "User Chooses Solution" option. Upon completion, choose the lattice from the list. If all the lattices are not displayed, increase the maximum residual.

How can I increase the reflections included during **Refine Spots**?
Adjust the resolution limits, I/sigma, and rejection limits.

Why is "test mosaicity" grayed out?
A range of images must be used for this refinement. You are using "**Refine on**" "**Reflection List.**" Set "**Refine on**" to "**Images,**" then select images in the "**To Use**" field.

Why is there is no reflection file list in the Refine dialog?
You are using "**Refine on**" "**Images**." Set "**Refine on**" to "**Reflection List**"

Why are all of the option boxes greyed out in the **Refine** dialog?
Select "**Single Step Refine**" under **Macro** to toggle on/off individual parameters, instead of using the preset macro.

Why are there more/less spots than prediction circles on my predicted image?
In the **Predict Spots** dialog increase/decrease the mosaicity and predict again. Or, you may have the wrong cell and need to re-index.

If the predictions are terribly wrong, how can I start from scratch with the images?
Open the **Processing State Display**, go to the State History, click the down arrow, scroll to the bottom, select one of the earlier states (like "1. Setup" or "2. Find Run") and "Set as current". Alternately you can just go back to Find Spots and reprocess the sample.

Why does refinement seem to stop during integration?
Try increasing the number of images per batch.

How can I integrate without refining mosaicity?
Set **Fix mosaicity** and enter a value in the Advanced tab of **Integrate Reflections.**

How can I get rid of ice rings during integration?
Go to **Integrate Reflections** Advanced tab and Add/Delete rings to the list (avoided area in integration).

How can I keep integration from stopping when it finishes all the images on disk so it waits for the images yet to be collected to appear?
Adjust the wait limit in the **Integrate Reflections** Advanced tab. If an image is not on disk when integration gets to that image, it queries the disk every 15 seconds until it finds the image (integrates and looks for the next image) or reaches the end of the wait limit (completes integration with the reflections it has to that point).

What should I do if **Laue** shows that I integrated the data in the wrong crystal system?
Here you need to think about what it is showing you. In general, if the symmetry using the lower symmetry unit cell during integration is higher than merited by Laue, then you should consider reintegrating. If the symmetry is higher than that for integration, it is recommended that you reintegrate, but you may still have good data which is usable for a first reflection file.

How can I get a different format for my reflection file?
In the CCP4 suite there is a conversion program to convert to mtz format from the d*TREK format.

Where do I look in order to use the d*TREK interactive mode?
From the CrystalClear menu bar, click **Processing >d*TREK Interactive**. Then click the DOS box in the task bar and enter your **d*TREK** command (or any DOS command.)

Trouble Shooting

How can I troubleshoot problems which occur during processing?
CrystalClear creates log files that help you see what settings were used during processing. The results of the log files can help in troubleshooting problems. To track the progress of an operation, you may view a log file during the execution of an operation. You may also view a log file as a text file following the operation.

How can I view log files?
To view a log file during a processing step, click **Windows > View Log File** on the CrystalClear menu bar. The selected log file is displayed on the screen as the operation is processed.
To view a log file or another file in text mode, use the **Tool Bar** menu and click the **View Log File**  button. The **Open Text File** dialog appears. Select the file to view. Click **Open**.
Alternatively, click **File > Open Log File** on the CrystalClear menu bar**.**

What is the name of the log file?
The name for a specific log file is based on two things: 1) the processing suite selected, and 2) the operation being processed. For d*TREK, they tend to correspond to step names. **Find Spots** writes dtfind.log, **Index Spots** writes dtindex.log etc.

How can I print a log file?

On the CrystalClear Menubar, click **File > Open Log File**. The **Open Text File** dialog appears, allowing access to all available log files. Select a log file, click **Open**. If you wish to print this **Log File**, select **Print** once the **Log File** is **Open** in the **Log File Viewer**.

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