BRUKER ADVANCED X-RAY SOLUTIONS

APEX2

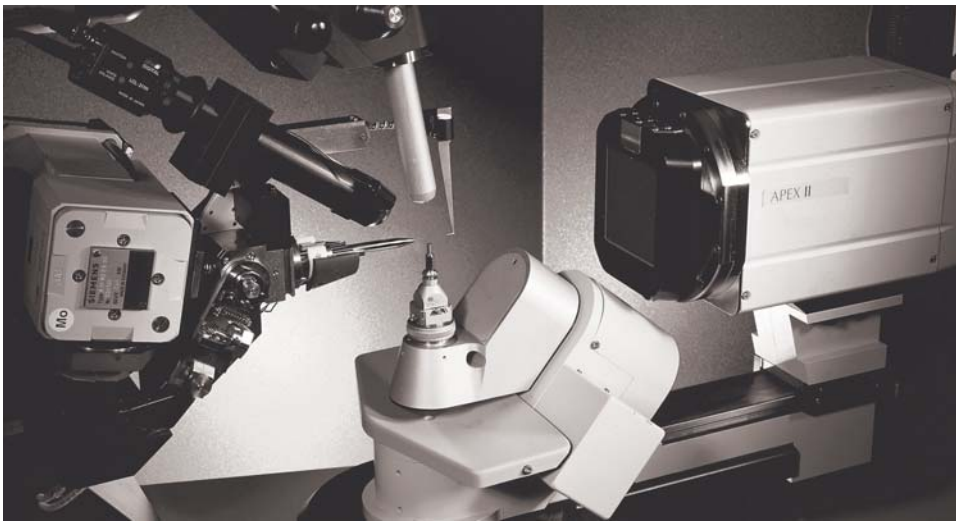
Version 2

USER MANUAL

M86-E01078 6/06

APEX2 Version 2

User Manual



This document covers the APEX2 software program. References to this document should be shown as M86-Exx078 APEX2 User Manual.

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Revision History

Revision	Date	Changes
0	2/05	Original release.
1	6/06	Revised and expanded for APEX2 Version 2.

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Table of Contents

1 About This User Manual

1.1 APEX II Systems for Chemical Crystallography

Bruker AXS Kappa APEX II and SMART APEX II systems are the newest members in the Bruker AXS product line of instrumentation for single crystal X-ray diffraction. These systems provide the tools for complete small molecule structure determination.

The APEX2 software features a redesigned start-to-finish graphical user interface (GUI) along with the optional Phase ID and AutoStructure software modules. The AutoStructure module simplifies structure determination more than ever before.

The hardware features the APEX II CCD detector (with four-port readout of a 4096x4096 CCD chip), radiation safety enclosure system, and a choice of two goniometers.

The Kappa APEX II system features the Kappa 4-axis goniometer.

The SMART APEX II system is an enhanced version of the SMART APEX fixed-chi system.

This User Manual and associated YLID test data are intended to provide you with a step-by-step guide to data collection and processing using the APEX2 software program.

The test data supplied was collected on a Kappa APEX II diffractometer with graphite-monochromated molybdenum radiation from a sealed tube generator. The high-quality (0.75 Å resolution) data allows easy refinement of the hydrogen atom positions and determination of the absolute structure of the crystal.

1.2 Terms and Conventions Used in this User Manual

Before using this User Manual, it is important to understand the terms and typographical conventions used. Certain kinds of formatting in the User Manual's text are used to identify special kinds of information.

1.2.1 Typographical Conventions

Table 1.1 shows typographical conventions used to help you quickly locate and identify information in this User Manual.

Convention	Usage
boldface	Software interface elements (such as icons, menu items, and buttons) to be selected as part of the current procedure.
italics	New terms and words requiring emphasis.
`monospace`	Information read from or entered into a command prompt.
>	Navigation through a hierarchical menu. For example, "Select **Start > Programs > Bruker AXS > APEX2**" describes navigating Windows' menus from **Start** to **Programs** to **Bruker AXS** to **APEX2**.
[square brackets]	Keyboard input.

Table 1.1 – Typographical conventions

1.2.2 Equivalent Terms

Frame/Image

In this User Manual, the terms "frame" and "image" are used interchangeably to describe two-dimensional X-ray data, whether read from the CCD camera or generated within APEX2.

Greek and Roman Text

This User Manual uses scientific terminology that may be rendered in Greek text. However, this User Manual follows a convention of using Roman text to the greatest extent possible.

Greek	Roman
2θ	2-theta
θ_1	theta1
θ_2	theta2
ω	omega
ϕ	phi
χ	chi
γ	gamma

Table 1.2 – Greek and Roman text

1.2.3 Warnings, Cautions, and Notes

This User Manual contains notices that you must observe to ensure your own personal safety, as well as to protect the product and connected equipment. These notices are highlighted in the User Manual by a warning triangle and are marked as follows according to the level of danger.

WARNING

The word “WARNING” alerts the reader to an immediate or potential hazard that can result in death, severe personal injury, or substantial property damage.

CAUTION

The word “CAUTION” alerts the reader to a potential practice or condition that could result in minor personal injury or damage to the product or property.

NOTE: The word “NOTE” in bold capital letters draws your attention to particularly important information on the product or handling of the product, or to a particular part of the product documentation.

1.3 Referenced Documentation

Table 1.3 contains a list of documentation referenced in this User Manual. It is recommended to have this additional documentation available as you work with this User Manual.

Documentation Part Number	Title
269-0175xx	SAINT Software Reference Manual
M86-Exx024	Microscope Focus and Sample Alignment
M86-Exx043	BCP and BIS Installation Notes
M86-Exx045	RLATT User Manual
M86-Exx087	APEX2 and PROTEUM2 Installation Notes
M86-Exx092	User Manager User Manual
M88-Exx099	License Manager User Manual

Table 1.3 – Referenced Documentation

1.4 X-ray Safety

WARNING

X-ray equipment produces potentially harmful radiation and can be dangerous to anyone in the equipment's vicinity unless safety precautions are completely understood and implemented. All persons designated to operate or perform maintenance on this instrument need to be fully trained on the nature of radiation, X-ray generating equipment, and radiation safety. All users of the X-ray equipment are required to accurately monitor their exposure to X-rays by proper use of X-ray dosimeters.

For safety issues related to operation and maintenance of your particular X-ray generator, diffractometer, and shield enclosure, please refer to the manufacturer's operation manuals or to your Radiation Safety Officer. The user is responsible for compliance with local safety regulations.

1.5 APEX2 Help and Technical Support

1.5.1 The "What's This?" Function

Context-based help is available at any time within APEX2 by simply clicking the "What's This?" icon:

Figure 1.1 – "What's This?" icon

After clicking the icon, click any window, control, or field within APEX2 to obtain detailed help in a pop-up window.

1.5.2 Technical Support

Users are invited to contact Bruker AXS whenever there are problems or questions related to the system. Before contacting Bruker AXS, please:

- If there is a software problem, determine the version of the program.
- Record any error messages.
- Determine conditions and steps that recreate the problem.

If the instrument is in North America, contact our North American Service Center:

Bruker AXS Inc
Customer Support
5465 E Cheryl Pkwy
Madison, WI 53711-5373
U.S.A.

Phone No: +1 (608) 276-3087
Toll-free: 1 (800) 234-9729
Fax No: +1 (608) 276-3015
E-mail: Customer.Service@bruker-axs.com
Internet: www.bruker-axs.com

If the instrument is outside North America, contact your local Bruker AXS Service Center.

2 Hardware Overview

APEX II systems share common hardware components with other Bruker AXS products. Other members of this new generation of instruments include the D8 ADVANCE, D8 DISCOVER, and D8 GADDS systems for general diffraction. Documentation on some of these common hardware and software components is available in the User Manuals for the D8 family of instruments.

The two hardware platforms for the APEX II systems are the Kappa APEX II (the four-axis advanced research instrument) and the SMART APEX II (the three-axis laboratory instrument). Software functionality is essentially the same for both platforms.

2.1 System Components

The system (Figure 2.1 and Figure 2.2) consists of the following basic components.

- APEX II CCD detector
- 4-axis Kappa or 3-axis SMART goniometer
- K780 X-ray generator
- Radiation safety enclosure with interlocks and warning lights
- D8 controller
- Refrigerated recirculator for the detector
- Instrument control (BIS) and crystallographic software (APEX2) computer
- Video microscope
- Optional Accessories (low- and high-temperature devices)

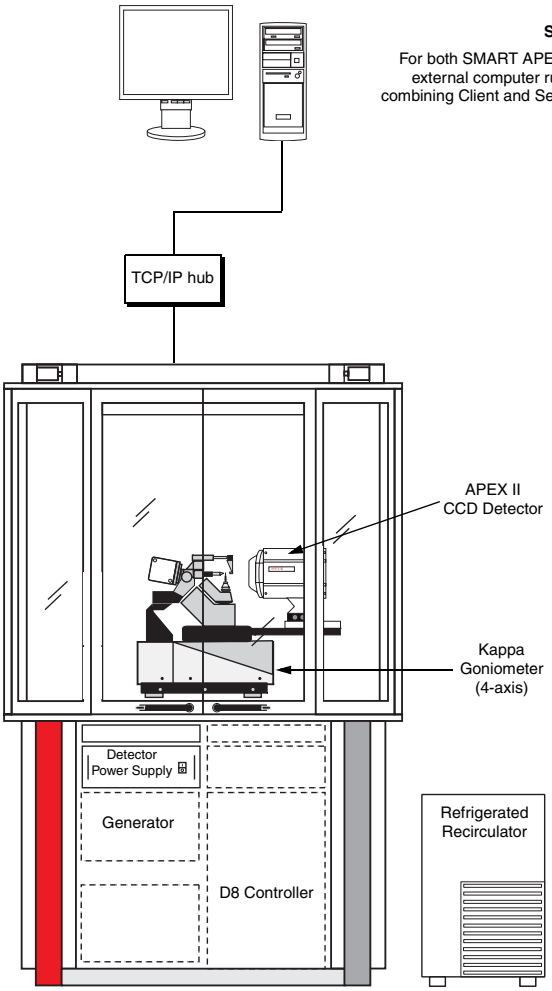


Figure 2.1 – Kappa APEX II system

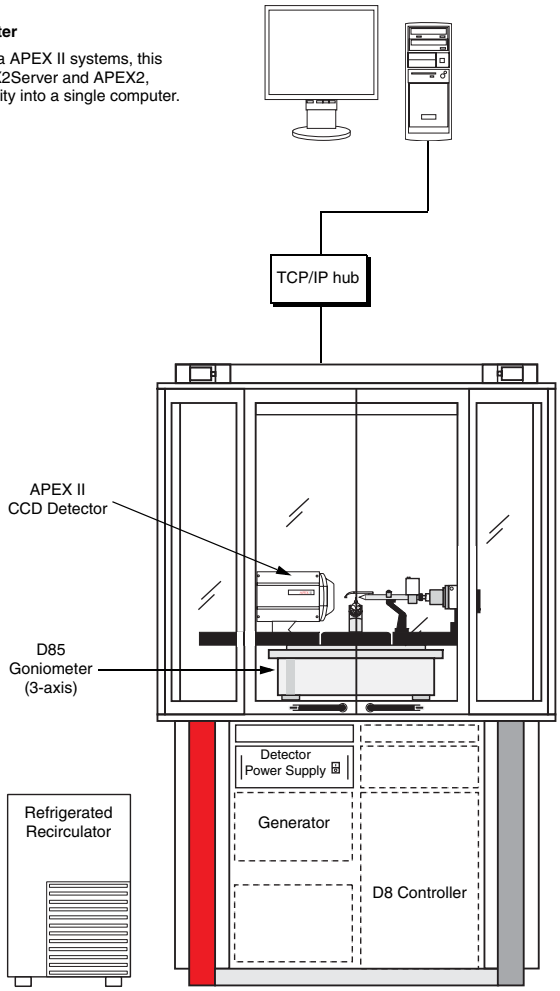


Figure 2.2 – SMART APEX II system

2.1.1 APEX II Detector

The APEX II detector is specific to this system. Status lamps on the top of the detector housing indicate when the detector is on (green) and off (red).

On Kappa APEX II systems, the detector is mounted on a motorized DX track. The camera distance is computer-controlled (a typical distance for the camera is 60 mm).

On SMART APEX II systems, the detector is mounted on a DX dovetail track. The track has a scale that is calibrated in mm to indicate the distance from the crystal to the detector face (a typical distance is 60 mm). The detector distance has to be changed manually.

An optional motorized DX track is available for the SMART APEX II.

2.1.2 Goniometer

The goniometer module and APEX II detector make up the unique hardware of the system. This is the part of the instrument that actually performs the experiment.

Several components make up the goniometer module with APEX II detector:

- Goniometer (3-axis or 4-axis)
- APEX II detector
- X-ray source (including shielded X-ray tube, X-ray safety shutter, and graphite crystal monochromator)
- K780 X-ray generator
- Timing shutter and incident beam collimator (with beamstop)
- Video microscope

Kappa APEX II Goniometer

The Kappa APEX II system uses a horizontally-oriented Kappa goniometer with 2-theta, omega, kappa and phi drives and a motorized DX track for setting the detector distance. It includes mounting points for the video microscope and for optional attachments such as the optional low-temperature attachment.

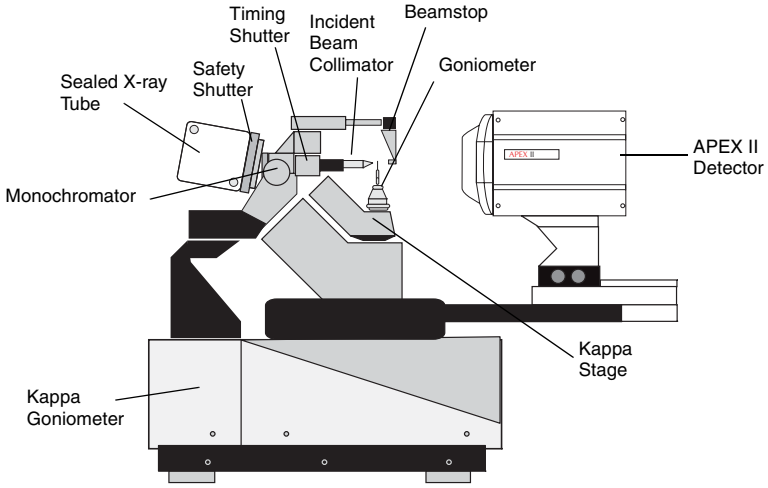


Figure 2.3 – Kappa 4-axis goniometer components

With the kappa angle, the crystal can be oriented at chi from -92° to 92°. This leaves the top of the instrument open for easy access. Kappa can be positioned so that the phi drive, which has unlimited rotation, can be swung under the incident beam collimator, allowing free rotation in omega.

SMART APEX II Goniometer

The SMART APEX II system uses a horizontally-oriented D8 goniometer base with 2-theta, omega and phi drives, dovetail tracks for the X-ray source and detector, and an additional mounting ring for accessories such as the video microscope and optional low-temperature attachment.

The 3-axis system incorporates a fixed-chi stage with a chi angle of approximately 54.74° and a phi drive with 360° rotation, which is so compact that it swings under the incident beam collimator, allowing free rotation in omega.

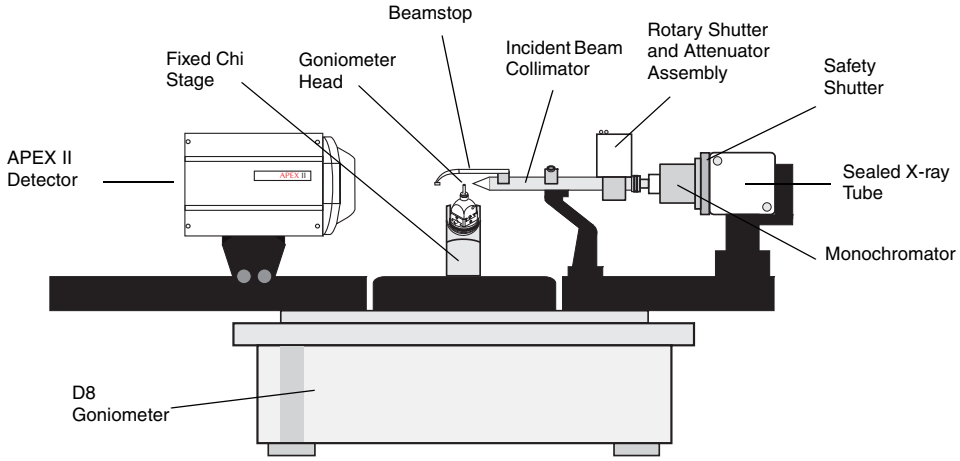


Figure 2.4 – SMART goniometer components

X-ray Source

Three components make up the X-ray source: a shielded X-ray tube, an X-ray safety shutter, and a graphite crystal monochromator.

The sealed-tube X-ray source produces the X-ray beam used by the system.

The X-ray safety shutter is built into the X-ray tube shield. The shutter opens upon initiation of a set of exposures and closes upon the end of collection. Status lamps on the shutter housing indicate when the shutter is open (red) or closed (green). The shutter is also interfaced to the controller and to the safety interlocks.

A tunable graphite crystal monochromator selects only the K_α line (λ_{avg} = 0.710730 Å for Mo radiation, λ_{avg} = 1.541838 Å for Cu radiation) emitted from the X-ray source and passes it down the collimator system.

K780 X-ray Generator

The K780 X-ray generator is a high-frequency, solid-state X-ray generator that provides a stable source of power for operations up to 60 kilovolts (kV) and 50 milliamps (mA).

Typical maximum power settings for the APEX II system with a fine focus tube are:

- 50 kV, 40 mA for Mo radiation
- 40 kV, 30 mA for Cu radiation

Either copper or molybdenum tubes may be used on APEX II systems. For both types of tubes, the kV setting should not exceed 50 kV and the power (kV x mA) should not exceed the power rating given on the tube cap.

Because the generator is interfaced to the controller, the power settings can be adjusted within the APEX2 software. This is usually not necessary as the software automatically increases the power to the user-defined values at the beginning of an experiment and lowers them when the instrument is inactive.

Timing Shutter and Collimator

On SMART APEX II systems, the monochromatic X-ray beam then passes through the labyrinth, the timing shutter, and the incident beam collimator before striking the crystal.

On Kappa APEX II systems, the monochromatic X-ray beam passes through a small labyrinth, the timing shutter, a secondary labyrinth and the incident beam collimator before illuminating the crystal.

- The labyrinth is a device that ensures that the collimator and shutter are tightly connected to prevent X-ray leakage.
- The timing shutter is a device which precisely controls the exposure time for each frame during data collection. Its status lamps indicate when the shutter is open (ON) and closed (OFF). For SMART APEX II systems, this assembly also houses an automatic attenuator. Kappa APEX II systems do not have an attenuator.
- The incident beam collimator is equipped with pinholes in both the front (near the crystal) and rear (near the source). These pinholes help to define the size and shape of the incident X-ray beam that strikes the crystal. (Collimators are available in a variety of sizes, depending on your application.)
- The beamstop catches the remainder of the direct beam after it has passed the crystal. The beamstop has been aligned to minimize scattered X-rays and to prevent the direct beam from hitting the detector. The entire collimator assembly is supported by a collimator support assembly that has been precisely aligned to guarantee that the X-ray beam passes through the center of the goniometer.

Video Microscope

The video microscope, an essential part of the system, allows you to visualize the crystal to optically align it in the X-ray beam. It also allows you to measure the crystal's dimensions and index crystal faces with APEX2's Face Indexing feature. The microscope is interfaced to the computer and is operated through the VIDEO program. The VIDEO program includes several computer-generated reticles and scales to make it easy to center and measure the crystal's physical dimensions.

2.1.3 Radiation Safety Enclosure with Interlocks and Warning Lights

A common component of all systems in the D8 family is the radiation safety enclosure. This new design is fully leaded (i.e., leaded windows, leaded metal sides and panels) to protect you from stray radiation. The enclosure also includes warning lamps (a government requirement) that alert you when X-rays are being generated. As a special feature, the enclosure also incorporates interlocks for both hardware and software: an automatic system-interruption device that senses when the doors and panels are open and prevents data collection and use of the shutter until you close the doors.

2.1.4 D8 Controller

The D8 controller is an electronic module enclosed in the rack behind the front panel of the instrument. It contains all of the electronics and firmware for controlling the generator, opening the X-ray shutters, and monitoring other instrument functions such as safety interlocks, generator status, and detector status.

The goniometer is controlled by the D8 controller.

2.1.5 Refrigerated Recirculator for the Detector

To minimize dark current in the APEX II detector, dual Peltier devices are used to cool the CCD chip to approximately -58°F (-50°C). The refrigerated recirculator uses an ethylene glycol/water mixture to absorb the heat from the Peltier devices.

2.1.6 Computer(s)

The SMART or Kappa APEX II system uses a single computer for control of the experiment, storage of raw frame data, integration of the data, and solution and refinement of the structure.

An additional client computer can be set up to improve the performance of the system.

The computer or computers are often attached to a network of similarly configured computers with access to local and/or network printers.

NOTE: Connection to the external network must be done with care. Consult with local security experts.

2.1.7 Accessories

Various devices can be mounted on the goniometer base. These include optional low- and high-temperature attachments. Both instruments can be used with diamond-anvil cells.

3 Software Overview

This section presents an outline of the APEX2 software, including a brief description of the software layout as well as the graphical user interface (GUI).

From a software and operational viewpoint, the APEX II systems use the GUI of the APEX2 software program to control all operations from crystal screening to report generation for a typical crystallography study. This is a complete departure from the command-driven, functionally separate modules of SMART, SAINTPLUS and SHELXTL. Enhanced versions of the proven and widely-accepted programs used by these modules (e.g., SAINT, SADABS, XPREP, XS, XM, XL, etc.) underlie the APEX2 GUI and provide powerful tools.

3.1 Introduction to Client and Server Functions

The various programs within the APEX2 Suite have a client/server relationship (Figure 3.1), in which the *server* program (BIS) executes commands given by one of several *client* programs (APEX2, APEX2Server, or BCP).

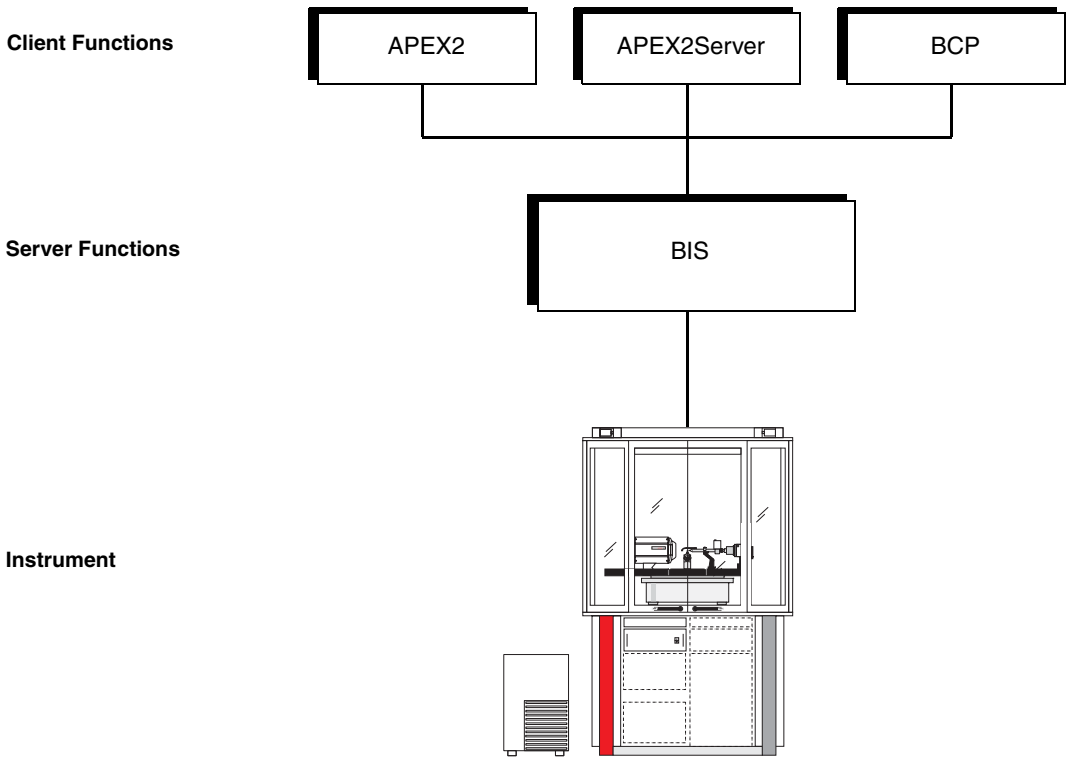


Figure 3.1 – APEX2 software relationships

3.2 Server Functions

3.2.1 APEX2Server

APEX2Server is operated on the computer that controls the instrument. It provides functionality for quickly centering a crystal and determining its diffraction quality.

APEX2Server contains two main modules: **Align Crystal** and **Simple Scans** (see Figure 3.2). APEX2Server is covered in more detail in Chapter 5.

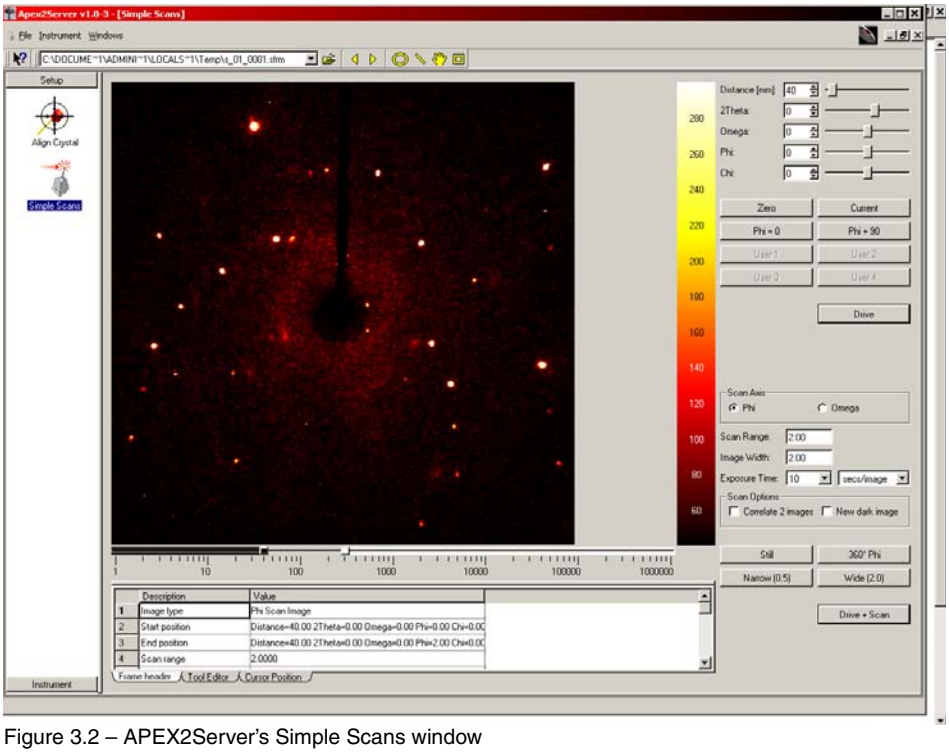


Figure 3.2 – APEX2Server's Simple Scans window

3.2.2 Bruker Instrument Service (BIS)

BIS acts as a server to the client programs APEX2Server, APEX2, and BCP, providing a link between the hardware and software. Once a connection is established, BIS executes hardware commands sent by the client programs. BIS can also be used as a service tool, displaying diagnostic messages during operation.

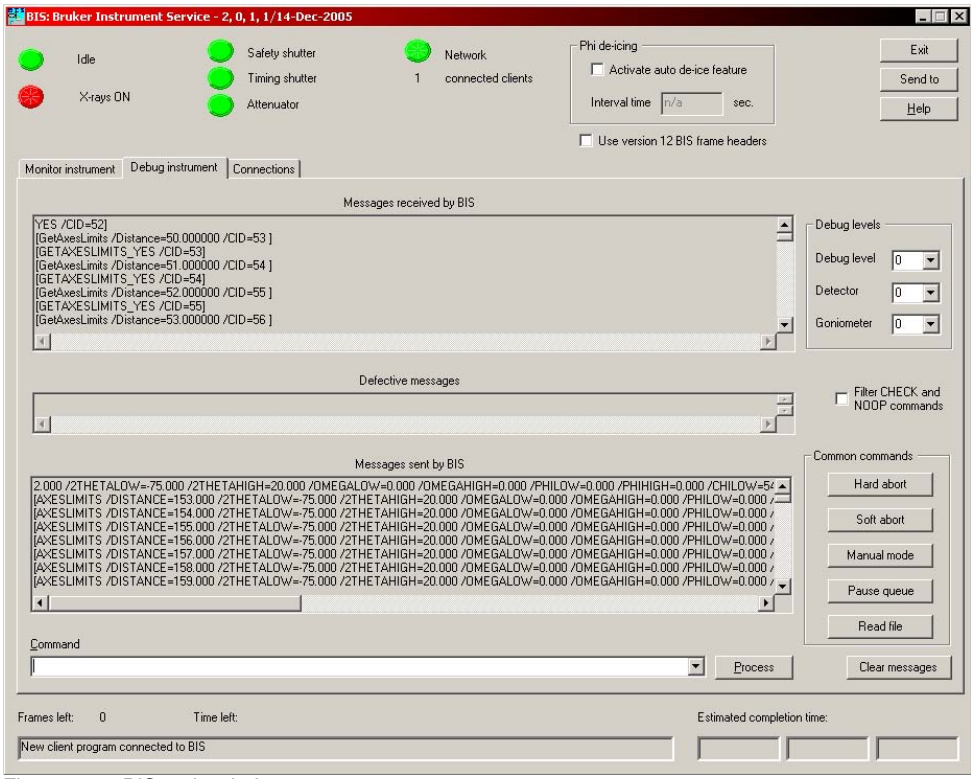


Figure 3.3 – BIS main window

3.3 Client Functions

3.3.1 APEX2 Program

The APEX2 program is a GUI with multiple plug-ins, or *modules*, for different aspects of an experiment. The APEX2 program includes a sample database that stores relevant data from each step in the experiment. Details of the functions available in the GUI will be explained in more detail later in this User Manual.

3.3.2 Bruker Configuration Program (BCP)

BCP is used to configure BIS, as well as to provide instrument control and alignment tools.

From the BCP menu (see Figure 3.4), default parameters for specific hardware elements can be updated. BCP writes these values to the file BrukerInstrument.ini, which is read by BIS. When changes are saved in BCP, BIS automatically reads in the new values. See M86-Exx043 BCP and BIS Installation Notes, in addition to the online help within BCP, for more information.

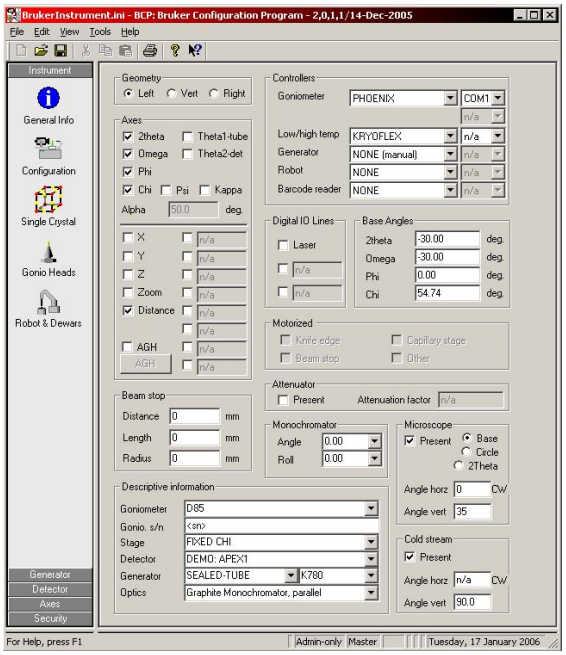


Figure 3.4 – BCP main window (Configuration screen)

3.3.3 The Sample Database

The sample database is used for the storage of data generated by the APEX2 Suite. It is designed to transparently handle data from all of the APEX2 modules without intervention from the user.

The sample database is used internally by the APEX2 Suite and is not available for user customization or manipulation. It is automatically started on computer startup, and it is automatically stopped before the computer is shut down.

Structure of the Sample Database

The tables in the database are divided into four categories.

First, there are tables with users' data, such as user names, passwords, and contact information. These tables provide information about a user's working group and information about for whom the sample is scanned. The administrator assigns each user to a working group or groups (See M86-Exx092 User Manager User Manual for more information).

The second set of tables contains data about the sample. These tables contain all of the sample data required by APEX2's various modules. Data with a single value per sample are contained in a table called "samples." Preliminary information about the sample, such as the name, ID, and color, are stored in the "samples" table as well. Data with multiple values per sample are contained in other tables linked to the "samples" table.

A third set of tables contains the values of standard crystallographic data, such as Laue classes, space groups, and point groups. These tables are referenced by the sample data and are not modified in any way by the user.

Finally, there are a few miscellaneous tables dealing with audit trails, version control of the database, and other assorted functions.

3.4 The APEX2 GUI

The APEX2 graphical user interface (GUI) has one main window (see Figure 3.5).

This window is divided into five sections:

- Title Bar
- Menu Bar
- Tool Icon Bar
- Task Bar
- Task Display Area

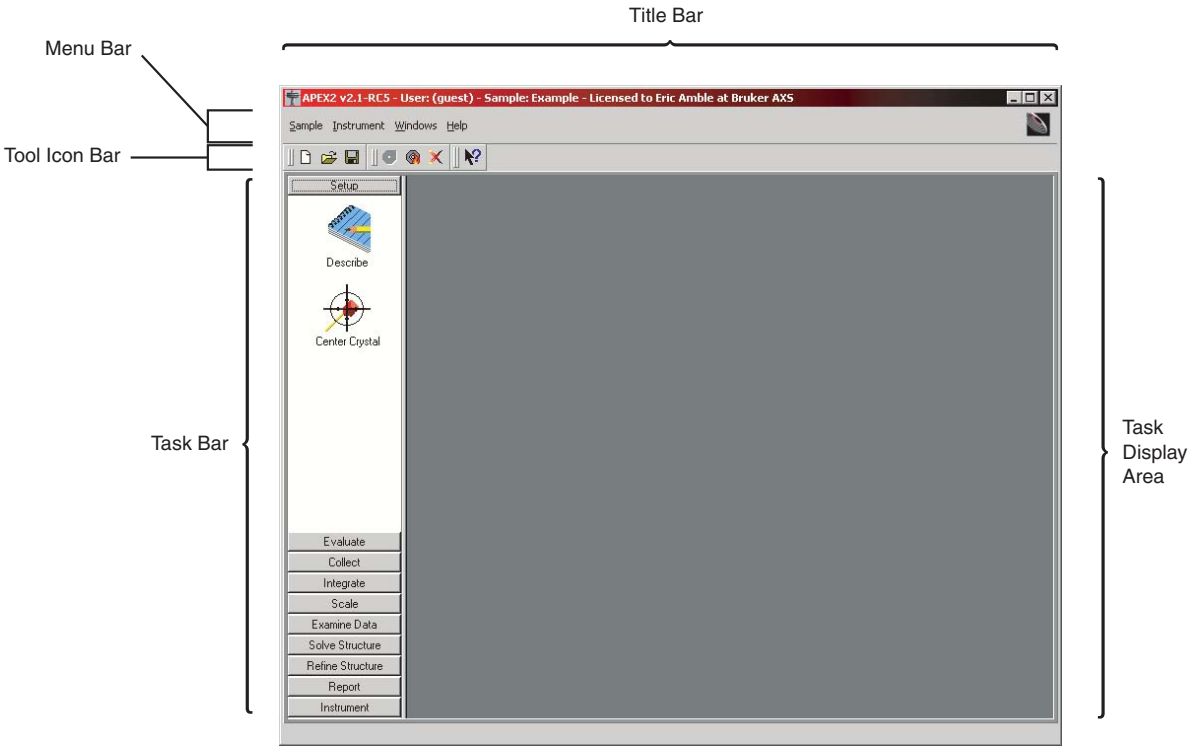


Figure 3.5 – APEX2 GUI

NOTE: Throughout the APEX2 user interface, input fields with pink backgrounds indicate invalid entries. Disabled (greyed-out) fields indicate that a feature is not available, i.e., not supported or dependent upon the instrument configuration.

3.4.1 Title Bar

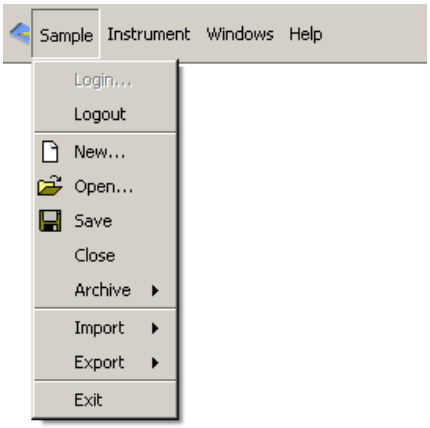
The Title Bar displays the name and version of the software, user name, sample name, license type, and option currently selected in the Task Bar. The right edge of the Title Bar also contains the three buttons  used by Windows for minimizing, restoring, and closing the APEX2 main window.

3.4.2 Menu Bar

The Menu Bar provides drop-down menus for a variety of file operations, image tools, and help files.

An icon for the currently active window appears on the left side of the Menu Bar. Clicking on this icon allows you to minimize, restore, resize, close, or move the currently active window.

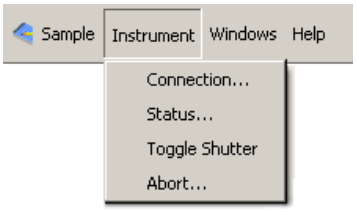
Sample Menu



Sample menu Command	Description
Login...	Opens a dialog window for logging into the Sample Database using your username and password.
Logout	Logs out of the Sample Database.
New...	Lets you create a new sample in the Sample Database.
Open	Opens a previously saved sample.
Save	Saves the current sample.
Close	Closes the current sample.
Archive	Opens a dialog for archiving samples in the Sample Database (to CD or .zip files). Previously archived samples can also be restored using this dialog.
Import	Allows you to import crystal information contained in a .p4p or .spin file into the Sample Database.
Export	Allows you to export crystal data for the current sample as a .p4p file.
Exit	Exits the application. This menu item has the same function as the button in the corner of the Title Bar.

Table 3.1 – Sample menu commands

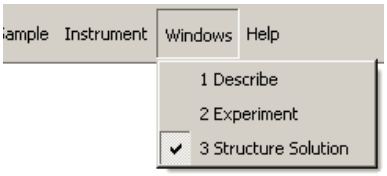
Instrument Menu



Instrument menu Command	Description
Connection...	Opens a connection to a server computer running BIS. If connected already, select this menu to disconnect.
Status...	Opens a window showing a summary of the current status of the instrument and your connection to it.
Toggle Shutter	Instructs BIS to send a shutter open or close command to the instrument. If all interlocks and safety circuits are satisfied, the shutter will open and close.
Abort...	Stops a currently-running data set.

Table 3.2 – Instrument menu commands

Windows Menu



Windows menu Command	Description
Window selection	Displays a list of active windows. Any of these windows may be brought to the front by clicking its title in the Windows menu.

Table 3.3 – Windows menu commands

Help Menu

Help menu Command	Description
About APEX2...	Displays version and copyright information for APEX2.

Table 3.4 – Help menu commands

3.4.3 Tool Icon Bar

Some icons on the Tool Icon Bar provide shortcuts to the options available through the Menu Bar.

Icon	Description
	Create a new entry in the sample database.
	Open an existing entry in the sample database.
	Save the current information to the sample database.
	Open the dialog for restoring a previously archived sample.
	Open the dialog for archiving the current sample.
	Delete samples from the database.
	“What’s this?” Context-sensitive help.

Table 3.5 – Tool Icon Bar: Menu Bar shortcuts

Other icons on the Tool Icon Bar are visible only when a frame is displayed.

Icon	Description
	Open a frame.
	Select the first frame in a run.
	Select previous frame.
	Sequentially display frames in reverse as a movie.
	If displaying frames as a movie, stop the movie.
	Sequentially display frames as a movie.
	Select next frame.
	Select the last frame in a run.
	Adjust the rate of display when viewing frames as a movie.
	Go down one run.
	Go up one run.
	Draw a resolution circle.
	Draw a plotting line.
	Change the part of the frame displayed while zoomed in.
	Select a region of the frame.

Table 3.6 – Tool Icon Bar: displayed frame controls

3.4.4 Task Bar

The Task Bar provides menus for all of the functions available in the APEX2 Suite.

NOTE: If the Task Bar cannot be seen, there is no open sample. Click **Sample > New...** or **Sample > Open...** to open or create a new sample.

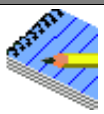
Setup	
	Describe – Specify crystal size, color, shape, etc.
	Center Crystal – Perform crystal centering functions.

Table 3.7 – Setup

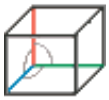
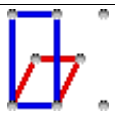
Evaluate	
	Determine Unit Cell – Determine unit cell and Bravais lattice type.
	Transform Unit Cell – Transform a unit cell.

Table 3.8 – Evaluate

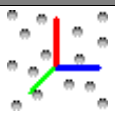
Evaluate	
	Reciprocal Lattice Viewer – 3D visualization of the lattice projected in reciprocal space.
	View Images – View and analyze diffraction images.

Table 3.8 – Evaluate

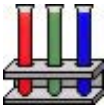
Collect	
	Data Collection Strategy – Simulate data collection and determine strategy.
	Experiment – Sequence editor for data collection experiments.
	Oriented Scans – Measure diffraction frames with the crystal aligned along axes.

Table 3.9 – Collect

Integrate	
	Integrate Images – Integrate diffraction data.

Table 3.10 – Integrate

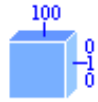
Scale	
	Crystal Faces – Determine Miller indices and distances of single-crystal faces.
	Scale – Scale intensities and perform absorption correction.

Table 3.11 – Scale

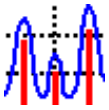
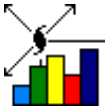
Examine Data	
	Phase ID (optional) – Display and integrate single and multiple frames.
	Space Groups and Statistics – Determine the space group.
	Compare Unit Cells – Compare unit cells to find twin laws.
	Precession Images – Create precession images based on measured frames.
	Space Group Determination – Run XPREP.

Table 3.12 – Examine Data

Examine Data	
	Find a Reflection – Find a reflection in measured frames.

Table 3.12 – Examine Data

Solve Structure	
	AutoStructure (optional) – Automatic solution and refinement with minimum user input.
	Structure Solution – Solve the structure.
	View Molecule – Visualize the molecule in 3D.

Table 3.13 – Solve Structure

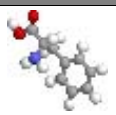
Refine Structure	
	Structure Refinement – Run XP, XL, or XSHELL.

Table 3.14 – Refine Structure

Report	
	Generate Report – Run XCIF to generate a report.

Table 3.15 – Report

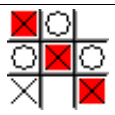
Instrument	
	System Calibration – Make calibration measurements.
	Unwarp and convert images – Convert frames between formats.
	Play Tic Tac Toe – Play a game of Tic Tac Toe!

Table 3.16 – Instrument

Stack Bar And Tree View

Right-click in the Task Bar to choose one of two views: **Stack Bar** or **Tree View**.

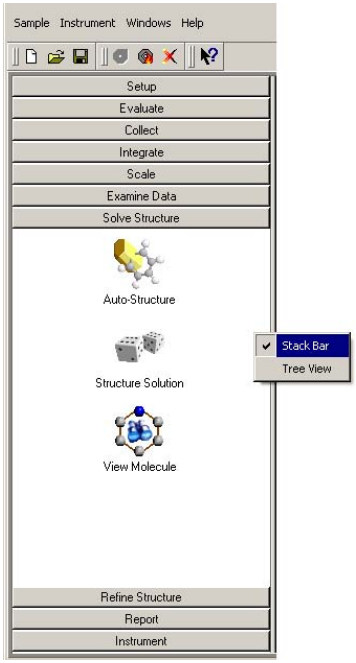


Figure 3.6 – Stack Bar view

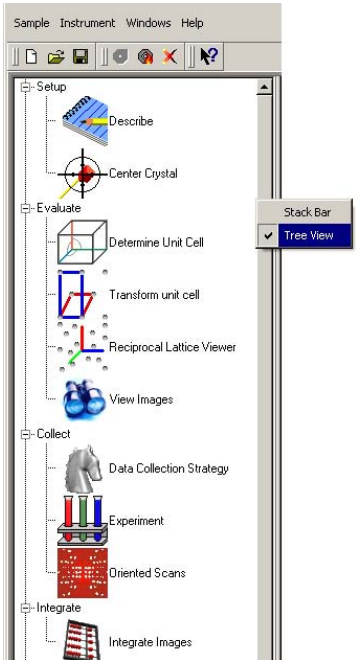


Figure 3.7 – Tree View

In Stack Bar view, click one of the Task Bar sections labeled **Setup**, **Evaluate**, **Collect**, **Integrate**, **Scale**, **Examine Data**, **Solve Structure**, **Refine Structure**, **Report**, or **Instrument** to display only the icons belonging to that section. All other Task Bar icons will be hidden.

In Tree view, sections may be expanded to display their Task Bar icons by clicking the plus or minus sign next to the section name.

3.4.5 Task Display Area

The Task Display Area is the main area for tasks, user input, and selected output. This area displays images, the reflections used in indexing, and observed and predicted diffraction patterns. It also displays the runs for data collection and solution and refinement.

NOTE: In APEX2 Version 2, XShell refinement and XCIF report generation do not use the Task Display Area; they open in new windows. All other modules open in the Task Display Area of APEX2's Main Window.

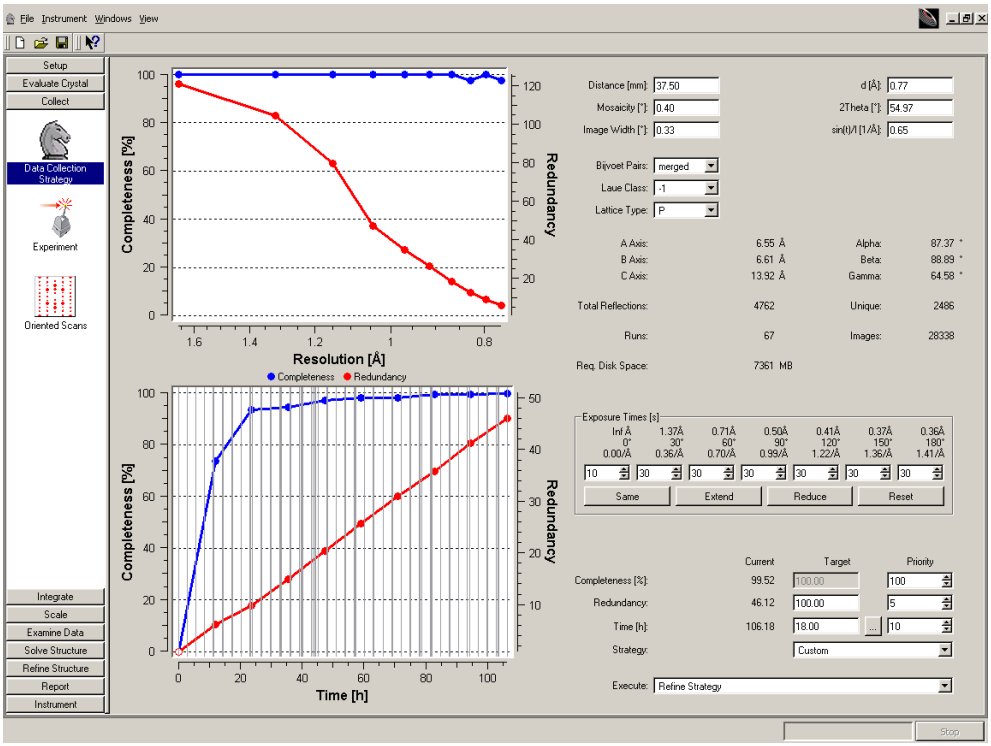


Figure 3.8 – Task Display area showing the Data Collection Strategy module

4 Program Startup and Shutdown

As discussed in Chapter 3, the APEX2 Suite is composed of several programs. All of the programs are started in a similar fashion. For ease of use there is usually a desktop icon for the folder containing these programs, and desktop shortcuts linked directly to these programs. However, the **Start > Programs > Bruker AXS** path is always available. This more explicit method will be used in this discussion.

4.1 Access to BCP and APEX2

Bruker software allows different user accounts to have different levels of access to the instrument:

- Service
- Security
- Administrator
- User
- Non-user

NOTE: You need Bruker Instrument Administrator rights to run BCP and Bruker Instrument User rights to run APEX2. Typically, your system administrator will already have your user account configured or you will be using the default accounts as provided by Bruker AXS.

4.2 Online and Offline Operation

APEX2 can operate in either *online* or *offline* mode.

In online mode, APEX2 is connected to BIS in order to communicate with the instrument for performing experiments. To use APEX2 in online mode, you must start BIS, APEX2, and optionally, VIDEO.

In offline mode, APEX2 is not connected to the instrument, but is still able to analyze and interpret existing data. To use APEX2 in offline mode, you only need to start APEX2.

4.3 Startup

4.3.1 Starting Bruker Instrument Service (BIS)

BIS is the link between the hardware and the APEX2 software. The BIS software executes hardware commands issued by APEX2.

1. To start BIS, select **Start > Programs > Bruker AXS > Administration > BIS** (Figure 4.1).

Figure 4.1 – Starting BIS from the Start menu

2. Once the BIS window appears, you may be asked to confirm the detector distance.

4.3.2 Starting APEX2

APEX2 is used to set up crystallographic experiments and to process crystallographic data.

1. To start APEX2, select **Start > Programs > Bruker AXS > APEX2**.

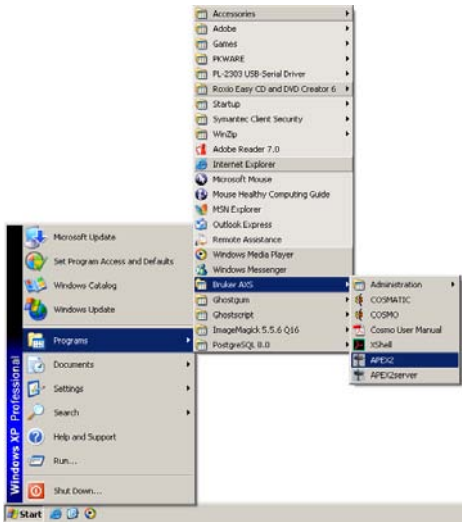


Figure 4.2 – Starting APEX2 from the Start menu

2. The Login window appears automatically when you start APEX2. At the Login window, enter your user name and password. As shipped by Bruker AXS, the default account is “guest” with password “guest.” Click **OK**.

Figure 4.3 – Login dialog

3. For online operation within APEX2, select **Instrument > Connection...**. In the Instrument Connection window, enter the instrument host name. The window shows the default host. Click **Connect**.

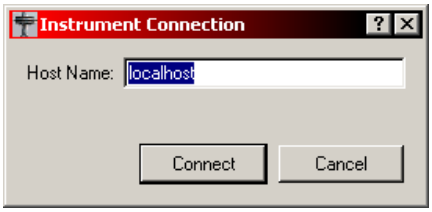


Figure 4.4 – Instrument connection dialog

NOTE: By editing the “bn-config.py” configuration file, APEX2 may be configured to start without displaying the Instrument Connection window. In this mode, APEX2 automatically connects to the user-specified IP address or DNS name of a computer running BIS. More information about the bn-config.py file may be found in M86-Exx087 APEX2 and PROTEUM2 Installation Notes and in Appendix D.

You are now ready to begin using APEX2.

4.4 License Considerations

You need a software license to activate your purchased software packages, options, and/or features.

APEX2 requires a valid license file to operate. This file, “bn-license.dat”, must be present in the root directory of drive C:\ for the software to start properly.

If the license file is not present or has expired, an error window will appear when you try to start APEX2. This window prompts you to run the License Manager application to obtain a valid license for APEX2.

For more information on licenses and License Manager, refer to M88-Exx099 License Manager User Manual.

Figure 4.5 – “No valid license” window

4.5 Shutdown

4.5.1 Shutting Down APEX2

1. First, log out of the sample database by going to **Sample > Logout** within APEX2. In the “Closing Sample” window, select whether to save or discard the changes to the sample database.

Figure 4.6 – “Closing Sample” window

NOTE: The “Closing Sample” window appears even if no apparent changes were made to the sample database because the date and time of the last access are held in the sample database and differ from the current date and time.

2. Within APEX2, select **Sample > Exit**. APEX2 closes.

4.5.2 Shutting Down BIS

1. To stop BIS, click the **Exit** button in the upper right-hand corner of the BIS window. You may be asked to place the generator into Standby mode.
2. At the confirmation dialog, click **OK**. BIS closes.

5 Crystal Centering and Screening

We are now ready to begin the process of data collection with the instrument. It is assumed that your system administrator has set up the system properly and that all system default parameters have been set appropriately.

The data collection process is divided into five steps, which will be covered in Chapter 5 and Chapter 6.

The steps in Chapter 5 are performed using the APEX2Server software on the computer controlling the instrument (i.e., the Server in a dual-computer setup).

The steps in Chapter 6 are performed using the APEX2 program (on the Client computer in a dual-computer setup).

See Chapter 5 for:

- Centering the crystal on the diffractometer (using APEX2Server's **Center Crystal** module)
- Crystal quality check (using APEX2Server's **Simple Scans** module)

See Chapter 6 for:

- Cell determination (using APEX2's **Determine Unit Cell** module)
- Data collection setup (using APEX2's **Data Collection Strategy** module)
- Data collection (using APEX2's **Experiment** module)

5.1 Start APEX2Server

1. If APEX2Server is not running on the computer controlling the instrument, start it by going to **Start > Programs > Bruker AXS > APEX2Server**.

2. Ensure that APEX2Server is connected to the instrument by checking the **Instrument > Status...** menu.

5.2 Mount the Goniometer Head on the Instrument

1. Open the enclosure doors by pushing either of the rectangular green **Open Door** buttons on the enclosure's side posts. This releases the door locks for approximately five seconds. While the door locks are released, pull outward on one or both of the handles to physically open the doors.

Figure 5.1 – D8 Enclosure **Open Door** button

2. Under **Setup** in APEX2Server's Task Bar, click **Center Crystal**. The centering buttons (Figure 5.2) appear and the VIDEO program window opens.

Figure 5.2 – Centering buttons

NOTE: If the VIDEO window does not stay on top of the APEX2Server window, select **Grab > On top** from the VIDEO program Menu Bar.

The bottom five buttons in Figure 5.2, **Mount**, **Left**, **Top**, **Right**, and **Center**, drive the goniometer to various pre-defined positions that are designed to simplify crystal centering. The two large square buttons rotate phi by either 90 or 180 degrees.

3. Click **Mount** to drive the goniometer to a convenient position for mounting the goniometer head.
4. Carefully remove the goniometer head containing the crystal from its case.

CAUTION

Use extreme care when handling the goniometer head to prevent damage to the crystal on the end of the small glass fiber.

5. Place the goniometer head onto its base on the phi drive. Line up the slot on the bottom of the goniometer head with the pin on the mounting base (see Figure 5.3).

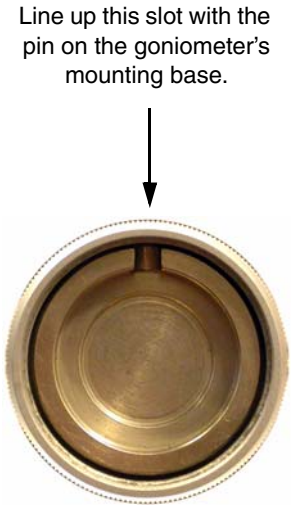


Figure 5.3 – View of the bottom of the goniometer head

6. Screw the goniometer head collar to the base so that the head does not move. Do not overtighten it (finger-tighten only).

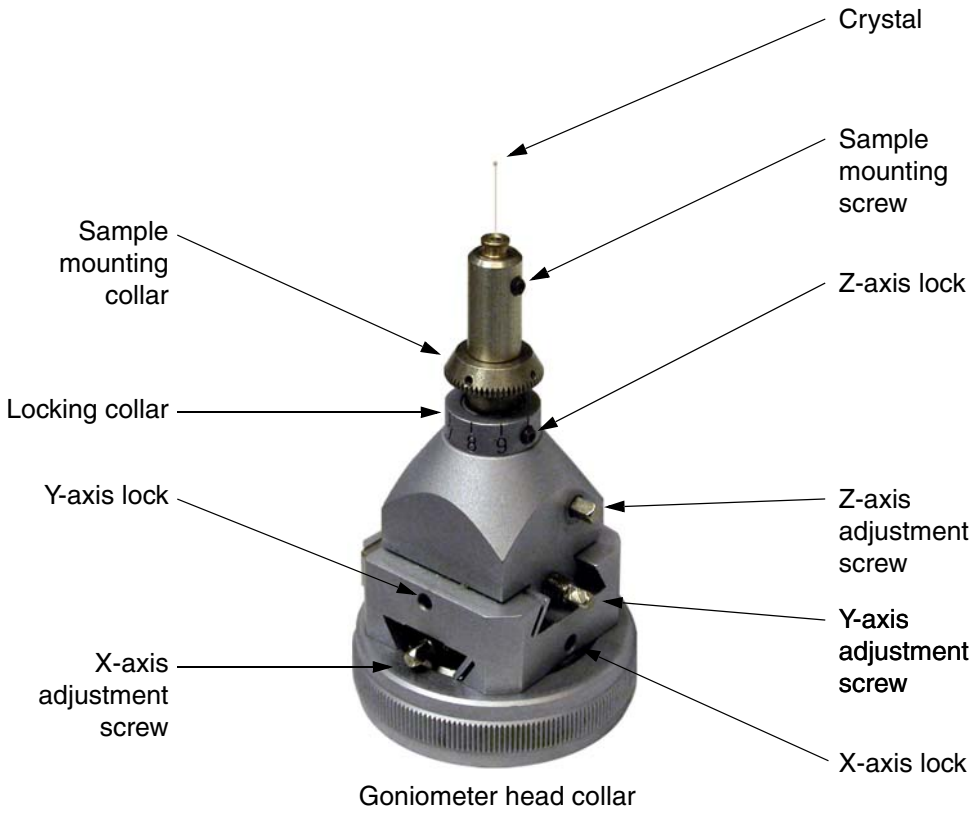


Figure 5.4 – Huber goniometer head in detail

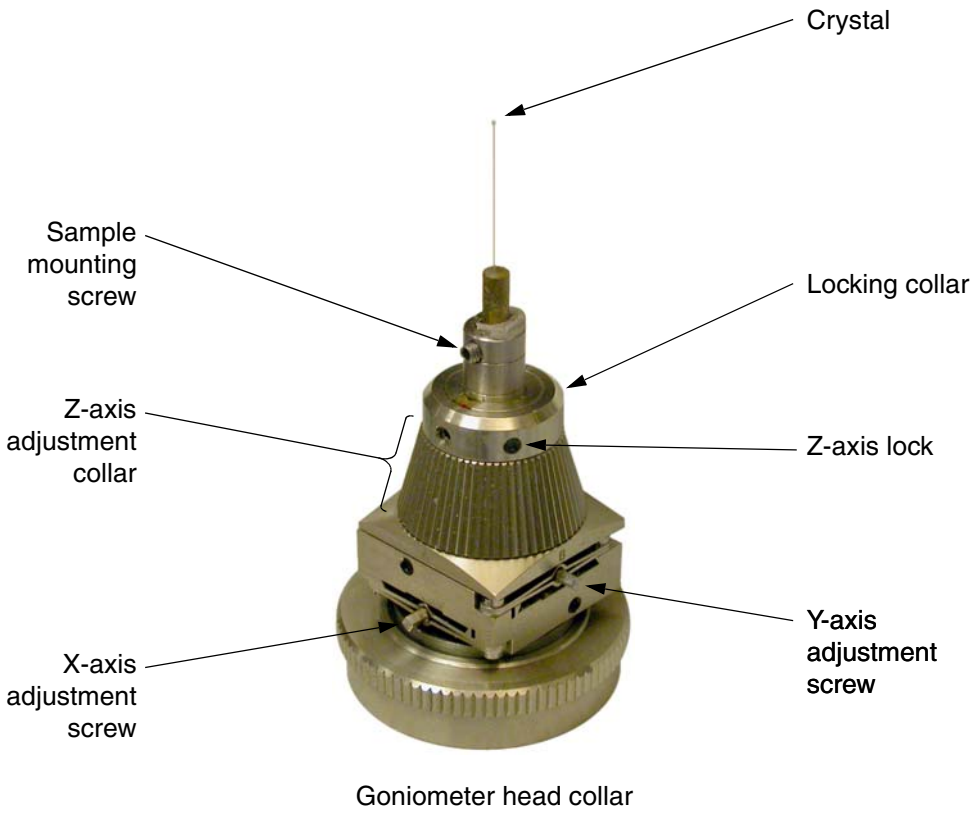


Figure 5.5 – Nonius goniometer head in detail

5.3 Center the Crystal

To obtain accurate unit cell dimensions and collect good quality data, position the crystal in the center of the X-ray beam and maintain this position for the entire experiment.

Your video microscope should be aligned so that the reticle of the video microscope coincides with the center of the goniometer and the center of the X-ray beam (for instructions on aligning the microscope to the center of the instrument, see M86-Exx024 Microscope Focus and Sample Alignment).

If the microscope is not centered, you can still center the crystal. A successfully centered crystal stays in the same place in the microscope's field of view in all orientations.

To center the crystal on a Kappa APEX II system, see Section 5.3.1.

To center the crystal on a SMART APEX II system, see Section 5.3.2.

NOTE: Use the thin end of the goniometer wrench to unlock the X, Y and Z locks at the beginning of the centering process and to lock them at the end. Locking needs only a very slight touch. The other end of the wrench is used to move the adjustment sleds. Do not overtighten the locks.

NOTE: Centering is often easier if the crystal is rotated to give a good view before the actual centering process is started (e.g., down an edge for a plate). To do this, click **Center** to drive to the initial centering position, loosen the crystal mounting screw, rotate the crystal to a suitable orientation, and then tighten the screw again.

5.3.1 For Kappa APEX II Systems

1. Click the **Center** button. The goniometer head drives so that its Y and Z translation axes are positioned perpendicular to the microscope. The Y- and Z-axis adjustment screws should be facing the front of the diffractometer. If they are not, click **Spin Phi 180**.

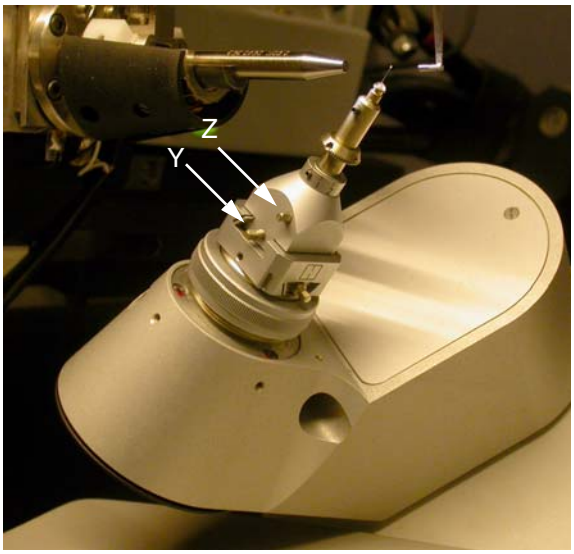


Figure 5.6 – Center position, Y- and Z-axis adjustment screws

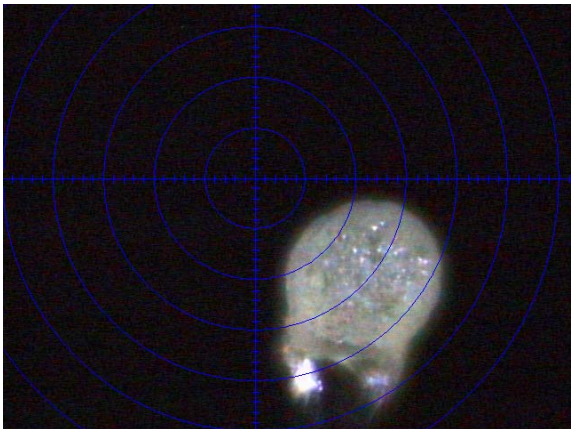


Figure 5.7 – Crystal initially mounted

2. Center the crystal in the video microscope reticle by making adjustments to the Y- and Z-axis adjustment screws.

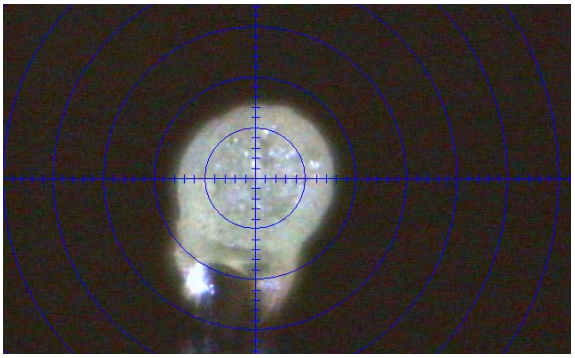


Figure 5.8 – Centered crystal after Y- and Z-axis adjustments

3. Click **Spin Phi 180** and check that, after rotating 180°, the crystal appears in the same position in the microscope reticle. If the crystal does not appear in the same position:

   3.1 Use the adjustment screw facing you to remove **half** of the difference shown in the reticle (Figures 5.9 and 5.10).

   3.2 Click **Spin Phi 180**.

   3.3 Repeat steps 3.1 and 3.2 until you are satisfied that the crystal remains in the same position in the microscope reticle when rotated by 180°.

NOTE: If the crystal consistently fails to appear in the same position when rotated by 180°, the position of the microscope reticle may need adjustment. For more information, refer to M86-Exx024 Microscope Focus and Sample Alignment.

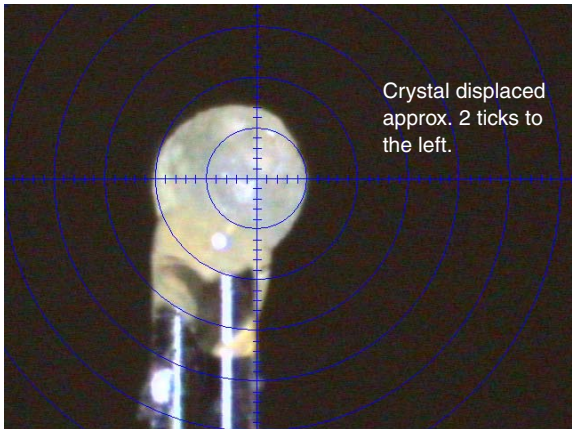


Figure 5.9 – Example: error in Y-axis

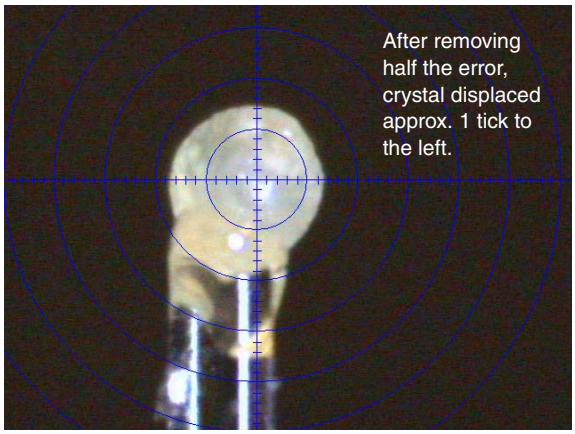


Figure 5.10 – Example: error removed by half

4. Click **Spin Phi 90**. Phi rotates so that the X-axis adjustment is facing forward. Any error in the crystal's position along the X-axis will now appear in the microscope.

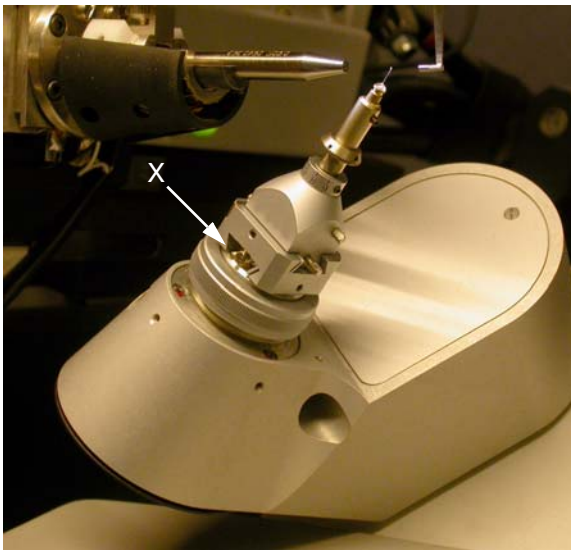


Figure 5.11 – Center position, X-axis adjustment screw

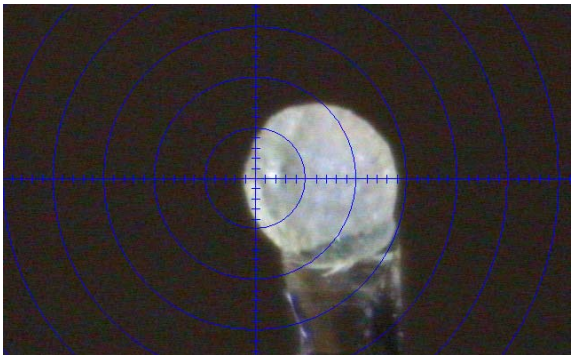


Figure 5.12 – X-axis error after spinning phi 90°

5. Center the crystal in the video microscope reticle by making adjustments to the X-axis adjustment screw.

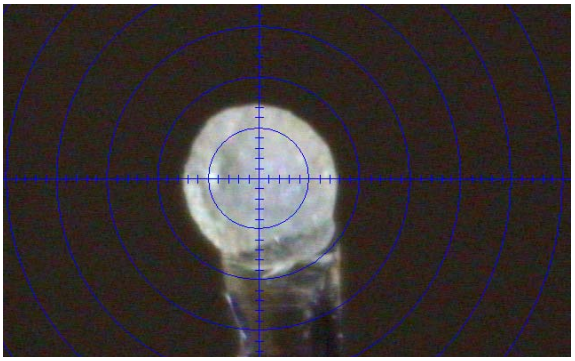


Figure 5.13 – Centered crystal after X-axis adjustments

6. Alternately click **Spin Phi 180** and **Spin Phi 90** to verify that the crystal stays in the same place in the microscope reticle through all motions of phi. If the crystal fails to stay in the same position as phi is rotated, make adjustments to the axes by repeatedly removing half the error as in step 3.
7. Click the **Left** button. The goniometer drives to place the fiber horizontal and to the left. Note the height of the crystal in the video microscope reticle.

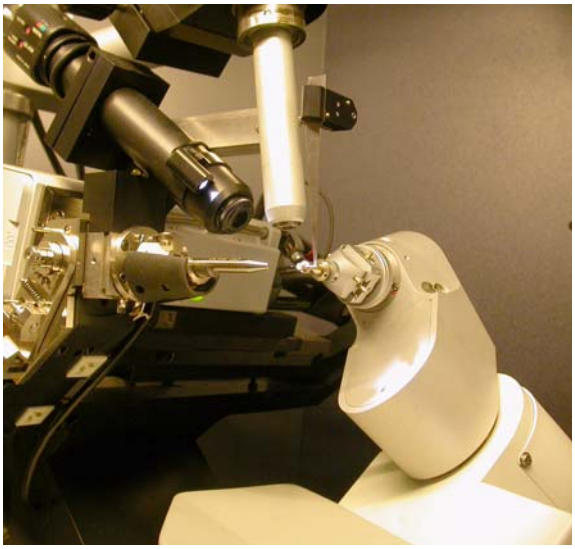


Figure 5.14 – Left position

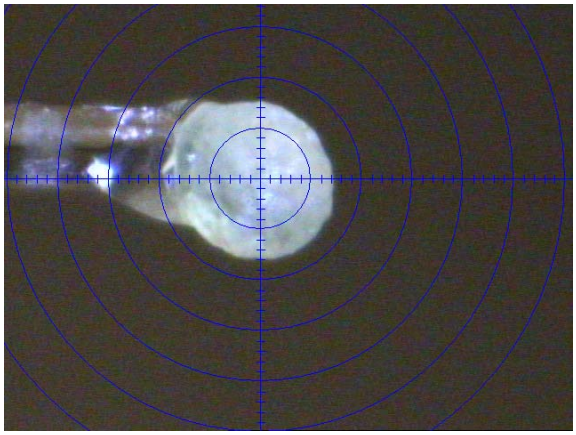


Figure 5.15 – Crystal in left position

8. Click the **Right** button. The goniometer drives to place the fiber horizontal and to the right.

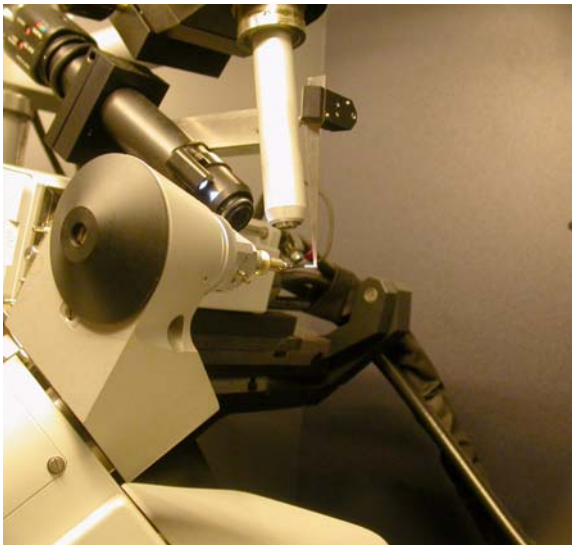


Figure 5.16 – Right position

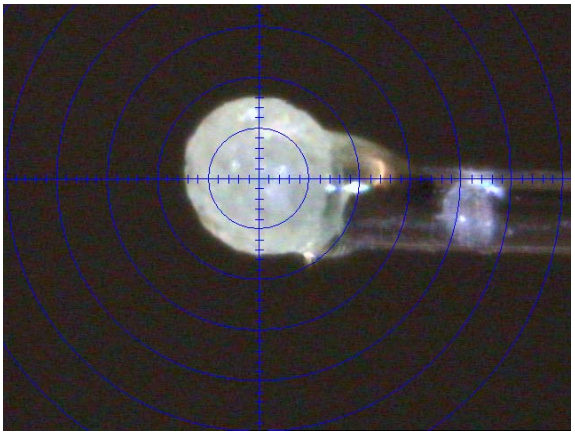


Figure 5.17 – Crystal in right position

9. Check that the crystal height is the same as the height you noted in step 7.

   9.1 If the height is the same, proceed to step 10.

   9.2 If the height is not in the same place, adjust to remove half of the difference, click **Spin Phi 180**, and repeat steps 7 to 9.

10. Click the **Top** button. The goniometer drives so that the phi axis is pointing directly into the microscope.

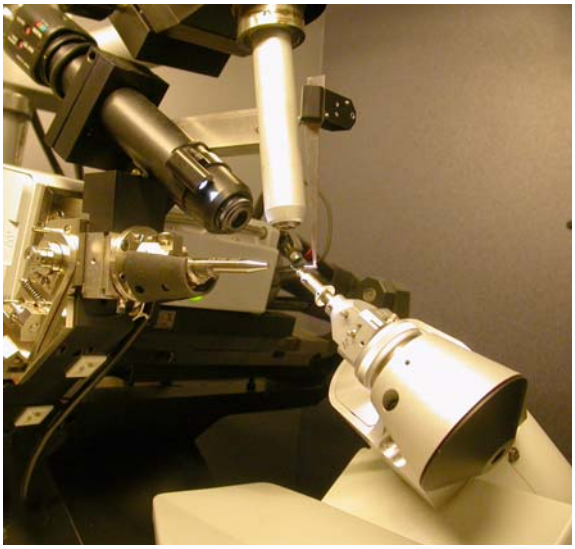


Figure 5.18 – Top position

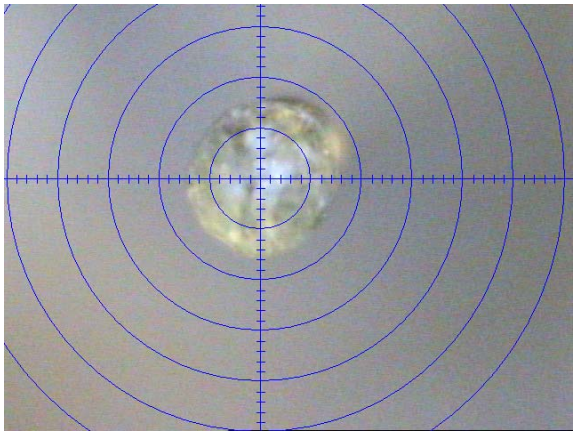


Figure 5.19 – Crystal in top position

11. Click **Spin Phi 180** a few times to verify that the crystal stays in the same position as Phi rotates. If the crystal does not remain centered, go back to step 1.
12. Click **Center** to drive the goniometer back to the center position.

The crystal is now centered on the instrument.

5.3.2 For SMART APEX II Systems

NOTE: If the image of the crystal is difficult to see, illuminate the crystal with a high-intensity lamp and/or temporarily place a light-colored piece of paper on the front of the detector.

1. Click the **Right** button. The crystal and goniometer head drive to a position perpendicular to the microscope. To center the crystal, make adjustments to the height with the Z-axis adjustment.

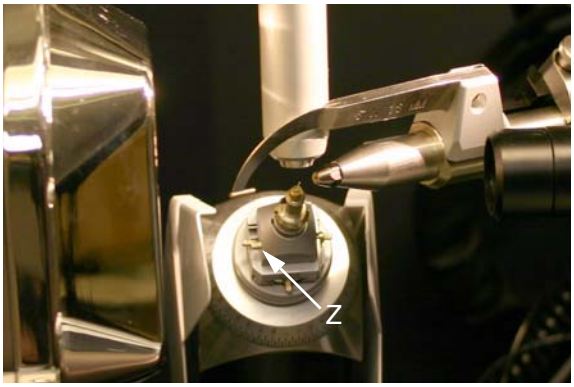


Figure 5.20 – Right position, Z-axis adjustment screw

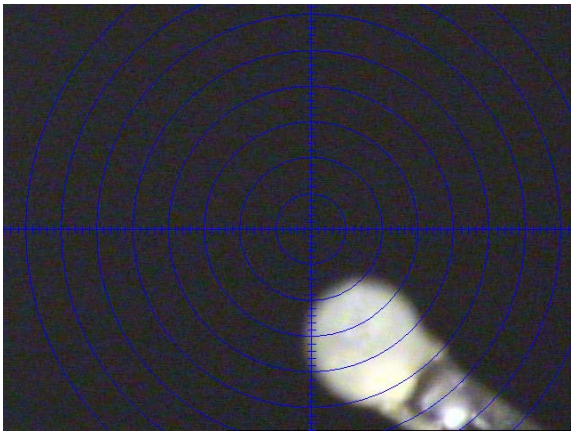


Figure 5.21 – Initial mounted crystal

2. Click the **Center** button. Move the crystal so that it is centered in the microscope reticle by adjusting the X- or Y- axis translation adjustment screw that is perpendicular to the microscope axis and facing you (see Figure 5.4 and Figure 5.5).

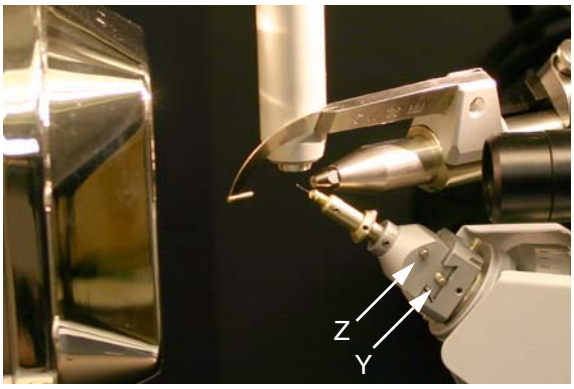


Figure 5.22 – Center position, Y and Z adjustments

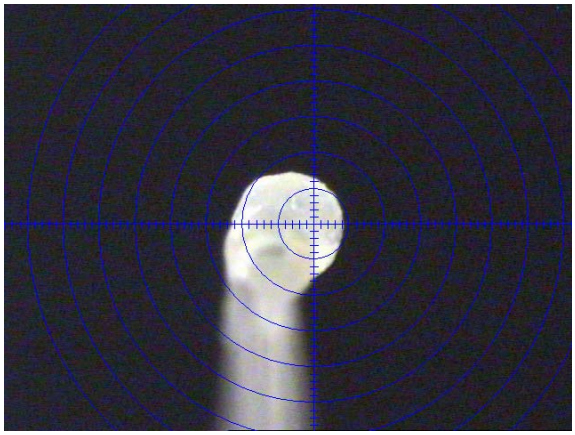


Figure 5.23 – Initial center position before X- or Y-axis adjustment

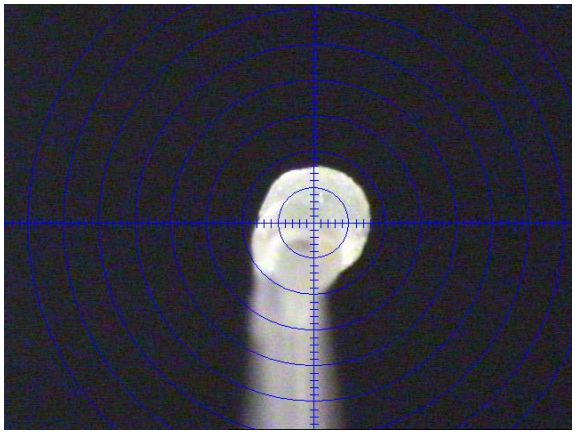


Figure 5.24 – Crystal after X- or Y- adjustments

3. Click **Spin Phi 90**. Remove half of the difference with the adjustment screw that is facing you.

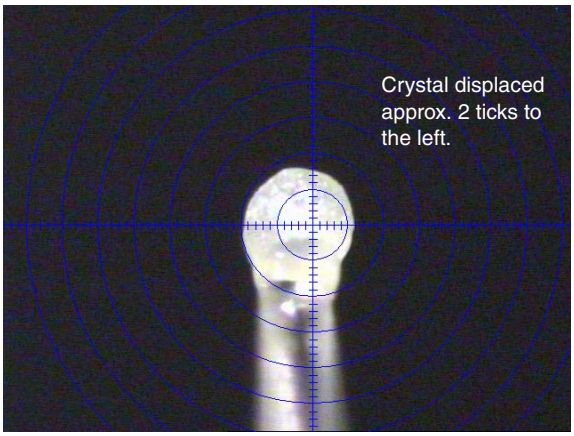


Figure 5.25 – Example: error in X-axis

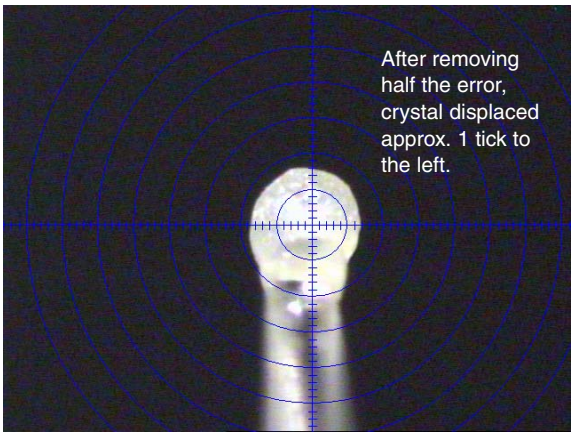


Figure 5.26 – Example: error removed by half

4. Click **Spin Phi 180**. Remove half of the difference with the adjustment screw that is facing you.
5. Click **Spin Phi 180** again.
6. If the crystal is centered, click **Spin Phi 90**.
7. If the crystal is not centered, adjust to remove half of the difference and click **Spin Phi 180**. Repeat until the crystal is centered. Click **Spin Phi 90**.
8. If centered, adjust the height. If not centered, repeat steps 3 through 7 until it is centered.

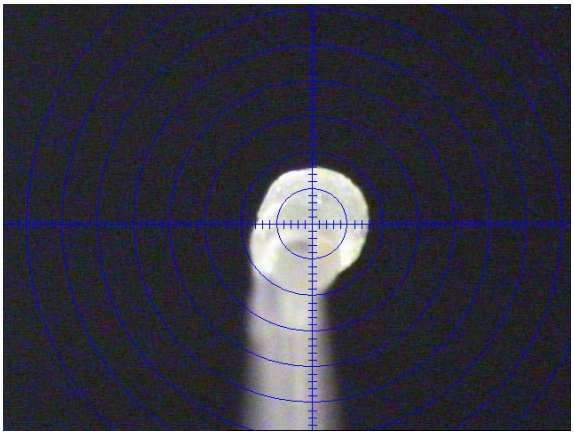


Figure 5.27 – Height adjusted

9. Click the **Left** button. Adjust to remove half of the difference. Adjust the height.

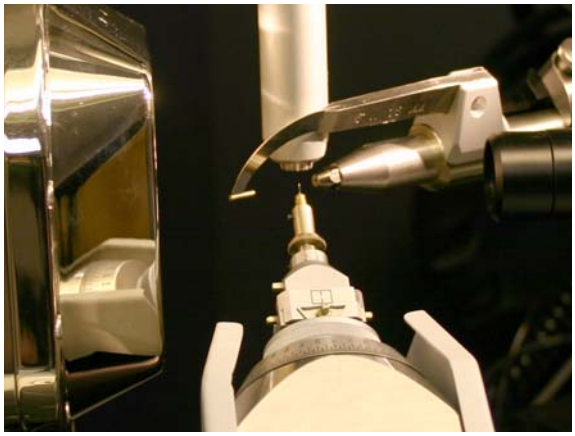


Figure 5.28 – Left position

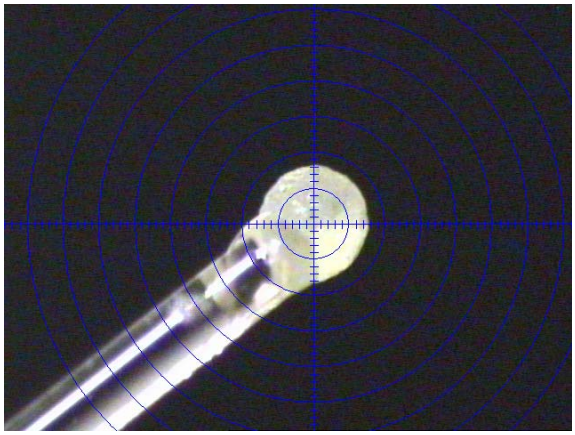


Figure 5.29 – Check height in left position

10. Click the **Right** button. Adjust the height. Adjust to remove half of the difference.

Figure 5.30 – Right position

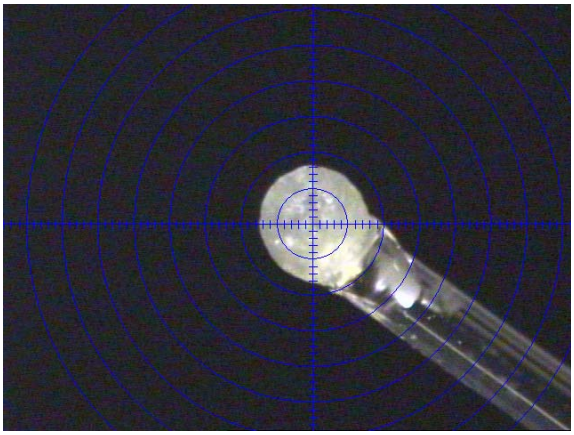


Figure 5.31 – Check height in right position

11. If a height adjustment was made in step 6 or 7, repeat those steps to check the height. If the height is adjusted, repeat steps 2 to 5 to check the centering. If no height adjustment was made, the crystal is centered.

The crystal is now centered on the instrument.

5.4 Simple Scans

APEX2Server's Simple Scans software module provides tools for rapid screening of the crystal to check crystal quality. It allows you to quickly set up scans to measure a 360° phi rotation as well as thin (0.5°) and thick (2.0°) still images.

1. Under **Setup** in APEX2Server's Task Bar, click **Simple Scans**. The menu shown in Figure 5.32 opens.

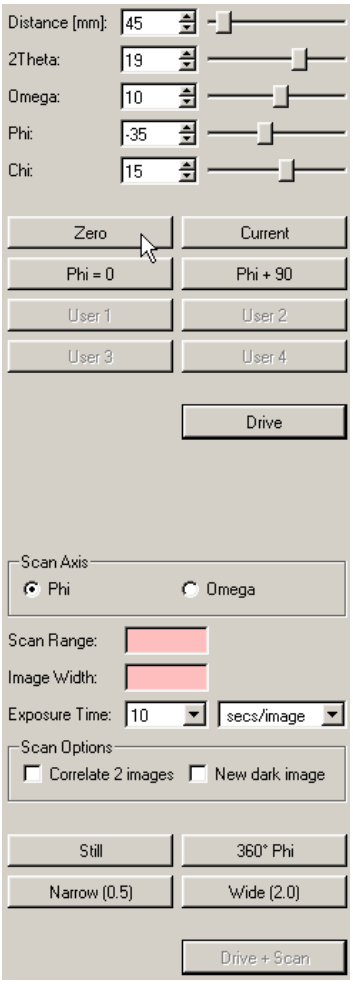


Figure 5.32 – Simple Scans menu

The sliders and data boxes at the top can be used to position the detector.

The buttons in the middle provide easy access to common movements.

There are four possible user-defined buttons.

The **Drive** button initiates the requested movement. If it is gray, an impossible movement has been requested.

The buttons and boxes at the bottom set up scans. In Figure 5.32, the **Drive + Scan** button is grey and therefore inactive because no scan has been requested.

1. Click **Zero** and then **Drive**.
2. Set the distance.
   - 2.1 On Kappa APEX II systems, check that the moveable beamstop is pushed in and set the desired position (typically 45 mm) for Distance in the data window.
   - 2.2 On SMART APEX II systems with movable DX, set the desired position (typically 50 mm) for Distance in the data window.
   - 2.3 On SMART APEX II systems with fixed DX, check that the distance displayed is the same as the actual distance in mm on the detector arm.
3. Click **360° Phi** and set the desired exposure time. The default of 15 seconds is usually sufficient.
4. Click **Drive + Scan**. Since these are evaluation scans, there is no need to request correlated images or new darks. The shutter opens and the phi scan begins. The resulting Phi 360° image is shown in Figure 5.33. The crystal diffracts nicely with lots of sharp spots. Figure 5.37 shows a Phi 360° scan with a bad crystal.

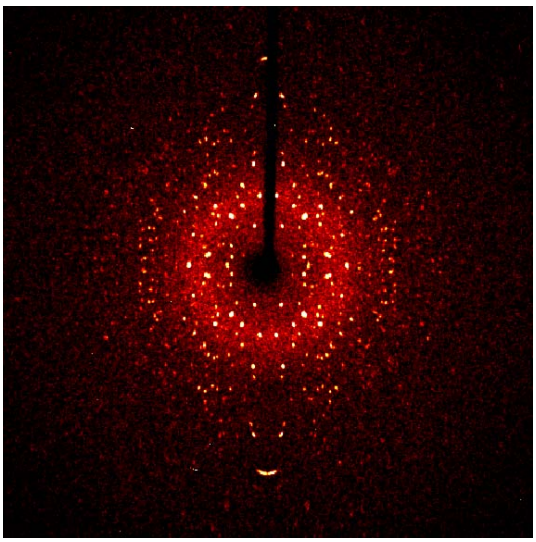


Figure 5.33 – A 360° Phi scan on a good quality crystal

5. Click **Wide (2.0)**. The phi scan range changes to 2°. An exposure time of 5 to 15 seconds is usually sufficient for a wide scan.

6. Click **Drive + Scan**. The resulting 2° scan is shown in Figure 5.34. The spots are sharp and clean. There are no peaks that are very close together. Figure 5.38 shows a 2° scan with a bad crystal.

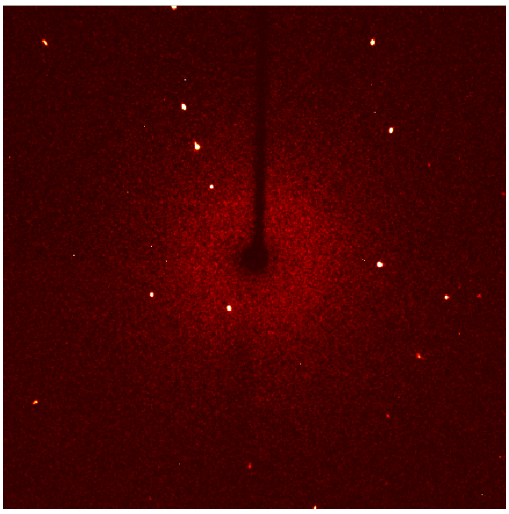


Figure 5.34 – A 2° phi scan on a good quality crystal. The spots' shapes are well-defined and the spots are well-separated.

7. Click **Phi + 90** in the middle row of boxes.
8. Click **Drive + Scan**. The resulting 2° scan is shown in Figure 5.35. This image is measured 90° from the previous one, giving a view of the diffraction pattern from a different (perpendicular) direction. Figure 5.39 gives a similar view for the poor crystal.

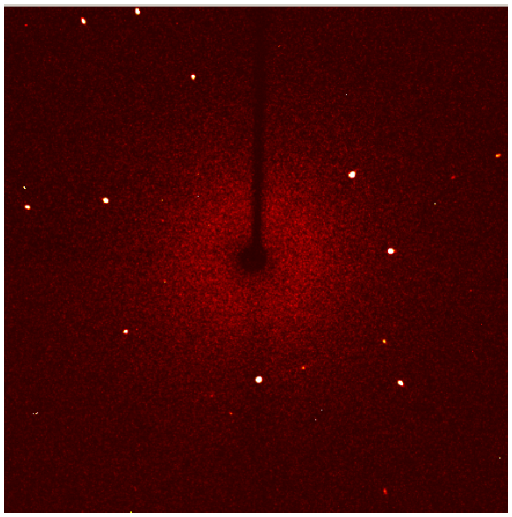


Figure 5.35 – A 2° phi scan at phi = +90° on a good quality crystal. The spots' shapes are well-defined and the spots are well-separated.

9. Set 2-theta to -30°. This will allow evaluation of the diffraction at higher angles.
10. Click **Drive + Scan**. The resulting image is shown in Figure 5.36.

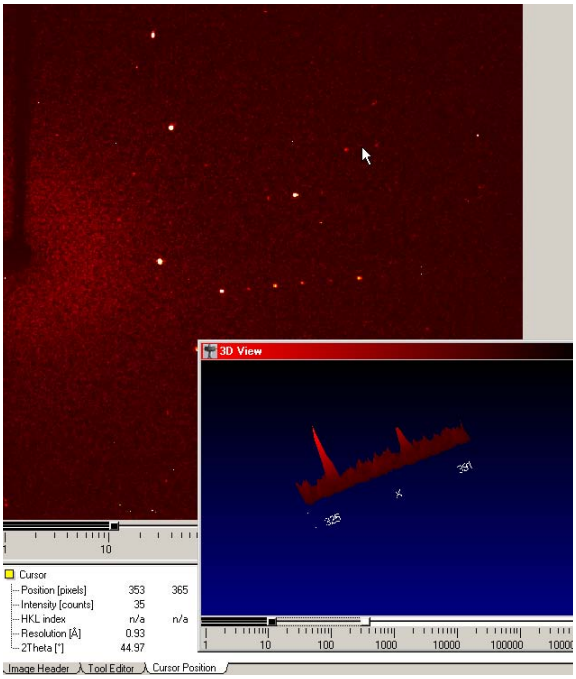


Figure 5.36 – A 2° phi scan on a good quality crystal at 2-theta = -30°. The cursor is pointing to an area between the two reflections shown in the 3D View window. The cursor position tab at the bottom left shows that the resolution is 0.93Å and 2-theta is 45°.

5.5 Examples of Poor Quality Crystals

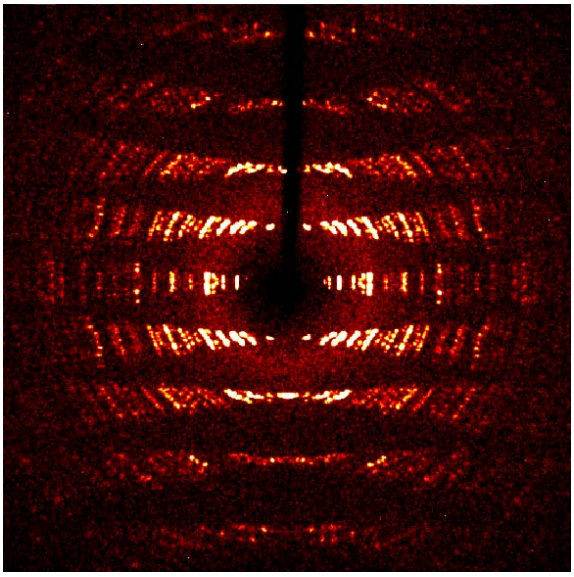


Figure 5.37 – A 360° phi scan on what is likely a poor quality crystal. The spot shape is poor and the spots tend to run together. The obvious bands on the image suggest that the crystal is nearly aligned along an axis; however, spots will always apparently run together in a nearly aligned crystal mounting. Figure 5.38 is much more convincing proof of a poor quality crystal.

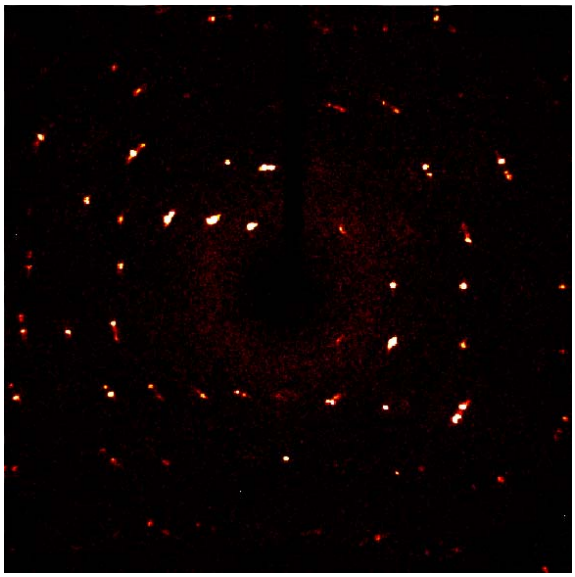


Figure 5.38 – A 2° phi scan on a poor quality crystal. The spot shape is poor and some spots seem split.

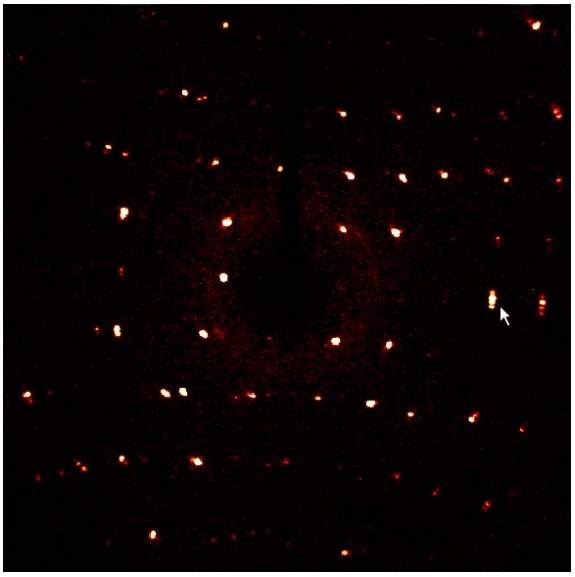


Figure 5.39 – A 2° phi scan on a poor quality crystal at plus 90 in phi. The spot shape is poor and the spots are very close together.

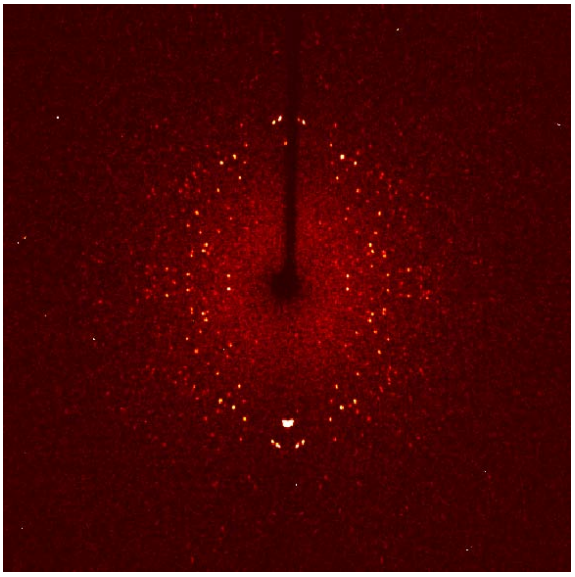


Figure 5.40 – A 360° phi scan on a small crystal. The diffraction power of the crystal is small, but with longer exposure times this is clearly a reasonable candidate for data collection.

6 Data Collection

The data collection process is carried out on the client computer using APEX2. Once data collection is started, exit APEX2 (optional). Data collection will continue regardless.

6.1 Create a New Sample

1. After starting APEX2 and logging in, select **Sample > New...**.
2. In the window that appears, enter the sample name. APEX2 will automatically create a directory for data storage.

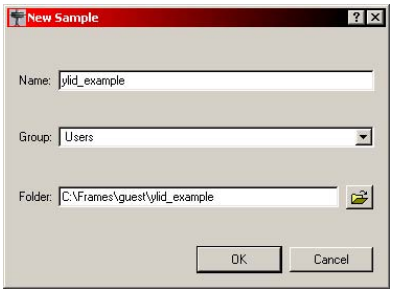


Figure 6.1 – New Sample window

3. Click **OK**. The Task Bar appears with the Setup section open.
4. Click **Describe**.
5. Enter the requested information into the Describe window.

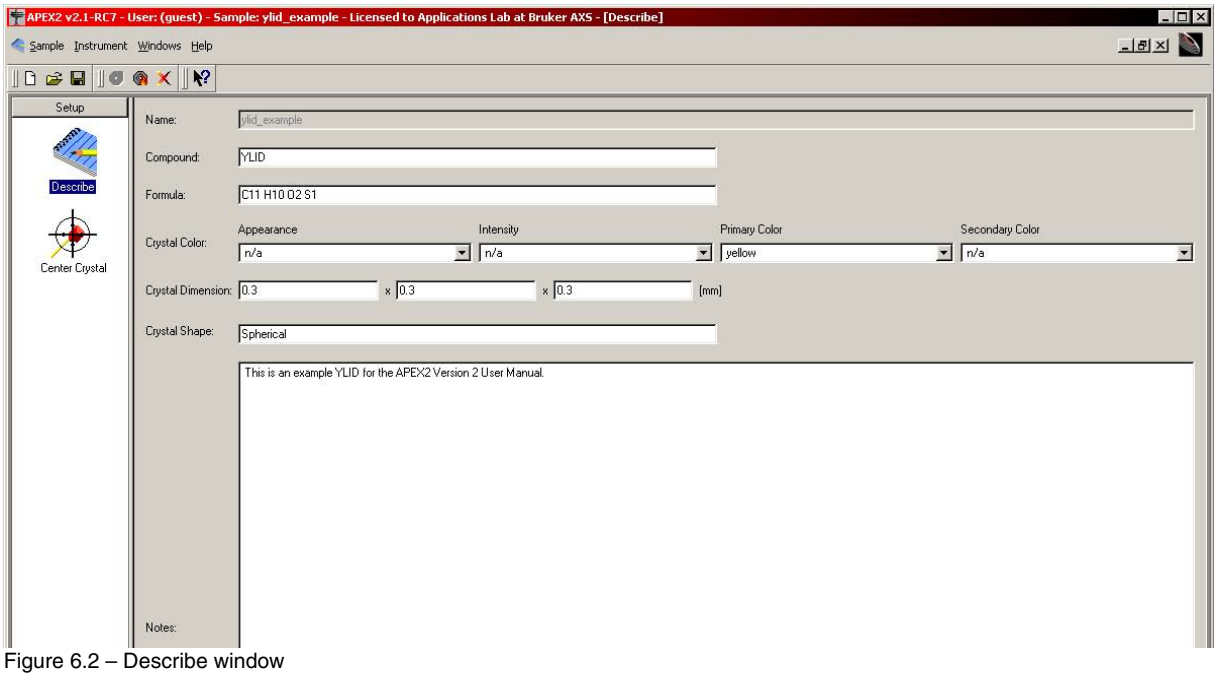


Figure 6.2 – Describe window

6. Close the Describe module by clicking the ✕ button on the right-hand side of the Menu Bar. APEX2 will automatically save the data to the sample database.

6.2 Determine the Unit Cell

Unit cell determination is performed in several steps:

- Data collection
- Harvesting of reflections from collected frames
- Indexing of harvested reflections
- Bravais lattice type determination
- Refinement

In APEX2 Version 2, these steps are integrated in the Determine Unit Cell module (located in APEX2's "Evaluate" category), which also contains a one-click solution for fully-automated unit cell determination.

The upper right-hand corner of the Determine Unit Cell module contains two sections:

- **Automatic Mode** - for fully-automated unit cell determination (Section 6.2.1).
- **Manual Mode** - for unit cell determination with user-defined parameters (Section 6.2.2).

NOTE: The Determine Unit Cell module automatically determines crystal mosaicity and provides a table of exposure time versus diffraction limit (Figure 6.3, lower right-hand corner).

This table is strictly informational and its contents are not used when determining exposure times for a data collection strategy. However, you can use the information in the table to manually set exposure times for your maximum desired resolution.

For more information on the Data Collection Strategy module, refer to Section 6.3.

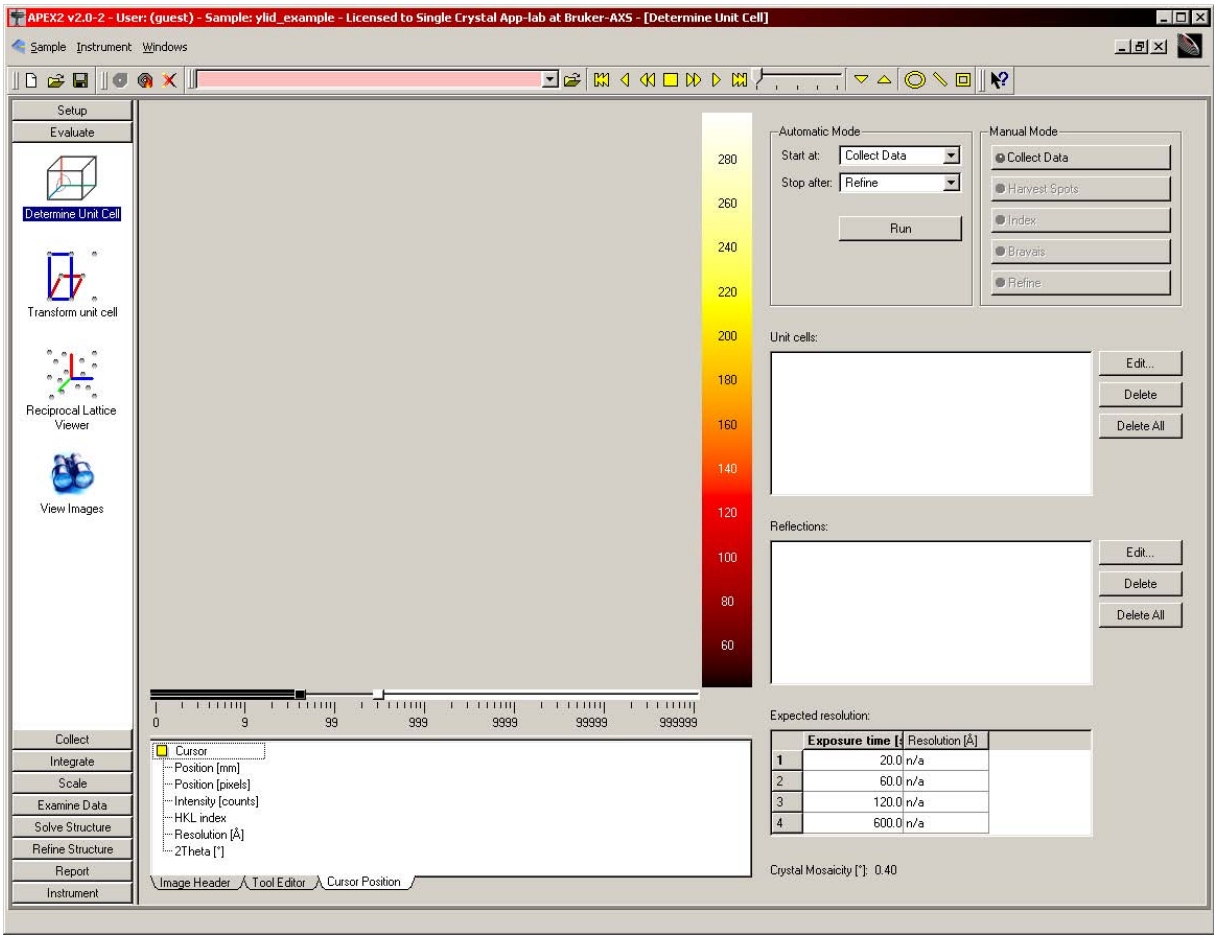


Figure 6.3 – Determine Unit Cell module

6.2.1 Automatic Mode

1. In the Task Bar, click **Evaluate** and then **Determine Unit Cell**. The Determine Unit Cell module appears (Figure 6.3).
2. From the **Start At:** pull-down menu, select the step at which you want APEX2 to begin automatic unit cell determination (useful if you want to find a unit cell from frames that have already been collected).
3. From the **Stop After:** pull-down menu, select the step at which you want APEX2 to stop automatic unit cell determination.
4. Click **Run**. Automatic data collection begins and collected frames appear in the Information Display Area. The area containing the Automatic and Manual mode options changes to a list showing APEX2's progress through the steps of unit cell determination.

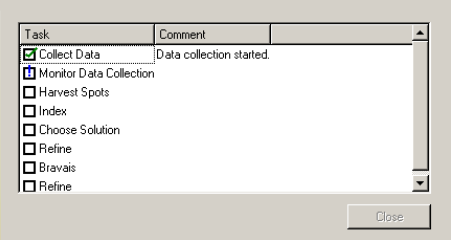


Figure 6.4 – Automatic Mode progress list

5. When automatic unit cell determination is complete, the progress list will show a check mark and comments for each completed step (Figure 6.5).

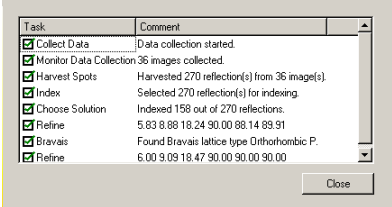


Figure 6.5 – Completed progress list

6. Click the **Close** button to close the progress list and return to the main Determine Unit Cell window. The right-hand side of the window displays the unit cell parameters obtained by APEX2, along with options to edit or delete the unit cell and reflection list if you wish to perform additional operations in Manual Mode (more information on Manual Mode operations is given in Section 6.2.2).

6.2.2 Manual Mode

The Determine Unit Cell Module's Manual Mode functions allow you a great degree of control over unit cell determination. Manual Mode consists of five sections, each with its own menu, corresponding to the five stages of unit cell determination:

- Collect Data
- Harvest Spots
- Index
- Bravais
- Refine

Collect Data

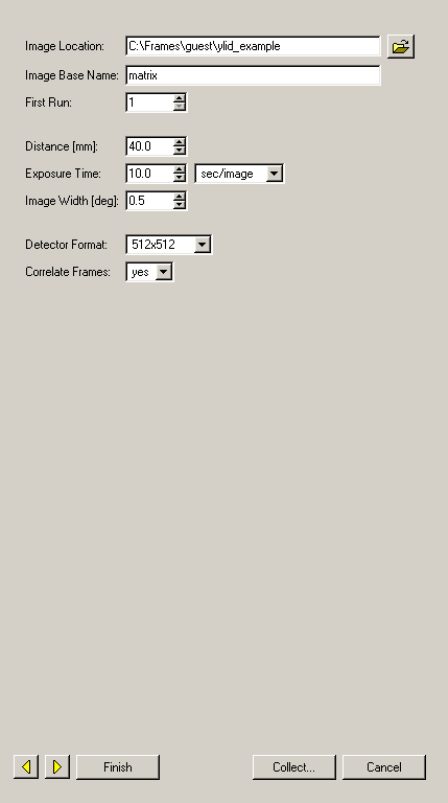


Figure 6.6 – Collect Data menu

Menu Item	Function
Image Location:	Location where collected frames will be saved.
Image Base Name:	Text string that is appended to each frame's filename and is used to identify frames by their filenames.
First Run:	Choose which run number will be the first run; useful for adding runs without overwriting previous ones.
Distance [mm]:	Detector distance.
Exposure Time:	Duration, in seconds, of each frame.
Image width [deg]:	Distance, in degrees, that the scan axis travels over the course of a single exposure.
Detector Format:	Resolution of the frames collected by the detector.
Correlate Frames:	Two frames are taken (each with half the duration of the overall Exposure Time), to correct for spurious events appearing on individual frames.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Collect...	Proceed with data collection according to the options set in the preceding menu items.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Table 6.1 – Collect Data menu items

Harvest Spots

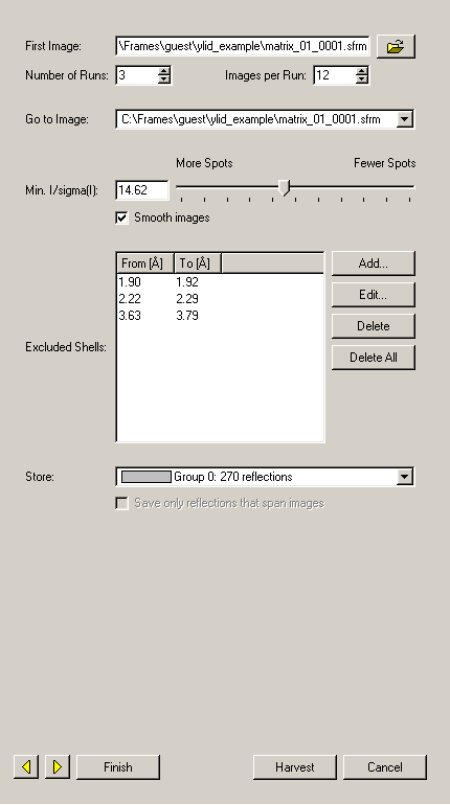


Figure 6.7 – Harvest Spots menu

Menu Item	Function
First Image:	Select the first image in the group of images to be examined for spots.
Number of Runs:	Number of runs to be examined.
Images Per Run:	Number of images to be examined in each run.
Go to Image:	Select a frame filename from this drop-down menu to display the frame in the Image Information Area.
Min. I/sigma(I):	Adjust the criterion for harvesting a spot based on its pixels' intensity versus their standard deviation. Slide the slider between "More Spots" and "Fewer Spots" to vary the minimum I/sigma(I).
Smooth images	A Gaussian filter is applied to the frames prior to harvesting, which reduces the noise and eliminates falsely harvested pixels. For very weak data, however, the Smooth images function can interfere with successful harvesting.

Table 6.2 – Harvest Spots menu items

Menu Item	Function
Excluded shells:	Add, edit, or delete resolution shells to be excluded from the harvesting process, for example in the case of rings caused by ice or amorphous diffractors (the three inner resolution shells for water ice appear as defaults). Excluded shells are defined by their starting and ending resolution (which may be found by placing the mouse cursor on a displayed frame and referring to the Resolution [Å] field in the Cursor Position Tab).
Store (Reflection Group Combo Box):	The Reflection Group Combo Box shows a list of reflection groups to choose from. For each group, the group name and number of reflections in the group are displayed. If, instead of the number of reflections, an entry is labeled empty, this indicates an unassigned entry. Choosing the empty entry creates a new group instead of appending to the current one. A color is associated with each reflection group. The color is displayed in the box to the left of the group name. The color helps in recognizing a group while navigating through the software. Right-click to edit or clear groups.
Save only reflections that span images	With this checkbox enabled, a spot is only harvested if it is found on multiple contiguous frames.
[left arrow]	Go to the previous step in the unit cell determination process.

Table 6.2 – Harvest Spots menu items

Menu Item	Function
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Harvest	Proceed with harvesting according to the options set in the preceding menu items.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Table 6.2 – Harvest Spots menu items

Index

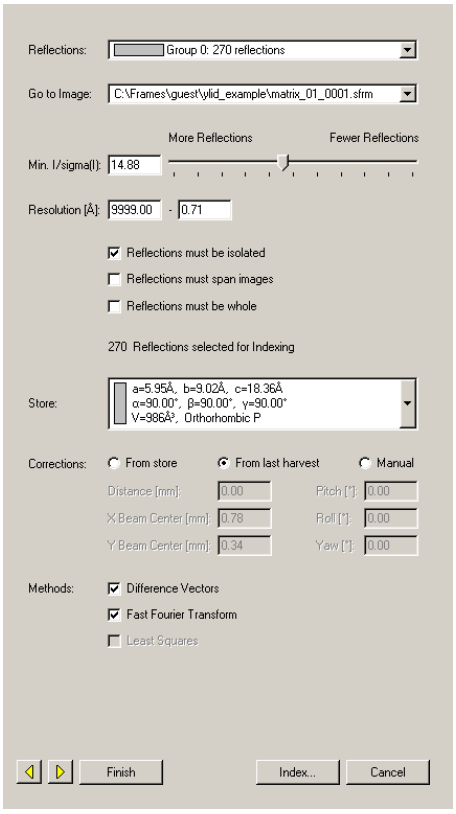


Figure 6.8 – Index menu

Menu Item	Function
Reflections:	Opens the Reflection Group Combo Box to allow you to select a certain group of reflections for indexing.
Go to Image:	Select a frame filename from this drop-down menu to display the frame in the Image Display Area.
Min I/sigma(I):	Adjust the criterion for indexing a reflection based on its integrated intensity versus its standard deviation. Slide the slider between “More Spots” and “Fewer Spots” to vary the minimum I/sigma(I) needed for a reflection to qualify for indexing.
Resolution(Å):	Set the resolution range from which spots will be used to index the unit cell.
Reflections must be isolated	If this checkbox is active, overlapping spots will not be used for indexing.
Reflections must span images	If this checkbox is active, only spots which registered on at least two adjacent images will be used for indexing.
Reflections must be whole	If this checkbox is active, spots that have only partially registered on the first and last images of the range from which they are harvested will not be used for indexing.

Table 6.3 – Index menu items

Menu Item	Function
Store (Unit Cell Combo Box):	The Unit Cell Combo Box shows a list of unit cells to choose from. For each unit cell, the unit cell parameters are displayed, as is the unit cell volume and, optionally, the Bravais lattice type. If, instead of the unit cell parameters, an entry is labeled empty, this indicates an unassigned entry. Choosing the empty entry creates a new unit cell instead of replacing the current one. A color is associated with each unit cell. The color is displayed in the box to the left of the unit cell. The color helps in recognizing a unit cell while navigating through the software. It is also used in the image display to color the overlay and indicate the unit cell that was used to calculate the overlay. Right-click to edit or clear unit cells.
Corrections:	Select corrections to detector parameters. Corrections are available from the stored parameters (i.e., BIS' configuration file), from the last harvest, or from manual input.
Methods:	Select methods employed in indexing: difference vectors, fast fourier transform, or least squares.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.

Table 6.3 – Index menu items

Menu Item	Function
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Index	Proceed with indexing according to the options set in the preceding menu items.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Table 6.3 – Index menu items

Bravais

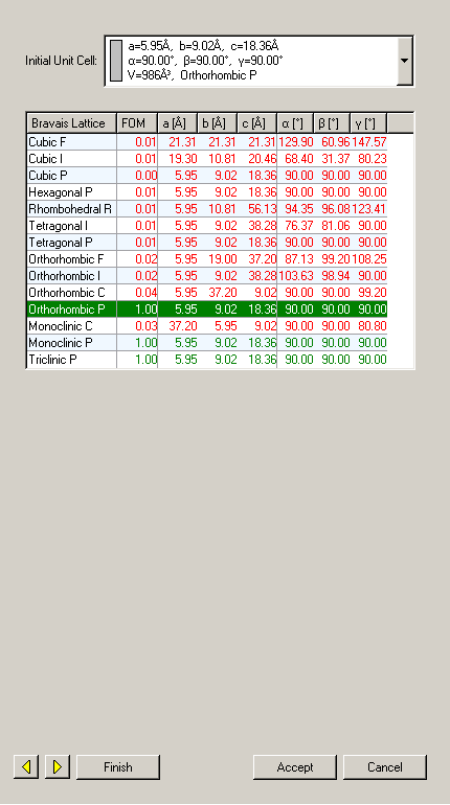


Figure 6.9 – Bravais menu

Menu Item	Function
Initial Unit Cell:	Use the Unit Cell Combo Box to select a unit cell to which to assign a Bravais lattice type.
Bravais Lattice:	This list shows the 14 Bravais lattice types for the selected unit cell parameters. For each entry, the list contains (from left to right) the Bravais lattice type, the figure of merit (ranging from 0.0 to 1.0, with 1.0 being perfect agreement), and the six unconstrained unit cell parameters for that Bravais lattice type. Bravais lattices that are in agreement with the unit cell are displayed in green, which those that do not are displayed in red. The most likely Bravais lattice type is chosen automatically. If necessary, you may override the program's decision by clicking on a different entry.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Accept	Accept the choice of Bravais lattice type given in the menu items above.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Table 6.4 – Bravais menu items

Refine

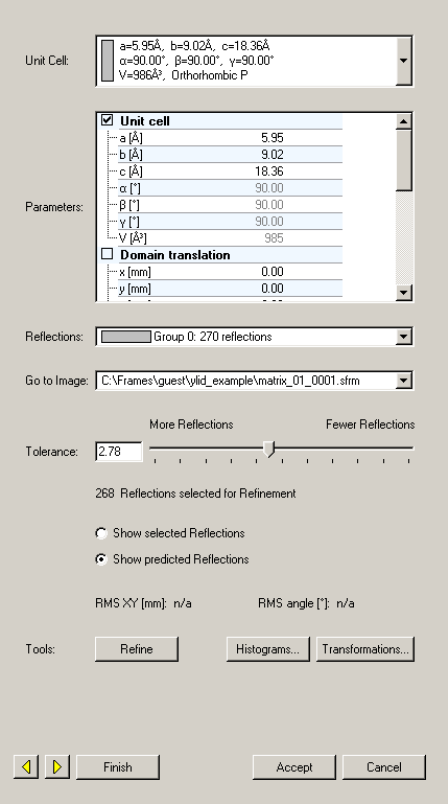


Figure 6.10 – Refine menu

Menu Item	Function
Unit Cell:	Use the Unit Cell Combo Box to select a unit cell to refine.
Parameters:	Within the Parameters window, several areas are available for refinement: Unit Cell, Domain Translation, Domain Orientation, Detector Translation, Beam Center, Detector Orientation, and Goniometer Zeros. By activating or deactivating the checkbox on the left, the parameters in this group are selected for refinement or are constrained to their current values during the refinement.
Reflections:	Use the Reflection Group Combo Box to the select a group of reflections for refinement.
Go to Image:	Select a frame filename from this drop-down menu to display the frame in the Image Display Area.
Tolerance:	Upon startup, the Refine dialog determines a useful value for the tolerance. this may take a few seconds depending on the number of reflections.
Show selected reflections	In the Image Display Area, reflections selected for refinement are marked with circles.
Show predicted reflections	In the Image Display Area, predicted reflections are marked with circles.

Table 6.5 – Refine menu items

Menu Item	Function
RMS XY [mm], angle [°]:	The root mean square of the deviation between observed and predicted spot positions in the XY image plane and along the trajectory of the spot while it passed through the Ewald sphere.
Tools:	Refine – Refine the unit cell using the selected parameters. Histograms... – Displays histograms of reflections' variances in H, K, L, detector X, detector Y, and rotation angle. Transformations... – Opens a dialog for transforming the unit cell.
 [left arrow]	Go to the previous step in the unit cell determination process.
 [right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Accept	Accept the options set in the preceding menu items, and proceed with a single cycle of least-squares refinement. The Refine dialog has a built-in safety which checks whether enough reflections are available for the refinement. If the number of reflections is critically low, a warning is displayed with the option to override.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Table 6.5 – Refine menu items

6.3 Determine the Data Collection Strategy

APEX2 includes a powerful algorithm for determining an efficient strategy that fully utilizes the flexibility of your instrument.

6.3.1 Operation and Initial Settings

1. Under **Collect** in APEX2's Task Bar, click **Data Collection Strategy**. The Data Collection Strategy module opens in the Main Window (Figure 6.11).

The Data Collection Strategy module uses information from the Determine Unit Cell module (Section 6.2) to set defaults. However, you can modify the suggested values.

2. Check the inputs for defining the data collection.

   2.1 Set the data collection distance. For SMART APEX II systems, this should be set to the actual detector distance. For Kappa APEX II systems, the distance is variable and will default to the shortest reasonable distance. For the APEX II detector, the distance in millimeters should generally be about the same as the longest cell dimension in angstroms. Typically, distances ranging from 35 mm to 45 mm are reasonable.

   2.2 Set the exposure time and press [Enter]. For normal crystals and an APEX II detector, five seconds is a reasonable time.

   2.3 Click **Same** to set all of the times to be the same.

NOTE: If the "Same" feature is not chosen, the times for shells can be set to collect high-angle data with longer exposures than inner shell data.

   2.4 Set the desired resolution (0.75Å is a reasonable value).

   2.5 Check the other values (Laue class, Lattice, etc.).

   2.6 Each time a value is changed, the Data Collection Strategy module recalculates the statistics for the runs. The results are displayed in the column labeled **Current**.

NOTE: The rescanning of runs can be interrupted by pressing [Esc]. No statistics will be displayed.

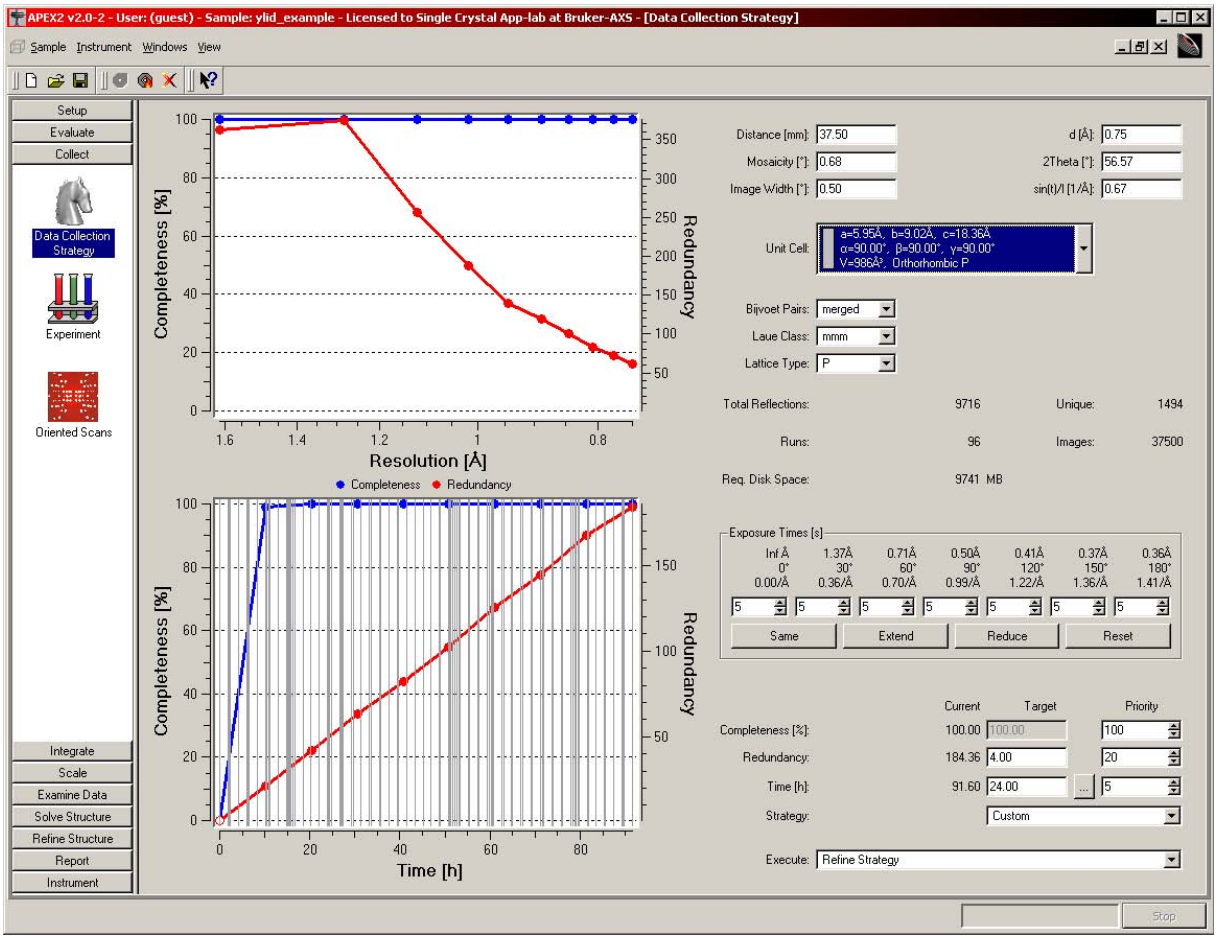


Figure 6.11 – Data Collection Strategy module

6.3.2 Refining the Strategy

At this point, if all of the runs available were collected it would take 91.60 hours and the data would have a redundancy of 184.36. Of course, this is not desirable.

1. Below the “Target” and “Priority” columns is a pull-down menu with several different strategies. Choose the one that best meets the needs of the experiment (for the YLID example, **Best in 2 hours** was selected). The “Target” column changes to reflect your choice.
2. Click in the **Execute:** field. A list of options appears. Click **Refine Strategy**. The strategy begins to refine, approaching the target Completeness, Redundancy, and Time set in step 1.

Figure 6.12 – Click Refine Strategy

3. When you are satisfied with the Completeness, Redundancy, and Time displayed in the “Current” column (whether the module is finished refining or not), click **Stop**. It is neither necessary nor advisable to wait until the refinement reaches 100%.

NOTE: When refining the strategy, the objective is to get good completion (98% or better) with high redundancy in a reasonable amount of time. When the Data Collection Strategy module is first started, it will tell you the Completeness, Redundancy, and Time for all of the available runs.

It is almost never necessary to let the Data Collection Strategy module run to completion. Typically, it should be stopped when completion is greater than 99% and the time is close to what is desired.

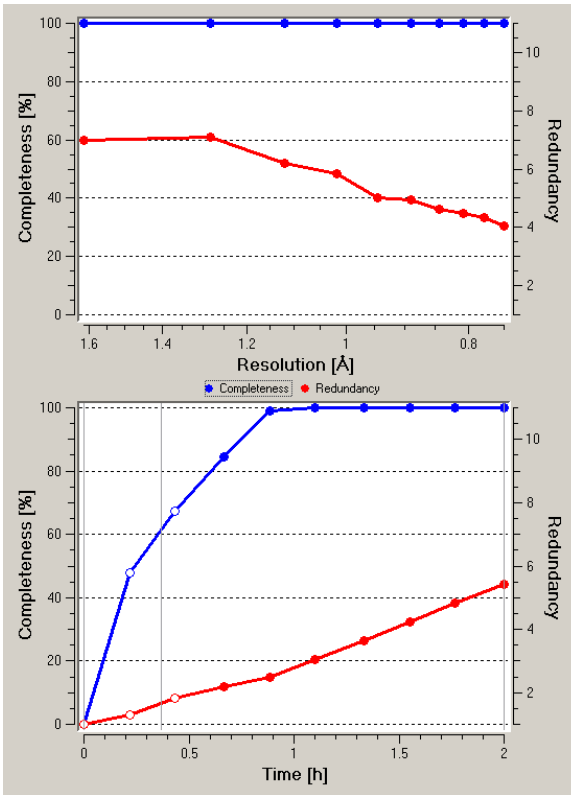


Figure 6.13 – Completeness and Redundancy chart

In the YLID example shown in Figure 6.14, the Completeness is 99.76% and the Time is approximately 2.33 hours.

NOTE: Time estimates are approximate. They depend on the number of rescans, general instrument overhead, backlash compensation, etc. If estimated times are consistently longer or shorter, modify the COSMO hardware profile.

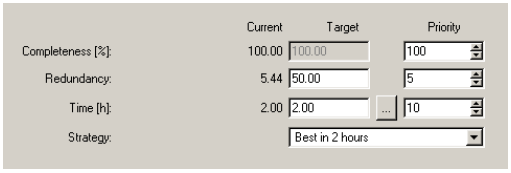


Figure 6.14 – Strategy Status and Priority control

4. Click in the **Execute:** field. A list of options appears. Click **Sort Runs for Completeness**.

Figure 6.15 – Click “Sort Runs for Completeness”

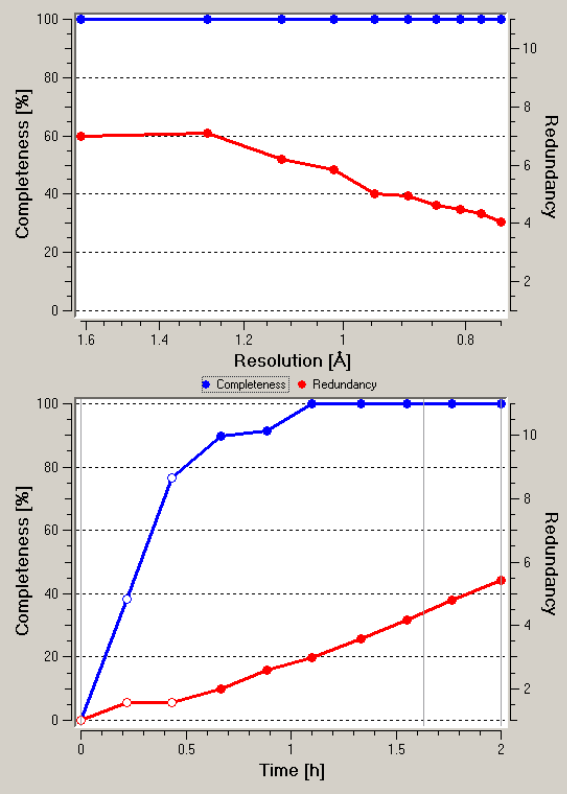


Figure 6.16 – Completeness and Redundancy charts after sorting for completeness

NOTE: If for some reason it is necessary to start over, change the distance slightly (by 0.02 for example) and press [Enter]. The Data Collection Strategy module will reload all of the possible runs.

6.3.3 Looking at the Current Strategy

1. To look at the actual runs chosen, select **View > Detailed Strategy**.

This opens a window that shows the runs to be collected (see Figure 6.17).

	Scan Type	CCD	2Theta	Omega	Phi	Chi	Axis	Width	#Images	Sweep	Time
1	Experiment	37.50	+45.50	-288.43	-9.77	-22.88	Phi	+0.50	719	359.50	13.8
2	Experiment	37.50	-7.00	-158.24	+27.83	+99.23	Omega	+0.50	64	32.00	13.8
3	Experiment	37.50	-49.50	-379.48	-141.44	-20.99	Omega	+0.50	84	42.00	13.8
4	Experiment	37.50	+38.00	-304.81	-3.54	-28.52	Phi	+0.50	473	236.50	13.8
5	Experiment	37.50	+40.50	-328.49	-340.58	+43.25	Phi	+0.50	719	359.50	13.8
6	Strategy	37.50	+45.50	+71.57	-9.77	-22.88	Phi	+0.50	739	369.50	13.8
7	Strategy	37.50	-7.00	-158.24	+27.83	+99.23	Omega	+0.50	64	32.00	13.8
8	Strategy	37.50	-49.50	-19.48	-141.44	-20.99	Omega	+0.50	84	42.00	13.8
9	Strategy	37.50	+38.00	+55.19	-3.54	-28.52	Phi	+0.50	473	236.50	13.8
10	Strategy	37.50	+40.50	+31.51	-340.58	+43.25	Phi	+0.50	739	369.50	13.8
11	Strategy	37.50	-2.00	+20.44	-42.54	-77.76	Phi	+0.50	739	369.50	13.8
12	Strategy	37.50	-2.00	-90.47	+47.30	+28.52	Omega	+0.50	70	35.00	13.8
13	Strategy	37.50	-27.00	-136.80	-249.40	+94.72	Omega	+0.50	0	0.00	13.8
14	Strategy	37.50	+38.00	+20.23	+45.24	+80.52	Phi	+0.50	404	202.00	13.8

Figure 6.17 – Current Strategy window

Each line in this list represents one run. It is specified by:

- Origin
- Crystal-to-detector distance
- Angular positions of the goniometer axes
- Name of the scan axis
- Image width
- Number of Images
- Total Sweep
- Exposure time + overhead

Each run is displayed in a color that indicates its behavior during the strategy refinement:

Black runs are permanent and always active during the refinement process. They are not altered during the refinement. This options is intended for runs that have already been collected and therefore definitively contribute to the data set.

Red runs are considered as-is and, unlike the permanent runs, can be disabled by the refinement algorithm. However, the configuration of such a run remains unchanged (i.e., the scan range will not change).

Blue runs can change their scan range during the course of the refinement. They are therefore called partial runs. This is the default option for all runs from the basis set, because it provides the refinement algorithm with the highest flexibility in finding a strategy that fulfills the criteria for the data set completeness, its redundancy, and the total data collection time.

Gray runs are disabled and are not taken into account at any time during the strategy refinement.

To change the behavior of a run:

1. Place the Data Collection Strategy algorithm in idle mode, i.e., make sure that it is not calculating the reflection list, refining a strategy, or sorting the run list.
2. Select the run or runs you wish to change by clicking inside the Current Strategy window or by dragging the mouse while holding down the left mouse button.
3. Right-click inside the Current Strategy window to open the contextual menu.
4. Select the desired behavior (permanent, as-is, partial, or disabled). The Current Strategy window and the contents of the Main Window will then be updated.

You are now ready to perform the experiment using the optimum strategy.

6.4 Data Collection/Run Experiment

1. Under **Collect** in APEX2's Task Bar, select **Experiment**. The Experiment module opens.

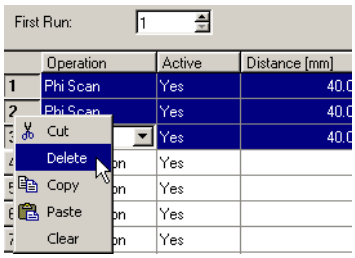


Figure 6.18 – Deleting the matrix runs

2. Click **Append Strategy**. The runs determined in Section 6.3 appear in the list of operations to be performed.
3. APEX2 changes the filename to the name of the current sample (in this example, "ylid_example").

NOTE: You can load and save your own experiments (in *.exp format) by using the **Load Table...** and **Save Table...** buttons at the bottom of the Setup Experiment tab.

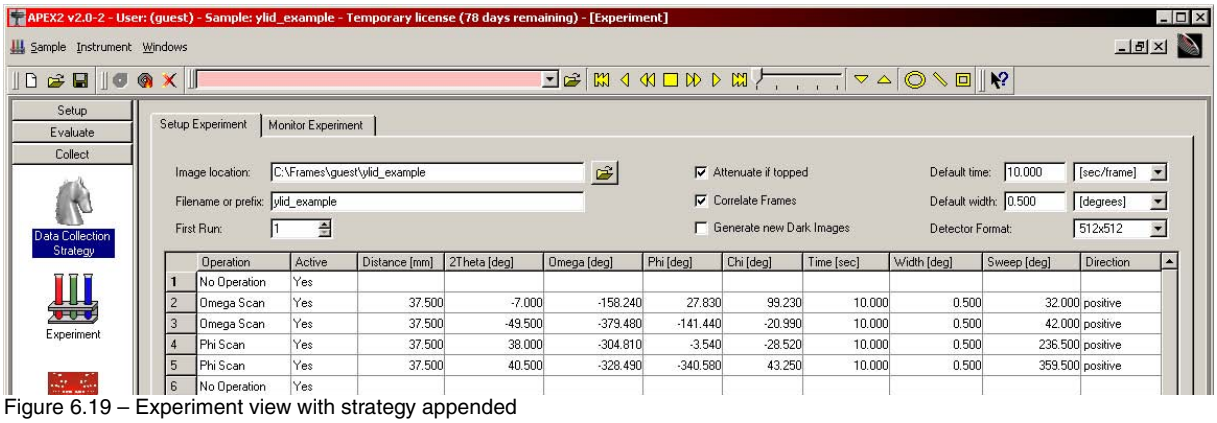


Figure 6.19 – Experiment view with strategy appended

NOTE: At the top of the Setup Experiment tab are controls for data collection. Usually, the default values are correct. For data collection times of less than five seconds, correlation can usually be turned off. If new dark frames are required, BIS will automatically collect them. Checking “Generate New Darks” forces the collection of darks before every run. In Figure 6.19, the time and width are explicitly set for each run, so changing the default width and time will have no effect. If the explicit time or width for a run is deleted so that the box is empty, the word “default” appears and the default values at the top right will be used.

4. If you choose to enter your own runs, click **Validate** to test the strategy for illegal movements by the goniometer. If all motions are valid, the “All operations are valid” window will appear (Figure 6.20).

Figure 6.20 – Operations valid window

5. To begin the experiment, click **Execute**. APEX2 shifts to the Monitor Experiment tab and diffraction images begin to appear. This may take a minute or two if new darks are being collected, or if the generator is being ramped up from a low-power state.

NOTE: If resuming after a data collection interruption, APEX2 will automatically skip images that were previously collected with matching angles and generator settings. Otherwise, APEX2 will ask if you want to overwrite the images.

NOTE: After data collection is started, the Experiment window can be closed and APEX2 can be stopped. The Server computer must be left on; in a single-computer setup, BIS must be left running. If communications are lost between the Client and the Server, frames will be stored on the Server. Typically, they will be in the directory C:\frames\. They should be copied into the correct project directory before starting integration.

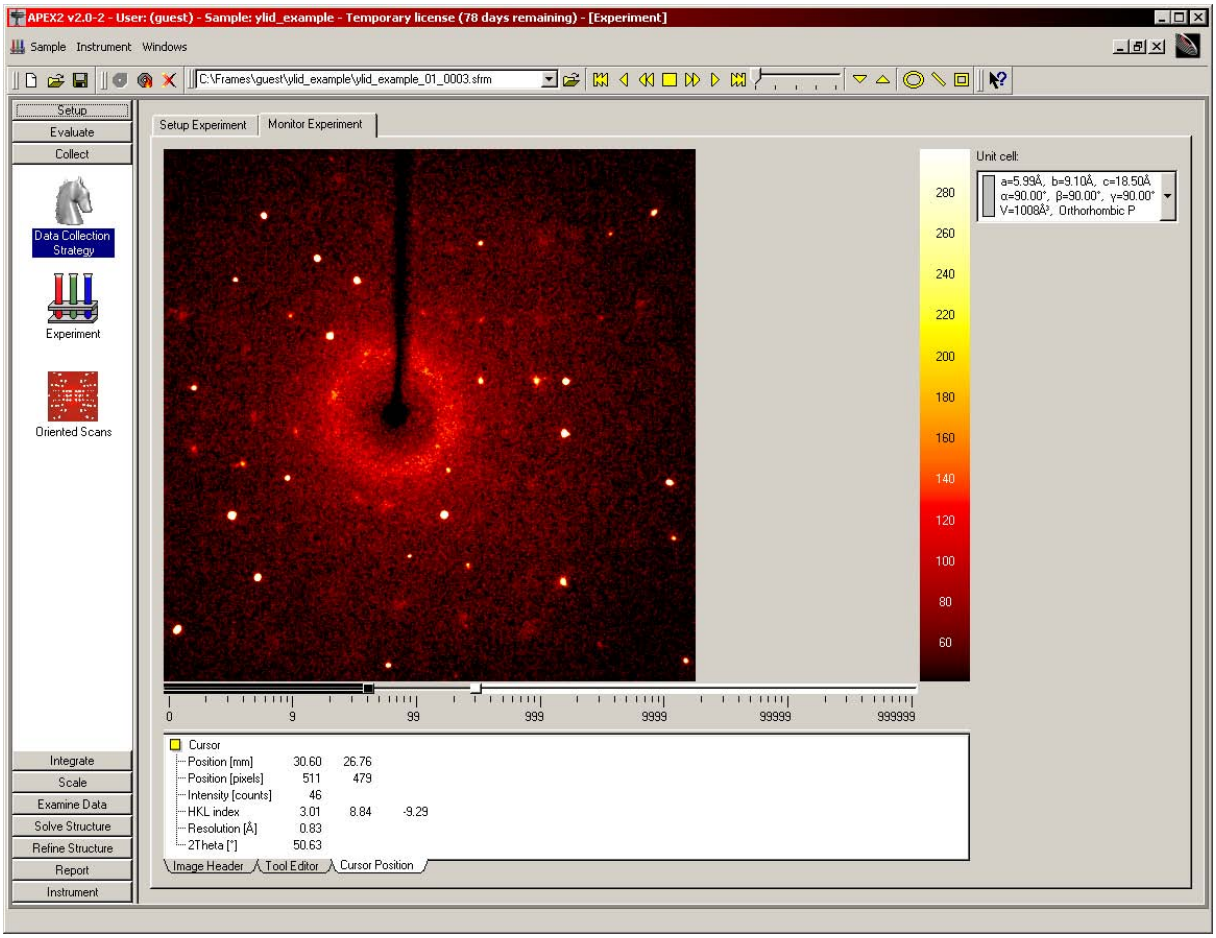


Figure 6.21 – Monitor Experiment tab

7 Data Integration and Scaling

Before the data can be used to solve and refine the crystal structure, it is necessary to convert the information recorded on the frames into a set of integrated intensities, and to scale all of the data.

7.1 Integration

1. Under **Integrate** in the Task Bar, click **Integrate Images**. The initial integration window opens.

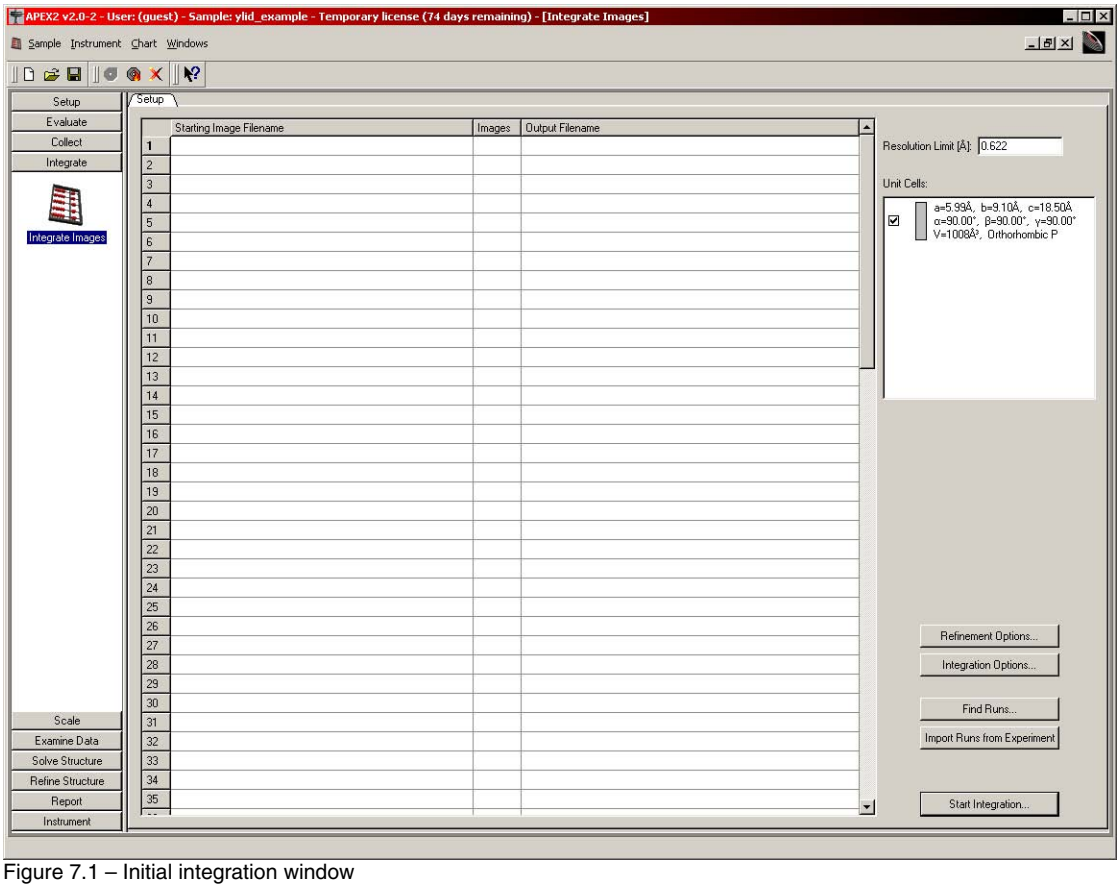


Figure 7.1 – Initial integration window

2. The Resolution Limit parameter in the upper right sets the upper limit of resolution for integration. The APEX2 Suite suggests a resolution cutoff. In this case, we will change the selected value to 0.75Å.

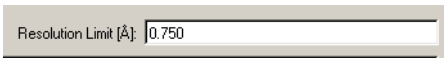


Figure 7.2 – Resolution Limit parameter

3. On the right-hand side of the window are two buttons for defining the data collection runs to be integrated.

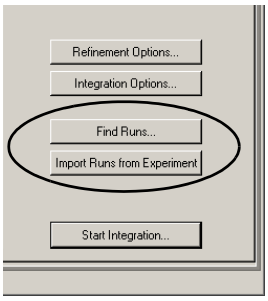


Figure 7.3 – Find Runs and Import Runs from Experiment buttons

The **Find Runs...** button is used to browse to the set of runs to be integrated. This button is normally used when the data collection is finished.

The **Import Runs from Experiment** button determines the runs to be integrated from the experiment that has just been submitted. Using this button allows you to start integration while the data is still being collected.

NOTE: When integrating while collecting data, the SAINT integration module will integrate all of the data currently measured and then wait for the next image, integrate that, wait for the next, etc.

---

4. Click **Find Runs**. The Find Runs window (Figure 7.4) opens. Usually, the Find Runs window has the correct directory and base name as the defaults. If these are not correct, then use the **Browse** button to find the runs for the experiment of interest.

   The window pane on the right side displays all of the runs available in the current directory. Any combination of runs may be selected by simply checking the checkboxes next to the groups and clicking **OK**.

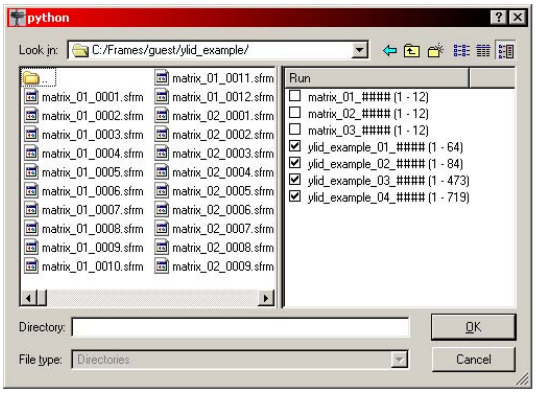


Figure 7.4 – Find Runs window

5. Both the **Import Runs from Experiment** and the **Find Runs** buttons generate a list of runs as in Figure 7.5.

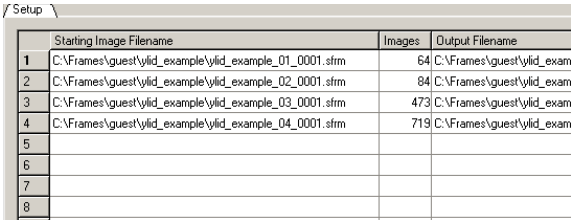


Figure 7.5 – Runs list with runs imported

NOTE: To integrate runs from a CD or DVD, browse to the device and find the runs to be integrated. APEX2 will automatically write the result in the work directory for the current project.

Figure 7.6 shows a run list entry being modified. (A) shows the original run information. (B) shows the run with the starting image number changed to 51. (C) shows the number of frames to process changed to 111.

Double-click any of the fields in this list to open up the value for editing.

Figure 7.6 – Example of manually editing a run list

7.1.1 Check the Refinement Defaults

Two buttons on the right-hand side of the initial integration window are used to change the default options for refinement and integration.

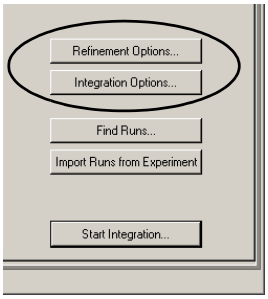


Figure 7.7 – Refinement and Integration Options buttons

The default values are generally very good. There is seldom any need to change the values in the Refinement Options window.

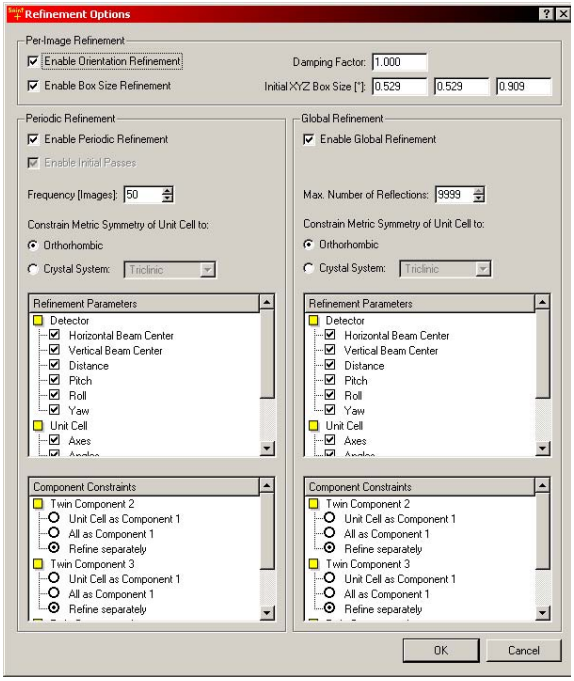


Figure 7.8 – Refinement Options window

7.1.2 Check the Integration Options

1. The Integration Options window has a few values that are changed more frequently. We will change the default options for integration in the Integration Options window.

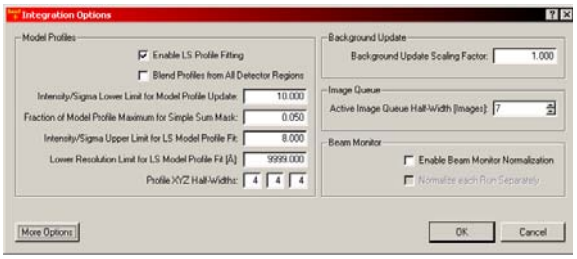


Figure 7.9 – Integration Options window

2. Click the **More Options** button. This results in an expanded window (see Figure 7.10) and gives access to several other useful features, in particular Active Mask (see Figure 7.11), Algorithm (see Figure 7.12), and Image Queue (see Figure 7.13).

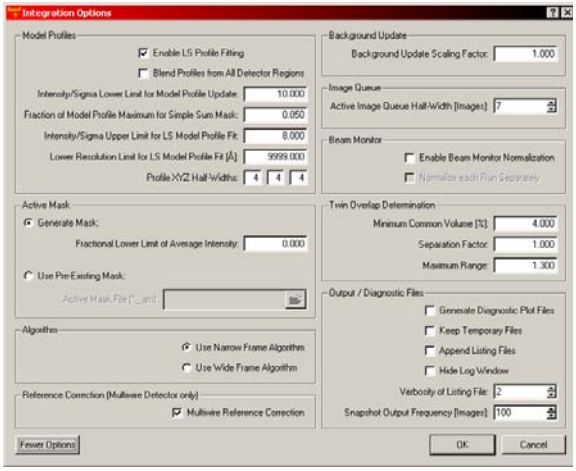


Figure 7.10 – Integration Options window (expanded)

Active Mask

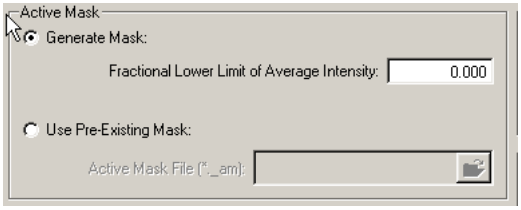


Figure 7.11 – The Active Mask tool

The Active Mask is used to mask out areas that are covered by the beamstop, low-temp nozzle, or other obstructions. Typically a value of **0.7** is good here. SAINT will determine an active mask for each run.

NOTE: If this option is set, you should examine the active mask images that are written into the work directory with the name basename_am_##.sfrm. There is one for each run. These images can be opened with the image viewer. They should be blank except for an area in a different color showing the shape of the obstruction, typically the beamstop.

Narrow/Wide Algorithm

Figure 7.12 – The Narrow/Wide Algorithm tool

The algorithm buttons are used to change from narrow to wide frame integration. Frames collected with narrow scan widths (less than 1.5°) should be integrated as narrow frames. Those greater than 1.5° should be integrated as wide frames.

Image Queue

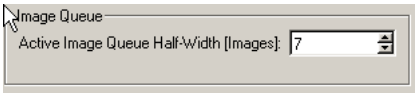


Figure 7.13 – The Image Queue tool

The Image Queue sets the number of frames being used to determine profiles. For crystals with high mosaic spread (i.e., large, wide reflections), increasing the queue size can improve integration.

7.1.3 Start Integration

1. Click the **Start Integration** button on the far right of the bottom row. This starts the integration and opens the SaintChart window (discussed in Section 7.2) for monitoring the integration.
2. Examine the output. Double-click any of the output entries (in the “Output Filename”) for one additional tool: the **ls** button at the far right of the line (see Figure 7.14). The **ls** button opens the SAINT listing file for that integration run.

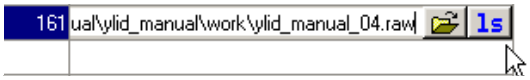


Figure 7.14 – The runs list’s ‘ls’ button

7.2 SaintChart

SaintChart is a powerful tool for monitoring the progress of the integration process and for graphically presenting the results of the integration process.

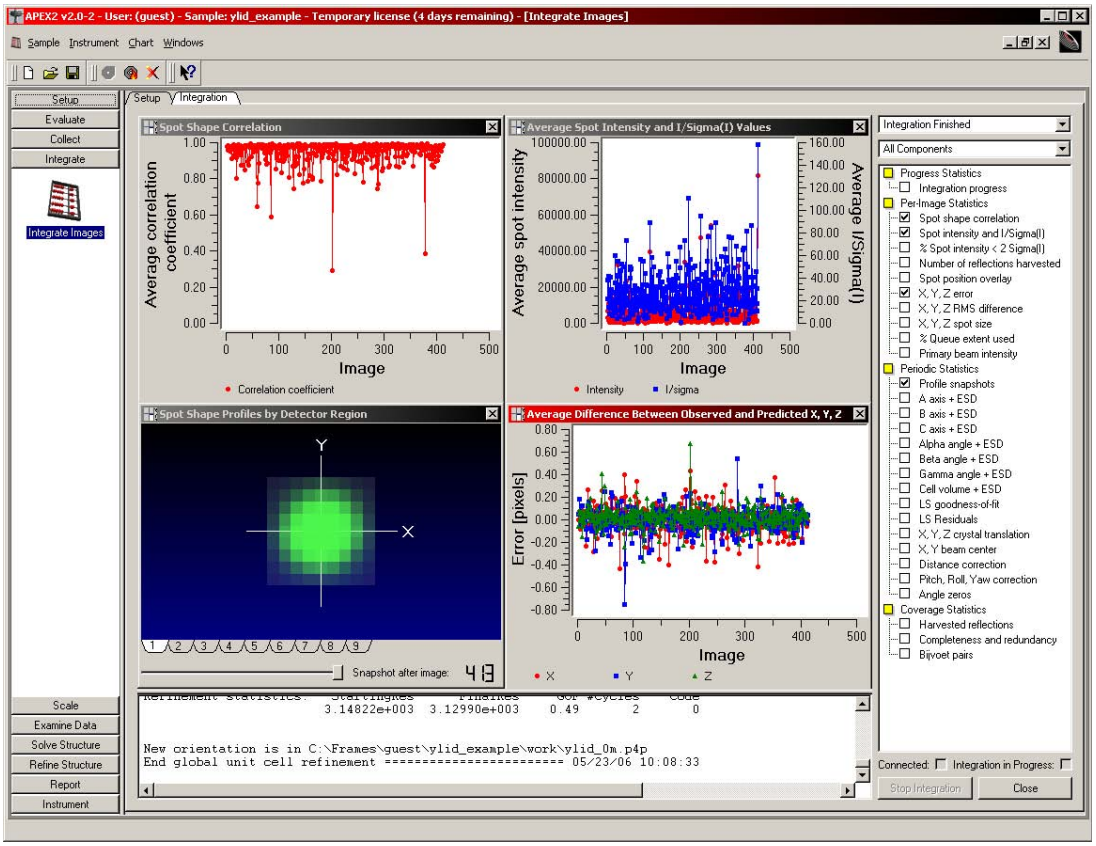


Figure 7.15 – SaintChart view

7.2.1 Monitor the Progress of the Integration

Change the text area by clicking and dragging on the line between the text and image areas. You can also expand the windows to fill the available image area by clicking **Chart > Tile**  in the Menu Bar.

To the right of the image area are a series of checkboxes that select the displays. Click the checkboxes to add or remove displays.

Average Difference

Errors in X, Y, and Z should be small (less than 1 pixel) and should not vary during the integration of a run. Large variations indicate problems with slipping crystals, misalignment of the instrument, or other problems.

Spot Shape Profiles by Detector Region

Look for spot shapes that are well-contained within the box. Split spot shapes indicate diffraction from split crystals or twins.

Spot Shape Correlation

the Spot Shape Correlation graph is the best indicator for a successful integration. After initial passes during the actual integration, one expects a high correlation factor at a constant level, i.e., >90% for very good crystals and >80% for good crystals. Correlation factors below 40% indicate that the orientation matrix does not describe the diffraction pattern correctly.

Integration Progress

1. Check **Integration Progress** to add the new display seen in Figure 7.16. When the integration is complete, the blue progress bars will be full and the remaining time will be zero.

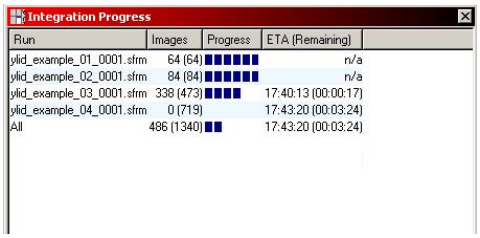


Figure 7.16 – Integration Progress display

Spot Position Overlay

Check **Spot Position Overlay** (in the “Per-Image Statistics” category) to add a window for monitoring the progress and quality of the integration. Pull up on the Window Tool Bar for Integrated Spots to display the entire window.

In this full view, the Integrated Spots window looks much like the image viewer used previously. At the bottom of the window is an entry box that can be used to change the display frequency of the Integrated Spots images (the default is to display every fifth image).

The shape of the overlay represents the reflections’ sizes and shapes determined during the integration.

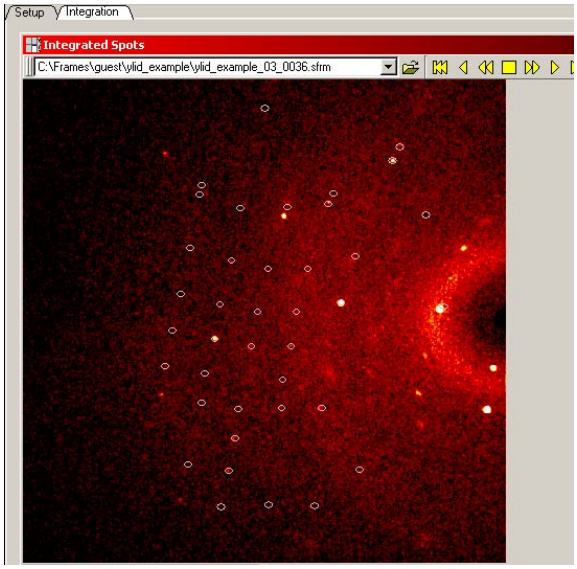


Figure 7.17 – Spot Position Overlay

7.2.2 Examine Final Results

At the end of integration, examine this text critically. In particular, look at the Overall R_{sym}, the Coverage Statistics and the Unconstrained Unit Cell refinement.

1. View the SAINT text output. At the bottom of the SaintChart window is the scrolling text window that displays text output from SAINT. (This window can be expanded by dragging the horizontal bar above the text.
2. Examine the final results.

Overall R_{sym} and Coverage Statistics

- The overall R_{sym} is usually less than 8%. Higher values may indicate problems with absorption, twinning or poor crystal quality.

The coverage statistics are reported by resolution shells.

- The values in the `Rsym` column give the cumulative agreement of equivalent reflections.
- The `Rshell` values give agreement within a particular shell. These change faster than the `Rsym` values.
- The `%<2s` column is useful for checking where the weak data becomes predominant (greater than 50 or 60%).
- The `#Sigma` column gives the average I/sigma for each shell. If that number is less than 1.5 or 2, then little significant data is being measured in that shell.

```
Overall      #  Pairs   Uniq   Merg %<2s       <I> <#Sig>    <Bg>  Rsym
  0.000   6853   1259   1400   6759  6.3  5521.869  45.67    2.53 0.023
  1.000

 Centric     #  Pairs   Uniq   Merg %<2s       <I> <#Sig>    <Bg>  Rsym
  0.000   1424    339    428   1370 13.3  7973.527  63.33    2.67 0.020
  1.000

Coverage statistics for ylid_0m.raw
                                                          .......Shell......
 Angstrms    #Obs Theory %Compl Redund   Rsym Pairs %Pairs Rshell #Sigma %<2s
to  1.615    179    180  99.44   7.27  0.019   178  98.89  0.019 103.49  3.6
to  1.283    332    332 100.00   7.30  0.020   329  99.10  0.024  42.35  2.3
to  1.121    477    478  99.79   7.01  0.021   472  98.74  0.026  29.81  2.2
to  1.019    618    619  99.84   6.61  0.021   606  97.90  0.030  19.90  4.9
to  0.946    768    769  99.87   6.18  0.021   745  96.88  0.037  15.78  7.2
to  0.890    909    910  99.89   5.85  0.022   875  96.15  0.045  11.91  9.3
to  0.845   1044   1045  99.90   5.56  0.022   992  94.93  0.059   9.13  9.2
to  0.809   1192   1196  99.67   5.30  0.023  1118  93.48  0.060   8.08 12.0
to  0.778   1320   1328  99.40   5.08  0.023  1220  91.87  0.064   6.54 19.2
to  0.751   1400   1462  95.76   4.89  0.023  1259  86.11  0.067   5.68 12.6
```

Overall R_{sym}

Coverage

Figure 7.18 – Final SAINT Overall R_{sym} and coverage statistics

Unconstrained Unit Cell Refinement

Check the angles in the unconstrained unit cell refinement to get a quick confirmation that the initial lattice determination was correct. In the unconstrained refinement, angles that are required to be 90° or 120° are allowed to refine. If they differ considerably from the expected value, then the data should be checked carefully.

```
Unconstrained global unit cell refinement ============== 05/30/06 18:17:01

Performing final unit cell least squares on file C:\Frames\guest\ylid_example\work\ylid_01._ma
Input file contains       4325 reflections

Refinement includes 1 sample
   Sample 1 contains component   1

Global Refinement for sample  1 of  1 -- single-component data...
Maximum allowed reflections =       9999
One reflection will be stored in memory per     1 reflection(s) read

Orientation least squares, component 1 in sample, 1 in integration (4325 input reflections)...
Wavelength, relative uncertainty:  0.7107300,  0.0000089

Reflection Summary:
'RLV.Excl' are reflections excluded after cycle 1 because RLV error exceeded 0.0250:
 Component     Input  RLV.Excl      Used  WorstRes   BestRes   Min.2Th   Max.2Th
         1      4325         0      4325    9.1954    0.7672     4.430    55.184

Orientation ('UB') matrix:
   0.0005223  -0.0652460  -0.0439134
  -0.1290715  -0.0572444   0.0203780
  -0.1071659   0.0686201  -0.0247583

         A         B         C     Alpha      Beta     Gamma           Vol
    5.9608    9.0378   18.3909    89.997    90.001    90.002        990.76
    0.0005    0.0007    0.0014     0.001     0.001     0.001          0.23
Corrected for goodness of fit:
    0.0002    0.0003    0.0007     0.000     0.000     0.000          0.11

Crystal system constraint:            0(Unconstrained)
Parameter constraint mask:            0

Eulerian angles:                   65.108     117.084    -134.154
```

Figure 7.19 – Unconstrained unit cell refinement (YLID unit cell shown)

7.3 Scale Data

The scaling process uses the SADABS program to put all of the measured data on the same scale. This process involves five steps:

- Set up input files
- Parameter refinement
- Error model refinement
- Display diagnostics
- Exit

7.3.1 Set Up Input Files

1. Under **Scale** in the Task Bar, click **Scale**. the initial Scale window appears. Figure 7.20 shows the right-hand portion of the Setup tab. The defaults and file names are typically correct.
2. Check that the defaults are correct. Generally, the Absorption Correction Type is the only value that might be changed. Numerical Absorption Correction requires indexed faces. Face indexing is discussed in Appendix B.
3. Click **Next**.

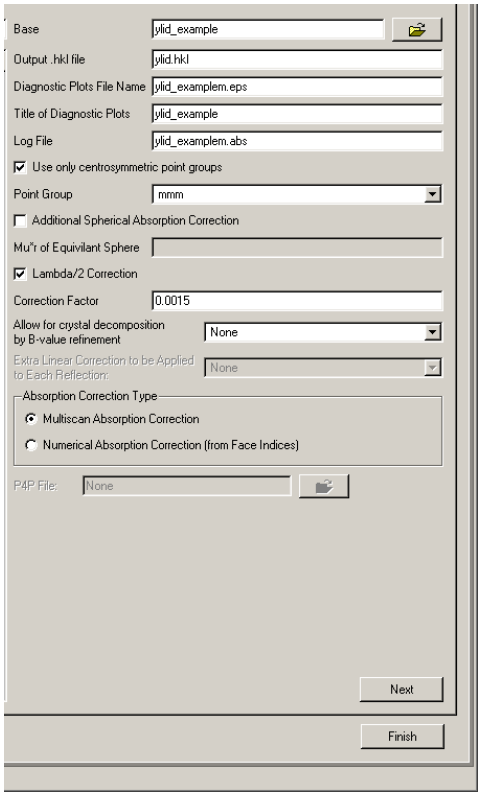


Figure 7.20 – Initial Scale window: Setup tab

7.3.2 Parameter Refinement

1. Click **Refine**. Figure 7.21 shows a typical refinement result. The R-values are reduced and the Mean Weight increases. Typical final values are 3–4% for the R-values and 0.95–0.98 for the Mean Weight.

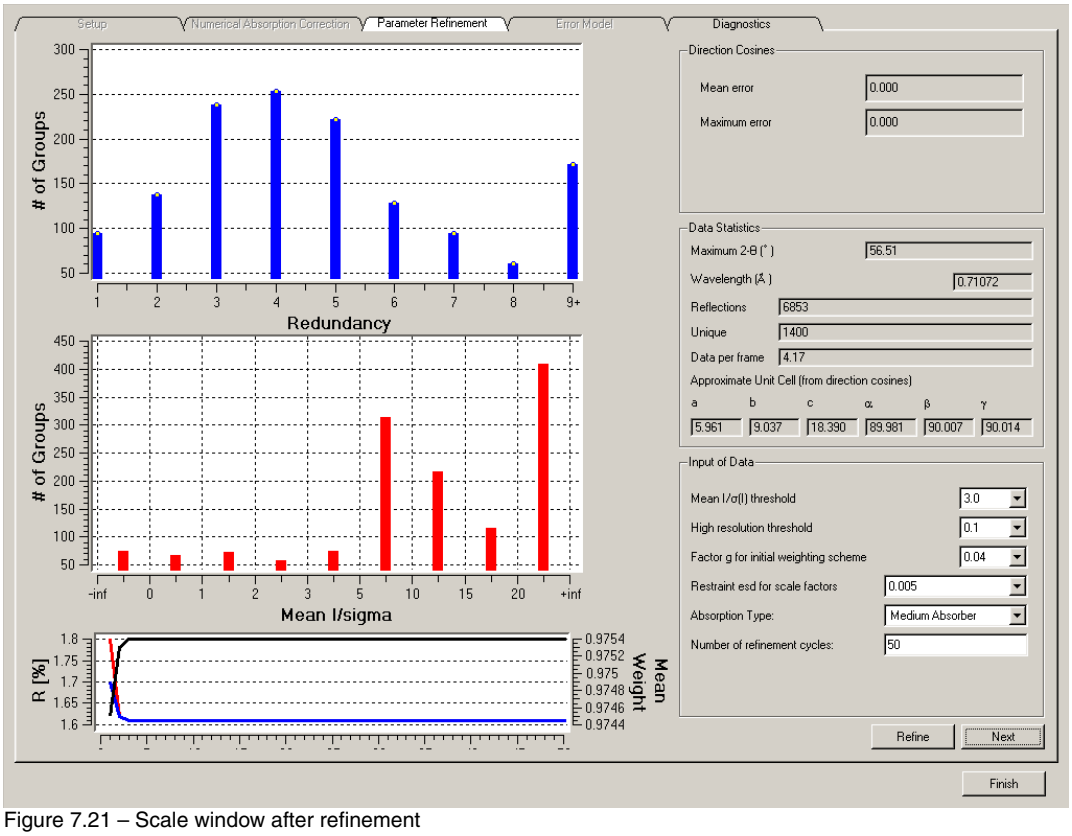


Figure 7.21 – Scale window after refinement

7.3.3 Error Model Refinement

1. Click **Next** to proceed to the Error Model tab, and click **Determine Error Model**. Figure 7.22 shows typical Error Model results.

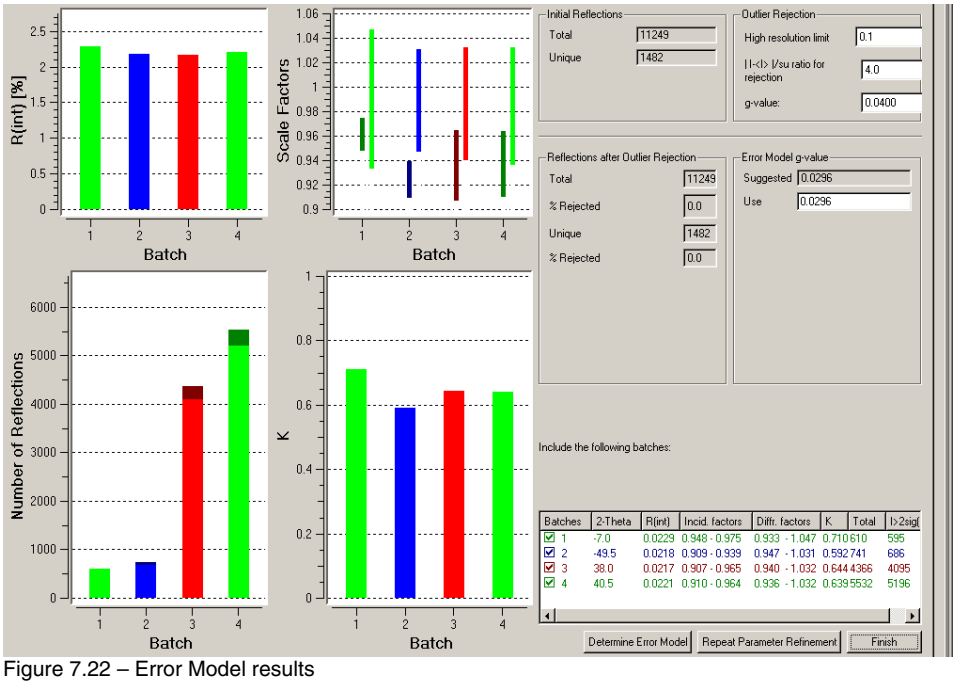


Figure 7.22 – Error Model results

NOTE: If the R-values in one run are significantly higher than the others, you can repeat the entire scaling calculation with that run omitted. Uncheck the checkbox next to the “bad” run and then click **Repeat Parameter Refinement**.

7.3.4 Examine Diagnostics

1. Click **Finish** to produce a series of diagnostic plots. Some of these are reviews of the plots that have already been displayed.

   The diagnostics provide valuable insight into the quality of the data and possible problems with the data. Click the tabs at the bottom of the screen to view the diagnostic data.

The **Scale Variations** plot (Figure 7.23) shows the overall variation in Scale and R(int) for the individual frames. The Scale plot should be flat (for highly absorbing, irregularly shaped crystals it will probably be sinusoidal) and the R(int) plot should not show large variations (more than 2%).

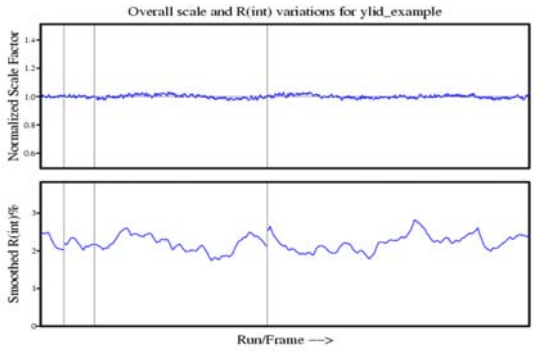


Figure 7.23 – Overall Scale and R(int) variations

The **Intensity Statistics** plots look at R(int) and $|E^2-1|$ as a function of resolution. Typical plots of R(int) versus resolution increase to the right as seen in Figure 7.24. An $|E^2-1|$ plot should have a constant value. $|E^2-1|$ is a strong indicator for centric and acentric space groups. The two horizontal lines indicate the expected values for centric (top) and acentric (bottom) space groups. This plot clearly suggests an acentric space group.

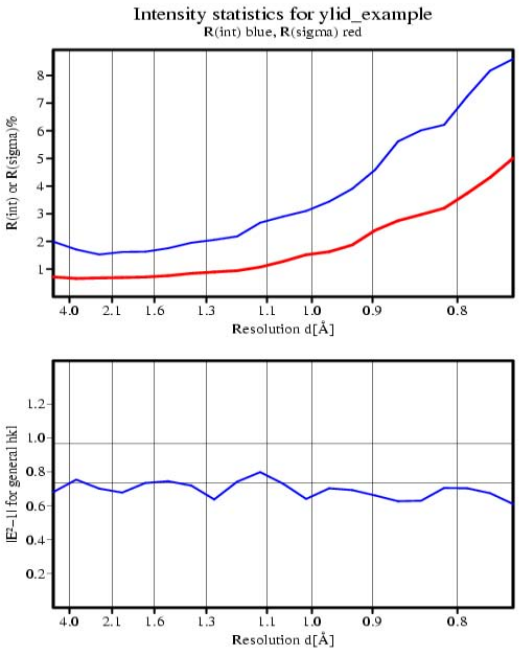


Figure 7.24 – Intensity Statistics

The plots of **Chi-Squared** values for the data as a function of resolution and intensity should be mostly flat. The plots shown in Figure 7.25 are typical.

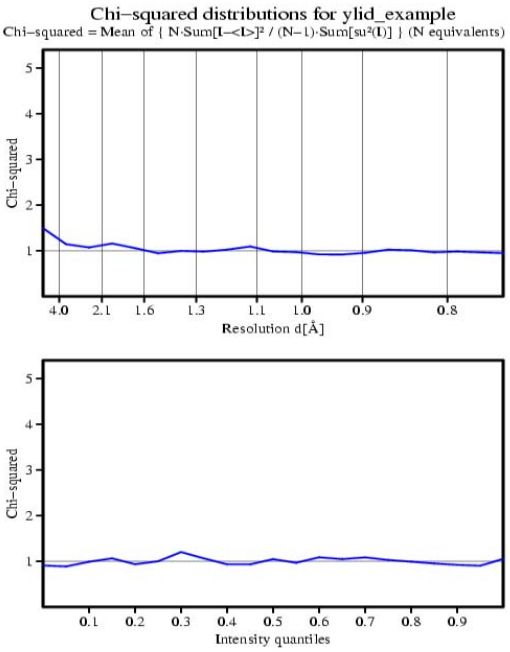


Figure 7.25 – Chi-squared

The **Spatial Distribution** plots are generated for each data collection run. They indicate spots that were either stronger or weaker than expected with a deviation from the mean intensity larger than three standard uncertainties. Figure 7.26 shows two spatial distribution plots side by side. This is the way they are typically output by the scaling process.

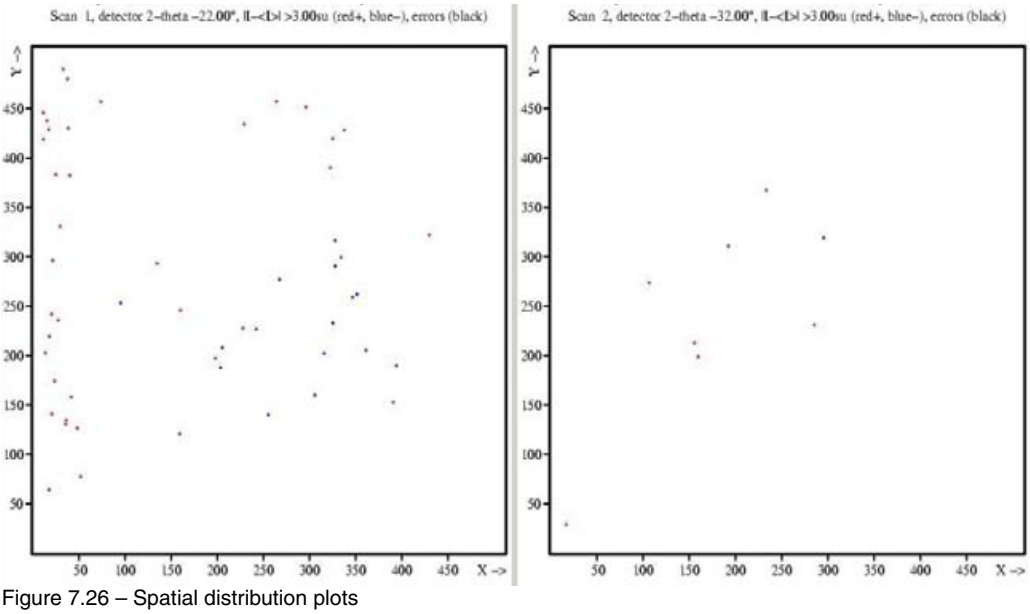


Figure 7.26 – Spatial distribution plots

Figure 7.27 shows a single plot so that the dots are easier to see. Note that the points of disagreement are spread fairly evenly over the entire detector face.

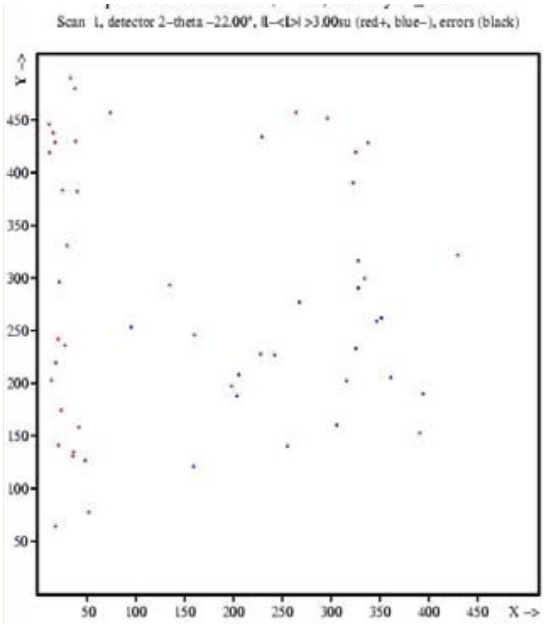


Figure 7.27 – A single Spatial Distribution

If the spots are clustered in an area or if there are significantly more spots of one color than there are of another, then the data should be examined critically. Figure 7.28 is from another data set in which the Active Mask was not used during integration. Consequently, the reflections collected in that area are consistently weaker than expected. This kind of problem may affect the final results.

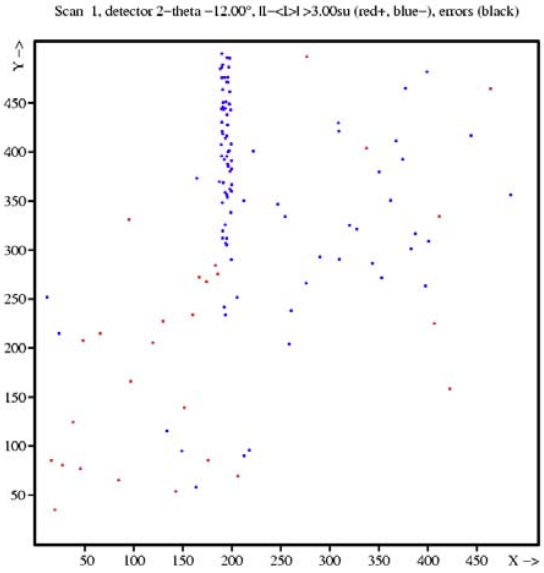


Figure 7.28 – A Spatial Distribution plot showing a problem area

7.3.5 Exit

1. Click **Exit AXScale** to close the Scale module.

Figure 7.29 – Exit AXScale

8 Examine Data

In this step, the space group for the crystal is determined and optional simulated precession photographs are calculated to further evaluate the overall quality of the data. APEX2 provides two tools for this: **Space Group Determination** (based on XPREP) and **Precession Images** for looking at undistorted slices of reciprocal space. This is the final step before beginning the structure solution and refinement process.

8.1 Space Group Determination With XPREP

1. Under **Examine Data** in APEX2's Task Bar, click **Space Group Determination**.
2. Check that the two files in the pop-up window are correct (see Figure 8.1) and click **OK**.

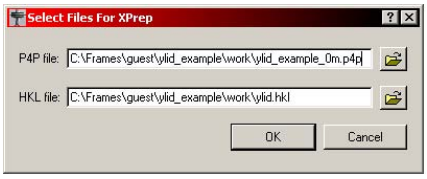


Figure 8.1 – Select files for XPREP input

NOTE: In this example, the integration process has created two files: ylid_example_0m.p4p containing the final unit cell parameters from integration and ylid.hkl containing the corrected intensities. Typically, these are the files to use for determining space groups, but you can browse to choose other files.

8.1.1 Determining Space Groups

1. Lattice type: XPREP evaluates the data and looks at the mean intensities and the mean int/sigma. Since these are large for all groups except P, XPREP suggests that the lattice is P (Figure 8.2). Press [Enter] to accept.

```
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  XPREP - Reciprocal space exploration  - Version 2005/2 for Windows  +
+  COPYRIGHT(c) 2005 Bruker AXS                    All Rights Reserved  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
Screen size: 1280 x 1024
Window size:  640 x  923
Font size:      8 x   16  (  125 x  178 )
Number of colors:    256


When xprep is started without a filename on the command line, the filename
is prompted for and then the type of data (SHELX, SCALEPACK, XDS or XENGEN)
requested.  To generate ideal data, a SHELX .ins or .res file, if necessary
made from a PDB file using SHELXPRO or XPRO, should be given.

'xprep name' reads a SHELX HKLF 4 format file name.hkl, then tries to find
name.spin or name.p4p to extract the cell dimensions and their esds.
'xprep name1 name2' reads name1.hkl and name2.p4p (or name2.spin).

-Ln on the command line allocates space for 1000000n data (default n=4).

  11249 Reflections read from file ylid.hkl
  Mean (I/sigma) =   20.49


Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =          0   5634   5622   5620   5623   8438   7497   7501  11249
N (int>3sigma) =     0   5157   5089   5194   5096   7720   6834   6834  10243
Mean intensity =   0.0  224.9  214.3  211.3  225.4  216.9  223.9  224.6  220.3
Mean int/sigma =   0.0   20.7   19.7   20.9   20.5   20.4   20.5   20.5   20.5

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:
```

Figure 8.2 – Lattice statistics

2. XPREP determines the reduced cell based on the lattice entered above. Since the lattice was primitive and the magnitudes of the cell dimensions were proper (a < b < c), the original and reduced cells are the same (Figure 8.3). Press [Enter] to search for a higher symmetry cell.

```
Current dataset: ylid.hkl                          Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.949   9.018  18.353   90.00   90.00   90.00   Vol     984.6
         Esds:   0.000   0.000   0.000    0.00    0.00    0.00       Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.949   9.018  18.353   90.00   90.00   90.00   Vol     984.6
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------


[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [H]:
```

Figure 8.3 – Reduced cell

3. For the YLID, no higher symmetry cell is found. The program has determined that the YLID crystal has an orthorhombic primitive lattice (Figure 8.4). Press [Enter] to accept.

```
Determination of reduced (Niggli) cell

Transformation from original cell (HKLF-matrix):
   1.0000  0.0000  0.0000     0.0000  1.0000  0.0000     0.0000  0.0000  1.0000

Unitcell:        5.949    9.018   18.353   90.00   90.00   90.00

Niggli form:     a.a =     35.39      b.b =     81.32      c.c =    336.82
                 b.c =      0.00      a.c =      0.00      a.b =      0.00

Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

-------------------------------------------------------------------------------
Option A: FOM = 0.000 deg.   ORTHORHOMBIC P-lattice   R(sym) = 0.015 [   3394]
Cell:    5.949   9.018  18.353   90.00   90.00   90.00    Volume:       984.57
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Option B retains original cell

Select option [A]:
```

Figure 8.4 – Higher symmetry cells

4. The next logical step is to determine the space group. XPREP suggests this (Figure 8.5). Press [Enter] to determine the space group.

```
Current dataset: ylid.hkl                          Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.949   9.018  18.353   90.00   90.00   90.00   Vol      984.6
          Esds:  0.000   0.000   0.000    0.00    0.00    0.00       Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.949   9.018  18.353   90.00   90.00   90.00   Vol      984.6
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic              Lattice: P
-------------------------------------------------------------------------------

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [S]:
```

Figure 8.5 – Determine the space group

5. If the space group is known or if the compound is known to be chiral, enter that information (Figure 8.6). Generally, it is sufficient to press [Enter] to start the space group determination.

```
[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu  or  [Q] QUIT program

Select option [S]:
```

Figure 8.6 – Space Group options

6. XPREP has chosen the crystal system [O] (Figure 8.7). Press [Enter] to accept.

```
[A] Triclinic,  [M] Monoclinic,  [O] Orthorhombic,  [T] Tetragonal,
[H] Trigonal/Hexagonal,  [C] Cubic  or  [E] EXIT

Select option [O]:
```

Figure 8.7 – Choose the crystal system

7. XPREP has chosen the crystal lattice [P] (Figure 8.8). Press [Enter] to accept.

```
Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =          0   5634   5622   5620   5623   8438   7497   7501  11249
N (int>3sigma) =     0   5157   5089   5194   5096   7720   6834   6834  10243
Mean intensity =   0.0  224.9  214.3  211.3  225.4  216.9  223.9  224.6  220.3
Mean int/sigma =   0.0   20.7   19.7   20.9   20.5   20.4   20.5   20.5   20.5

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:
```

Figure 8.8 – Choose the lattice

8. XPREP evaluates the data and looks at the systematic absences for all possible glide planes and screw axes (Figure 8.9). These are displayed across the middle of the figure. By examining the number of reflections with I > 3 sigma(I), the mean intensities, and the mean int/sigma, which should all be very small for a systematic absence, XPREP derives a suggested space group, P2(1)2(1)2(1). Press [Enter] to accept.

8.1.2 Reflection Statistics

```
Mean |E*E-1| = 0.687 [expected .968 centrosym and .736 non-centrosym]

Systematic absence exceptions:

        b--   c--   n--  21--   -c-   -a-   -n-  -21-   --a   --b   --n  --21

N       457   455   460    16   360   374   358    19   233   230   223    17
N I>3s  388   394   386     0   281   282   229     0   192   203   171     0
<I>   201.1 300.0 317.2   0.5 449.8 377.8 201.2   0.5 290.7 311.3 277.0   0.6
<I/s>  22.1  22.6  23.6   0.4  23.9  21.5  15.9   0.6  20.8  22.7  21.7   0.6

Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type  Axes  CSD  R(sym) N(eq)  Syst. Abs.   CFOM

[A] P2(1)2(1)2(1)  # 19  chiral   1  5917  0.015   3394  0.6 / 15.9   0.89

Select option [A]:
```

Figure 8.9 – Systematic absences and a suggested space group

1. XPREP returns to the general menu seen previously (Figure 8.5). This time, D is chosen to evaluate the data set. Press [Enter] to accept.
2. There are multiple choices for data manipulation (Figure 8.10). “S” chooses a display of statistics.

```
Index   # Data   Filename or Source of Data

   1    11249    ylid.hkl  <- current dataset

[M] Sort-MERGE current data (no scaling)     [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets   [W] WRITE dataset to file
[I] INCLUDE Rfree flags from another file    [R] READ in another dataset
[S] Display intensity STATISTICS             [D] DELETE stored dataset
[F] FACE-indexed absorption corrections      [P] PSI-scan absorption corr.
[T] Copy file, TRANSFORM hkl and cosines     [A] MAD, SAD, SIR or SIRAS
[H] Apply HIGH/low resolution cutoffs        [N] NORMALIZE/scale sigmas
[G] Generate simulated powder diagrams       [U] Anisotropic scaling
[Z] Expand data to triclinic                 [E] EXIT to main menu
[X] Parsons' Q values and Flack x parameter  [Q] QUIT program

Select option [S]:
```

Figure 8.10 – The data manipulation menu

3. The data can be merged in several ways. Choose the “Merge ALL equivalents including Friedel opposites” option [A] (Figure 8.11). Press [Enter] to accept.

NOTE: This merge will not average the reflections in the final data file. It is only for the calculation of statistics.

```
Current dataset: ylid.hkl

[R] Define resolution ranges (currently selected automatically)
[P] Output R(pim) rather than R(sigma)
[N] Do NOT merge reflections
[I] Merge IDENTICAL indices only
[S] Merge SYMMETRY equivalents (but not Friedel opposites)
[A] Merge ALL equivalents (including Friedel opposites)
[E] EXIT to merge datasets menu
[Q] QUIT program

Select option [A]:
```

Figure 8.11 – The merge data menu

4. A table of statistics appears (Figure 8.12). Examine the data presented. Is the completion near 100%? Is the redundancy good? Are R_{int} and R_{sigma} small and increasing smoothly from top to bottom? In particular, look at the last two lines which compare all of the data with the high-resolution data. The completion should be near 100% for both the high-resolution shell and the complete data set. The redundancy and R_{int} should be similar for the two. The Mean Intensity and the Mean I/sigma(I) will usually be quite different. The Mean I/sigma(I) for the high-resolution data should be greater than 3.0.

Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rint	Rsigma
Inf - 2.23	75	77	97.4	7.08	1377.5	103.56	0.0177	0.0085
2.23 - 1.73	75	75	100.0	9.09	575.4	102.60	0.0171	0.0084
1.73 - 1.47	79	79	100.0	9.28	447.7	105.41	0.0175	0.0084
1.47 - 1.32	81	81	100.0	9.62	246.1	89.44	0.0191	0.0099
1.32 - 1.21	76	76	100.0	9.03	211.7	82.63	0.0212	0.0104
1.21 - 1.14	77	77	100.0	9.08	227.9	78.60	0.0224	0.0108
1.14 - 1.08	79	79	100.0	8.56	124.2	59.78	0.0284	0.0132
1.08 - 1.02	86	86	100.0	7.77	98.5	53.04	0.0298	0.0159
1.02 - 0.98	78	78	100.0	7.78	105.3	51.83	0.0321	0.0161
0.98 - 0.94	95	95	100.0	7.80	71.5	43.72	0.0374	0.0200
0.94 - 0.90	103	103	100.0	7.20	62.5	35.89	0.0402	0.0231
0.90 - 0.87	89	89	100.0	6.63	46.5	30.32	0.0513	0.0289
0.87 - 0.84	99	99	100.0	6.74	38.5	27.15	0.0624	0.0327
0.84 - 0.82	89	89	100.0	6.66	43.1	26.73	0.0569	0.0313
0.82 - 0.80	82	82	100.0	6.40	30.6	21.75	0.0685	0.0412
0.80 - 0.78	93	93	100.0	6.29	30.8	21.51	0.0735	0.0418
0.78 - 0.76	100	100	100.0	6.14	27.1	18.60	0.0850	0.0488
0.76 - 0.74	26	28	92.9	4.18	28.3	15.58	0.0699	0.0560
0.84 - 0.74	425	427	99.5	6.24	33.2	22.11	0.0685	0.0401
Inf - 0.74	1482	1486	99.7	7.57	199.6	52.87	0.0225	0.0120

Merged [A], lowest resolution = 9.18 Angstroms, 363 outliers downweighted

Enter <CR> to continue

Figure 8.12 – Intensity statistics

5. Press [Enter] to continue.
6. Press [Enter] again to exit to the main XPREP menu.

8.1.3 Preparing an Output File

1. In the general menu, choose [C] to define the unit cell contents.

   A window opens displaying the current formula, Z, the density, and the atomic volume (see Figure 8.13). In this example, the formula is incorrect and Z has been set to six to try to achieve a reasonable density and atomic volume. Since this formula is incorrect, it must be modified now. The correct chemical formula for the YLID crystal is $C_{11}H_{10}O_2S$.

2. At “Select option”, do not accept the default answer of E. Type in [F] to enter a new formula.
3. In response to the question “Enter Formula,” type the correct formula [C11 H10 O2 S1] and press [Enter].
4. Check that the information is correct. Check that Z seems reasonable for the space group, that the density is as expected (1.1 to 1.4 for organic molecules, higher for inorganic compounds), and that the atomic volume is around 17 or 18. Significant variation from the expected values may indicate an incorrect molecular formula or missing counter ions or solvates. The values at the bottom of the window look fine for the YLID. Press [Enter] to accept.

```
Current dataset: ylid_manual_0m.hkl                Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.957   9.027  18.366   90.00   90.00   90.00    Vol     987.7
          Esds:  0.001   0.001   0.002    0.00    0.00    0.00        Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.957   9.027  18.366   90.00   90.00   90.00    Vol     987.7
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic  Space group: P2(1)2(1)2(1)  # 19 [chi]   Laue: 3
-------------------------------------------------------------------------------
Formula: C11 H10 O2 S1                                    Formula wt:   206.25
Z:  4.00  Density:  1.387  At.vol:  17.6  F(000):    432.00  Mu[mm-1]:    0.30
-------------------------------------------------------------------------------

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [Q]: f

Output file name (without extension) [ylid_manual_0m]:
```

Figure 8.13 – Defining and checking the unit cell contents

5. The next default action for XPREP is to write out the files necessary for the structure solution process (see Figure 8.14). Press [Enter] to accept.

```
Current dataset: ylid_manual_0m.hkl                Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.957   9.027  18.366   90.00   90.00   90.00    Vol     987.7
          Esds:  0.001   0.001   0.002    0.00    0.00    0.00        Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.957   9.027  18.366   90.00   90.00   90.00    Vol     987.7
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic  Space group: P2(1)2(1)2(1)  # 19 [chi]   Laue: 3
-------------------------------------------------------------------------------
Formula: C11 H12 O2 S1                                    Formula wt:   208.27
Z:  4.00  Density:  1.401  At.vol:  17.6  F(000):    440.00  Mu[mm-1]:    0.30
-------------------------------------------------------------------------------

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [F]:
```

Figure 8.14 – Requesting output files

6. The program asks for an output file name. Press [Enter] to accept.

```
Current dataset: ylid_manual_0m.hkl                Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.957   9.027  18.366   90.00   90.00   90.00    Vol     987.7
          Esds:  0.001   0.001   0.002    0.00    0.00    0.00        Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.957   9.027  18.366   90.00   90.00   90.00    Vol     987.7
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic  Space group: P2(1)2(1)2(1)  # 19 [chi]   Laue: 3
-------------------------------------------------------------------------------
Formula: C11 H12 O2 S1                                    Formula wt:   208.27
Z:  4.00  Density:  1.401  At.vol:  17.6  F(000):    440.00  Mu[mm-1]:    0.30
-------------------------------------------------------------------------------

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [F]:

Output file name (without extension) [ylid_manual_0m]:
```

Figure 8.15 – Changing the file name

7. After entering the file name or pressing [Enter] to accept the default file name, an input file for the structure solution module is created and displayed on the screen, and the program asks, “Do you wish to (over)write the intensity data file ylid_manual_0m.hkl?” Since the file name has been changed, this question **must** be answered with a “y” (see Figure 8.16).

```
Select option [F]:

Output file name (without extension) [ylid_manual_0m]: ylid_manual_0m


File ylid_manual_0m.ins set up as follows:

TITL ylid_manual_0m  P2(1)2(1)2(1)
CELL 0.71073    5.9573    9.0270  18.3664  90.000  90.000  90.000
ZERR    4.00    0.0005    0.0008   0.0019   0.000   0.000   0.000
LATT -1
SYMM 0.5-X, -Y, 0.5+Z
SYMM -X, 0.5+Y, 0.5-Z
SYMM 0.5+X, 0.5-Y, -Z
SFAC C H O S
UNIT 44 48 8 4
TEMP 0
TREF
HKLF 4
END

Do you wish to (over)write the intensity data file ylid_manual_0m?  [N]: y
```

Figure 8.16 – The input file for structure solution and a final question

8. Exit XPREP (see Figure 8.17). Press [Enter] to exit the program.

```
[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [Q]:
```

Figure 8.17 – Exit XPREP from the general menu

NOTE: There are many other features in XPREP that can be accessed from the general menu: resolution cutoff, reciprocal space plots, simulated powder patterns, and a test for merohedral twinning are very useful tools.

8.2 Simulated Precession Images

1. Under **Examine** in the task bar, click **Precession Images**. The Precession Images module provides an undistorted view of layers of the reciprocal lattice. APEX2 generates simulated precession images by finding the appropriate pixels in a series of ".sfrm" images. You must specify the images to examine and the zones to calculate.

2. To open the File Selection window, click the folder beside the file name. Choose the sets of images that you want to use for the calculation by clicking on the check boxes (see Figure 8.18).

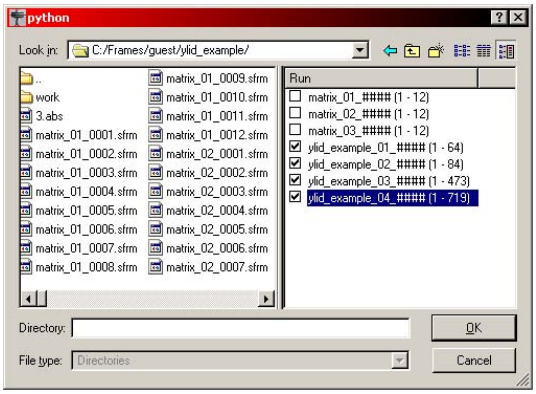


Figure 8.18 – File Selection window with matrix runs deselected

3. Check the default input value. Modify as needed and press **Calculate**.

Calculating more zones does not significantly increase the calculation time. In the example, 1kl, h1l and hk1 have been added to the defaults of 0kl, h0l and hk0. Fractional values (e.g., 0.5kl) are allowed. Reducing the resolution may speed the calculations slightly. The thickness defines the range of pixels above and below the requested range. For example, if the 0kl zone is requested with a thickness of 0.1, then the simulation is looking for all pixels that have -0.1 < h < 0.1, and any value (including fractional values) for k and l.

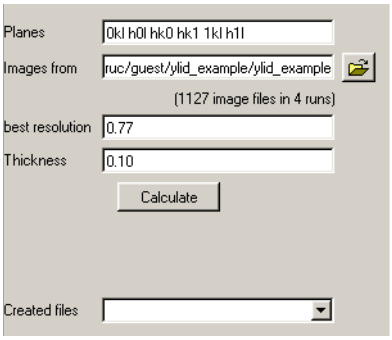


Figure 8.19 – Detail of the input section

4. A progress bar appears (Figure 8.20) and after approximately 5-10 minutes (depending on the number of ".sfrm" files read) the simulated precession image will appear on the screen. Display other planes by clicking on the calculated images to the bottom right of the work area or by browsing as usual with the **View** module.

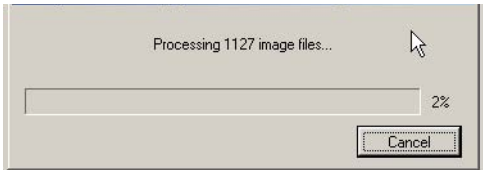


Figure 8.20 – Progress bar

5. Use the simulated patterns to check space group symmetry (see Figure 8.21, Figure 8.22, and Figure 8.23) and to find signs of twinning (Figure 8.24).

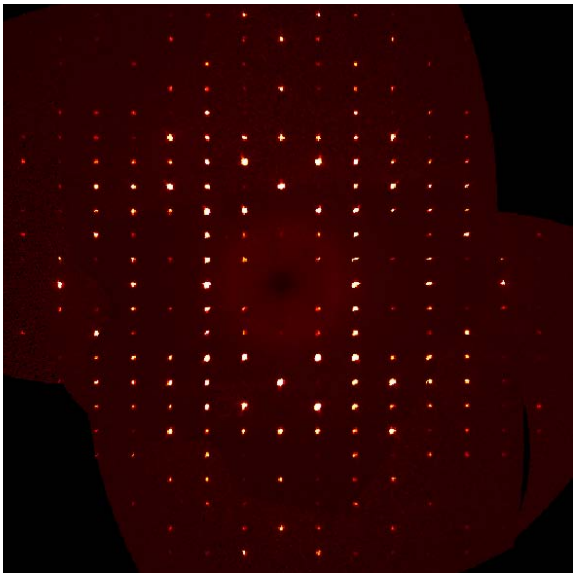


Figure 8.21 – The 0kl plane for the test YLID

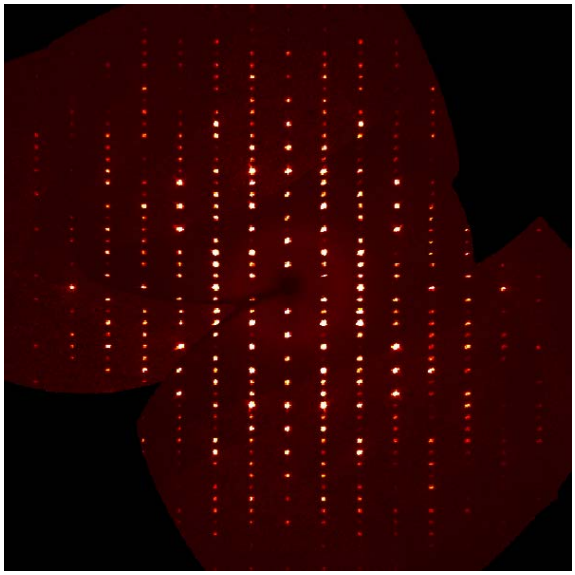


Figure 8.22 – The h0l plane for the test YLID

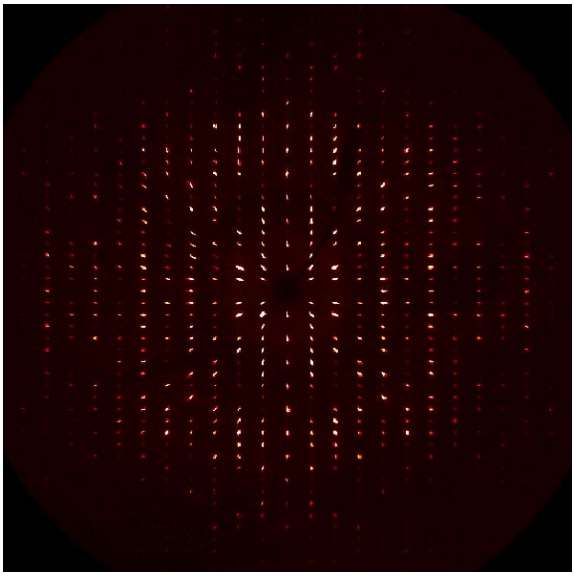


Figure 8.23 – The hk0 plane for the test YLID

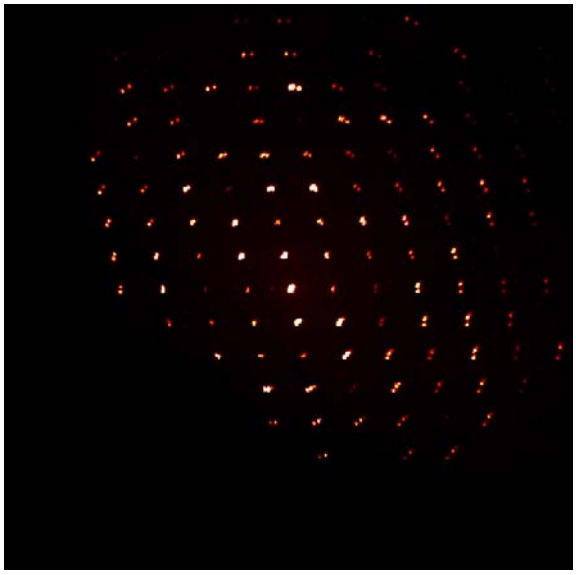


Figure 8.24 – A plane from a rotationally twinned crystal showing the two lattices

9 Structure Solution and Refinement

9.1 Overview

You are now ready to solve and refine the crystal structure. The various steps in solving and refining the structure are carried out within the APEX2 GUI. XPREP has prepared the input files for a standard direct methods run. There are two files: .ins and .hkl. In this example, the two files are ylid_0m.ins and ylid_0m.hkl.

These files are all that is required to begin the structure solution and refinement process. The various steps of solving and refining the structure are carried out using the **Solve** and **Refine** functions of the APEX2 Suite.

9.2 Solve the Structure

The process of obtaining an initial model of the compound is started by clicking in the Task Bar on **Solve Structure** and then on **Structure Solution**. This opens the Structure Solution module.

Check that there is a reasonable formula in the formula window.

1. If you have a correct formula in the formula field, then you are ready to start a normal direct methods run.
2. If the formula is incorrect, change it and press [Enter]. The input file will be updated.

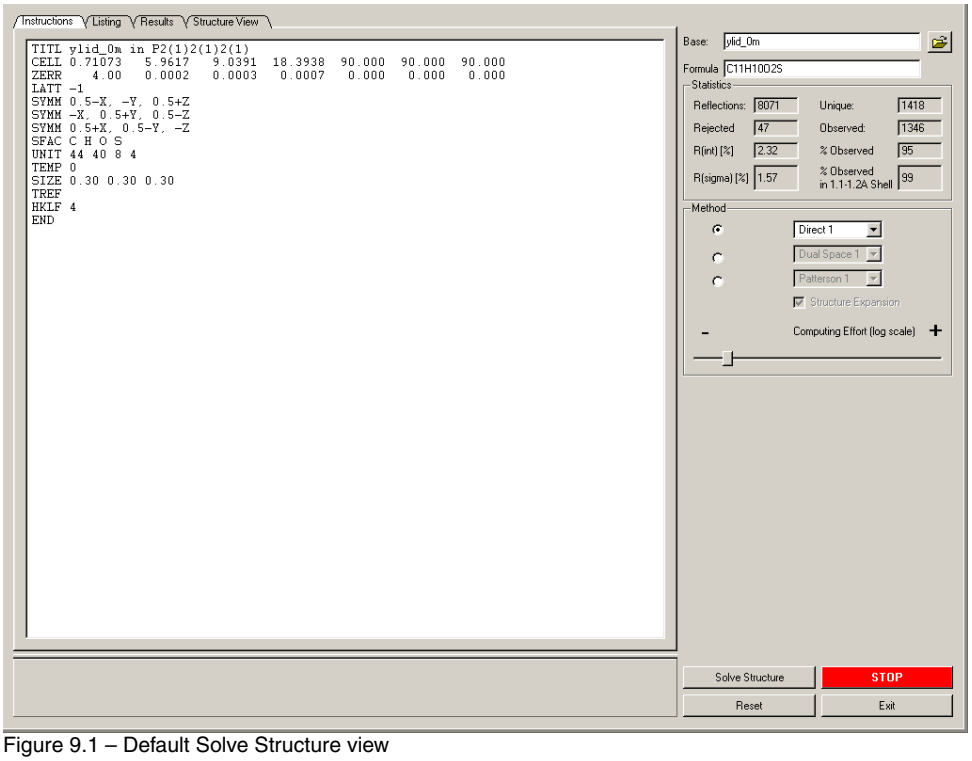


Figure 9.1 – Default Solve Structure view

3. Click **Solve Structure** to start the direct methods calculations. The upper window displays the Q-peaks of the initial model, and the text area displays the progress of the calculations.

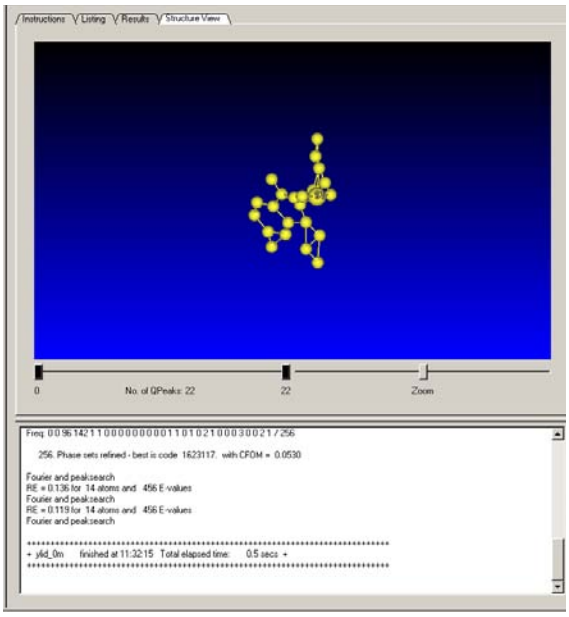


Figure 9.2 – Structure Solution output

9.2.1 Options for Direct Methods

Clicking on the arrow to the right of Direct 1 in the Method box (Figure 9.3) gives three preset choices for structure solution using direct methods.

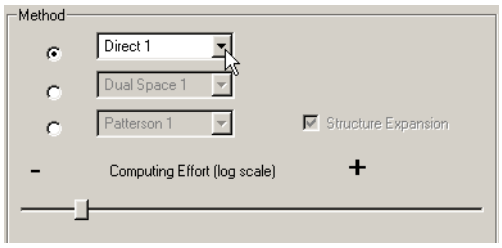


Figure 9.3 – Method box

In most cases the default values will give a good initial model, but several other options are available for more difficult problems. Chose the solution method by clicking the appropriate radio button on the left in the Method box.

Preset	Description
Direct 1	Standard settings which should be appropriate for a wide range of circumstances. Sets up a default run with a simple TREF instruction.
Direct 2	Brute force method with a higher number of direct methods attempts. Sets up an extended run with TREF 10000. By adding 10000 to the TREF command, more attempts are made to determine a solution. This will take more time, but if you see a good solution in the output window it is possible to stop at that point by clicking on the STOP button. Good solutions typically have a CFOM of 0.06 or less.
Direct 3	Brute force method with even more direct methods attempts. Sets up an extreme run with two instructions: ESEL 1.0 and TREF 100000. By adding ESEL 1.0, more reflections are used in the solution process. Increasing the number of tries in the TREF command to 100000 runs (until ended by clicking on the STOP button) is particularly useful for acentric triclinic structures and for pseudo-symmetric structures.

Table 9.1 – Direct Methods presets

At the end of the direct methods runs, the output display will look like Figure 9.2. Good figures of merit are near 1.0 for Sigma-1 and M(abs) and less than 0.06 for R_{alpha} and the CFOM. Structures can be solved with figures of merit that deviate from these numbers, but they may require more effort.

If the initial model looks reasonable, click **OK** in the output display and **Exit** in the Structure Solution module. APEX2 automatically switches to the Display tab and displays the initial solution. The next step is structure refinement using XShell (Section 9.3).

9.2.2 Options for Dual Space Methods

Two predefined options are available for the Dual Space method.

Dual Space methods are good for larger organic molecules and polypeptides.

More information on Dual Space methods and their use within APEX2 is available from the “What’s This?” Help function.

9.2.3 Options for Patterson Methods

Two predefined options are available for Patterson methods.

Patterson methods are good for finding heavy-atom positions. In general, Patterson methods favor a small number of strong scatterers.

More information on Patterson methods and their use within APEX2 is available from the “What’s This?” Help function.

9.3 Refine the Structure with XShell

The structure solution step produced statistics indicating that a solution had been found. The real proof, however, is in the initial model that is produced. XShell provides the tools to view and refine the model. A quick glance at the results of the direct methods run is often all that is needed to see that the results make chemical sense.

Control of the refinement process is quite straightforward using XShell.

1. Under **Refine Structure** in APEX2's Task Bar, click the **Structure Refinement** icon.

   This opens the Structure Refinement module, which contains tabbed sections for viewing the instructions file, listing file, results file, and a 3D model of the structure.

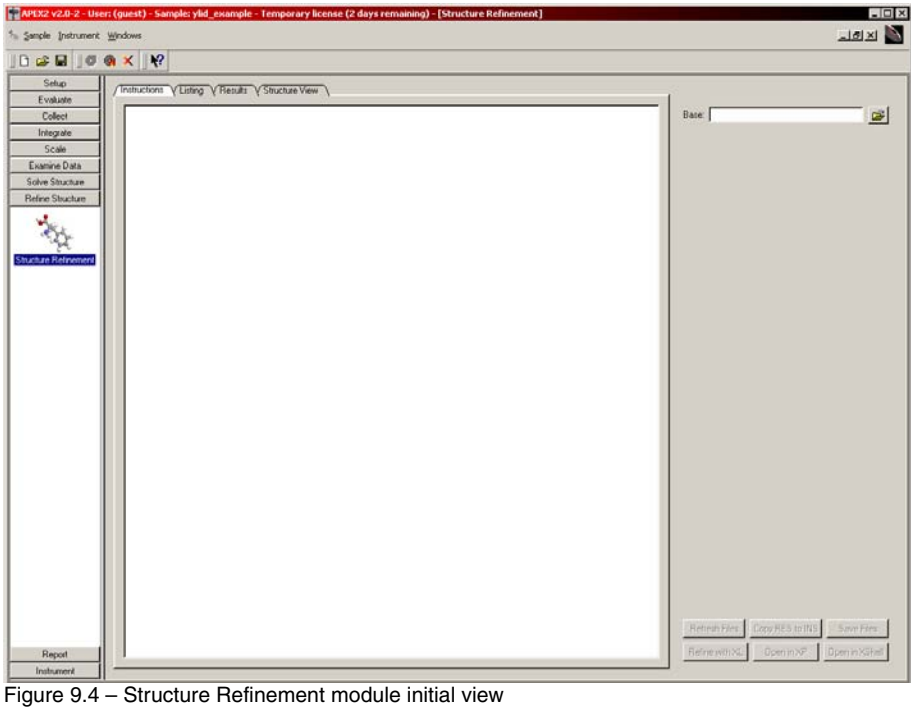


Figure 9.4 – Structure Refinement module initial view

2. Click the **Browse** icon on the right-hand side of the “Base:” field. A dialog opens in which you can select the desired .ins file.

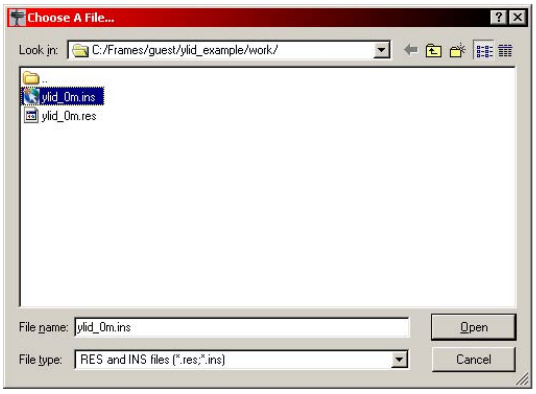


Figure 9.5 – Open file dialog

3. Click **Open**. The contents of the .ins file and any other files with the same base name are displayed under their respective tabs. Also, the six buttons in the lower right-hand corner of the module become available.

Button	Function
Refresh Files	Refresh the tabs using the absolute latest contents of all the files.
Copy RES to INS	Copies the contents of the results file into the instructions file.
Save Files	Saves any changes you have made to the files.
Refine with XL	Refines the instruction file using XL.
Open in XP	Opens XP for editing the results file.
Open in XShell	Opens the selected file in XShell.

Table 9.2 – Structure Refinement module buttons

4. Click **Open in XShell**. The XShell Main Window appears.

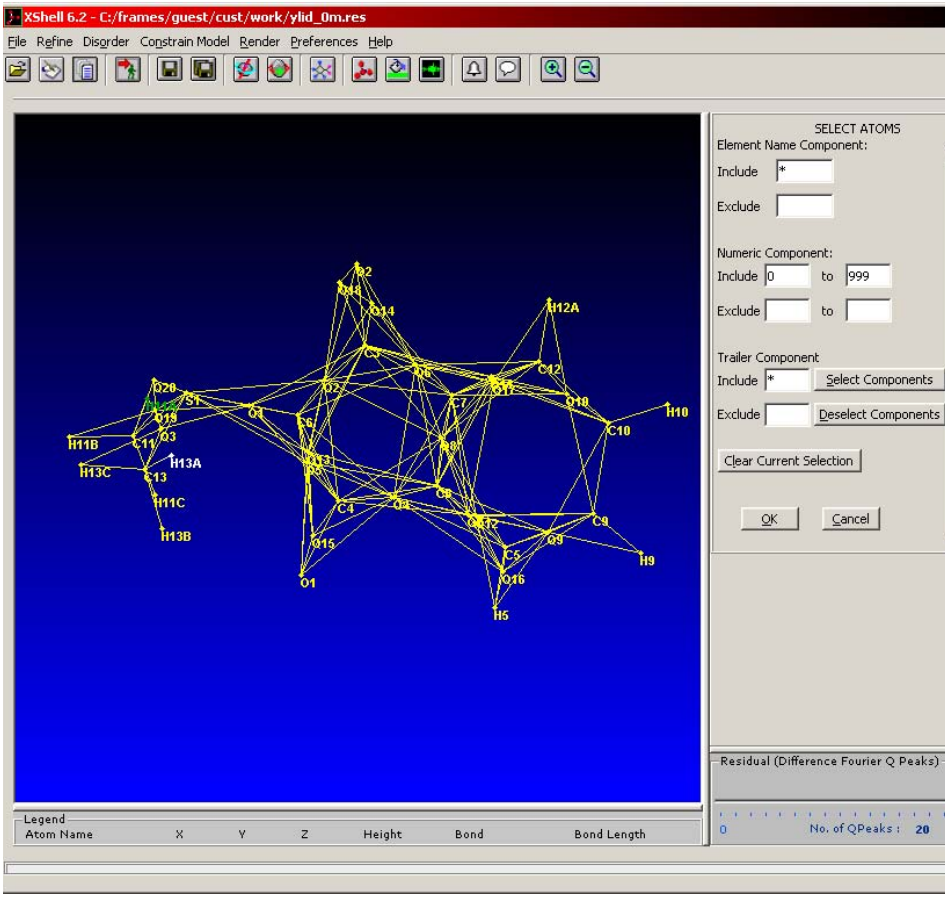


Figure 9.6 – Initial view of the YLID molecule

5. Right-click in the background to open a pop-up window with image display options.

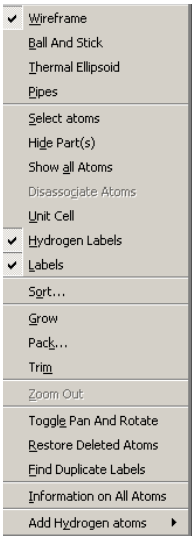


Figure 9.7 – XShell menu

6. Select the **Information on All Atoms** option to open a window displaying a list of the peaks and their heights (see Figure 9.8). The drop in height between Q13 (the last real peak) and Q14 (the first noise peak) is typical of a correct solution. Click **OK** to close this window.

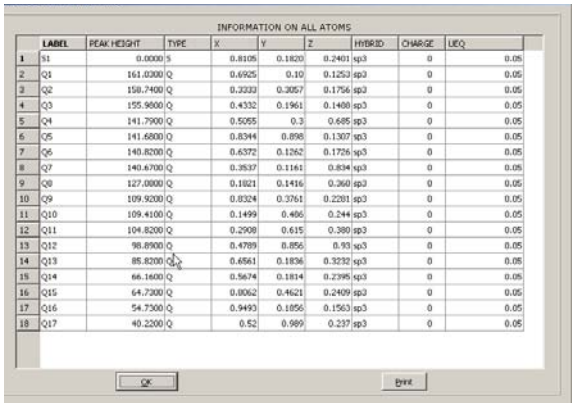


Figure 9.8 – Atom information screen

At the bottom right of the main window is a slide bar which is used for deselecting peaks. The arrow can be moved by left-clicking and dragging the arrow or by left-clicking on either side of the arrow. Left-clicking to the left of the arrow (i.e., where the mouse cursor is pointed in Figure 9.9) removes peaks.

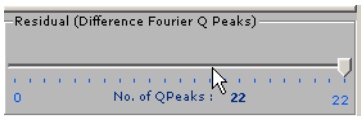


Figure 9.9 – The Q-peak slider

7. Slide the pointer down while watching the molecular display. Change the number of Q-peaks to 15. At 15 the molecule is much cleaner, but there are still peaks that do not make sense.

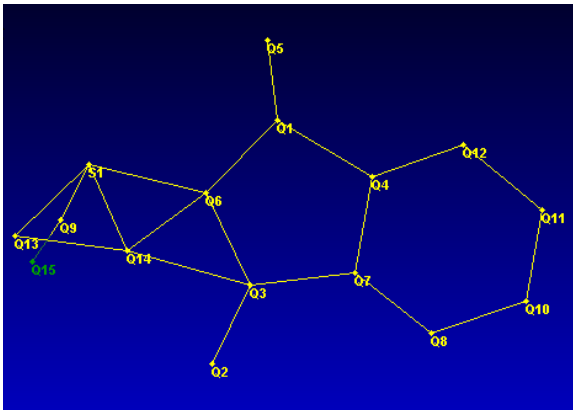


Figure 9.10 – Image after the number of Q-peaks was changed to 15

Click left on the slider until you find a reasonable model.

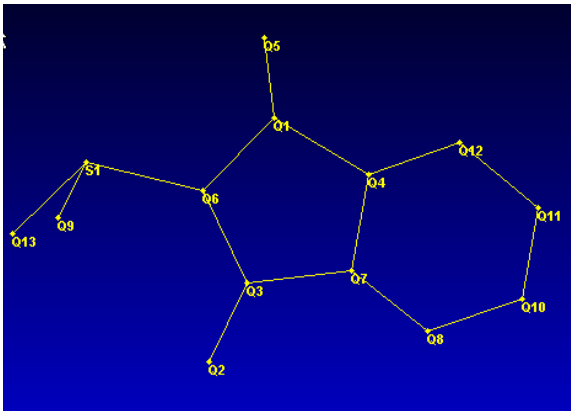


Figure 9.11 – Image with all noise peaks removed

8. Put the cursor over the bonds to check distance. The distance is displayed at the bottom right.

9.3.1 Label the Atoms

1. Label the atoms and set the atom types. Left-click atoms to select them. Left-click the peaks for the two oxygen atoms (Q2 and Q5 in this example).

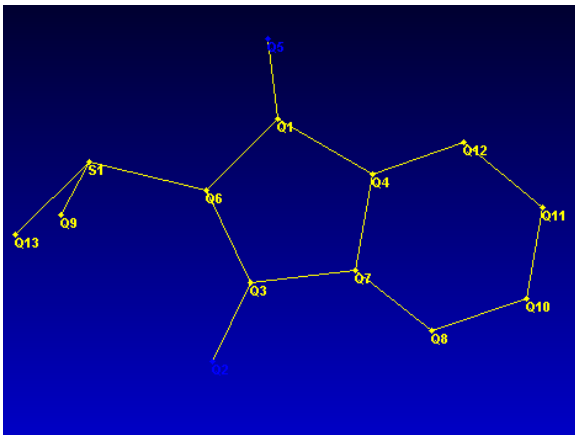


Figure 9.12 – View of model with the probable oxygen peaks selected

2. If it is difficult to see the color and labels, change the color scheme with **Preferences > Background Color**. Choose colors and click **Apply**. Click **Cancel** to exit the background color mode. Note that the selected peaks are blue in Figure 9.14.

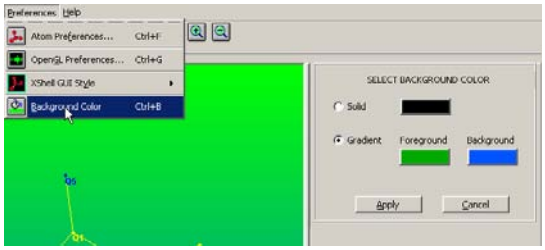


Figure 9.13 – Selecting the background color

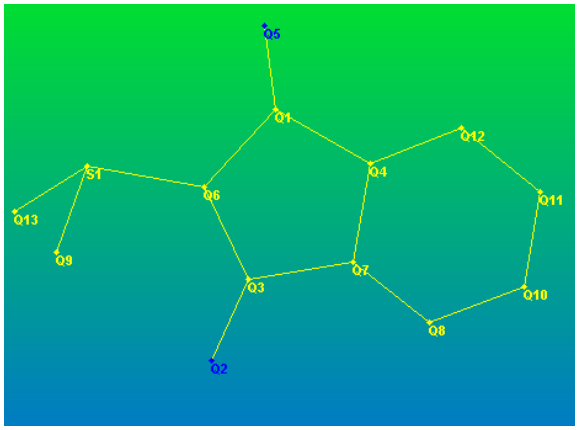


Figure 9.14 – New background color

3. Right-click to view a pop-up menu and select **Labelling...**. The Atom Labelling box opens.

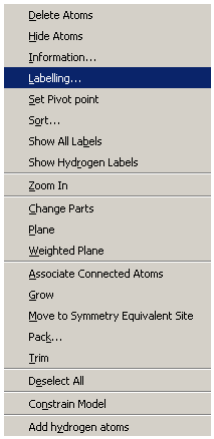


Figure 9.15 – Select Labelling

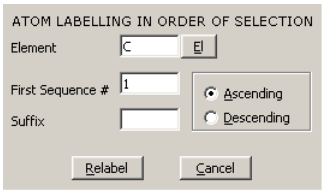


Figure 9.16 – Atom Labelling box

4. The two selected atoms need to be changed to oxygen atoms. Do this one of two ways:

- Click the Element field and type in the element symbol (case does not matter).
- Click the **El** button to the right of the Element field to open a periodic table. Click the appropriate element symbol to select it (the periodic table will automatically close).

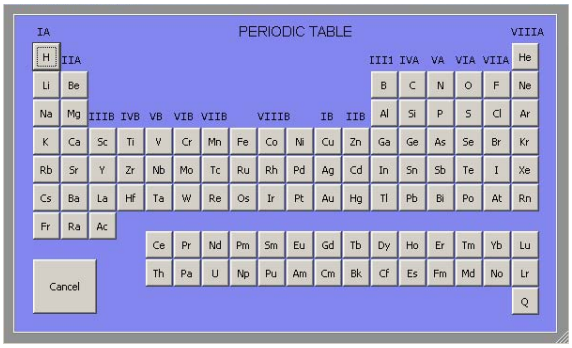


Figure 9.17 – The periodic table for selecting atom types

5. Once the element type is set correctly, click **Relabel** to number the peaks sequentially in the order they were selected, starting with the number in the First Sequence # field.

6. Seeing the color of atoms can be difficult in the Wireframe view used so far. Right-click in the background with no atoms selected to get a slightly different action menu. In this menu, highlight and left-click **Pipes**. This makes the bonds thicker and makes it obvious that there is an incorrectly labeled atom. This will be corrected when the carbon atoms are labeled.

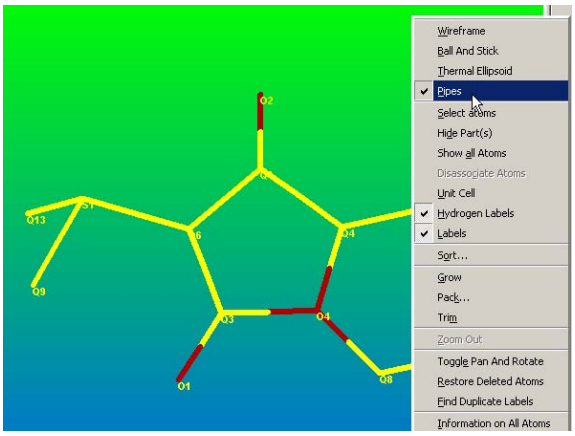


Figure 9.18 – Select the pipes view

7. Click on the remaining peaks in the order that you want them labeled.

NOTE: If working on YLID test data, look at Figure 9.19 and click on the peaks to give the same order as used here. Right-click and select **Labelling**. Select Carbon and apply the labels.

8. In the Labelling tool, change the element type to C (carbon). The starting atom number changes to 1—the first available number for carbon atoms.

9. Click **Relabel**.

10. Click **Cancel** to close the window. The labeled YLID molecule is now ready for refinement.

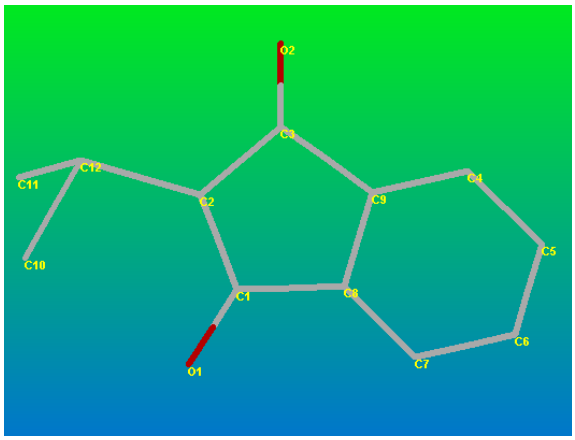


Figure 9.19 – Correctly labeled model

9.3.2 Refine the Model

1. Click the **Refine** button in the Tool Icon Bar (the cursor is pointing to it in Figure 9.20), type [Ctrl+R], or select **Refine > Refine** in Xshell's Menu Bar. This opens the Refine Box.

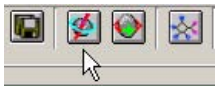


Figure 9.20 – The Refine icon

2. Click **Refine** to launch XL (the least-squares refinement program) using the default parameters as given in Figure 9.21.

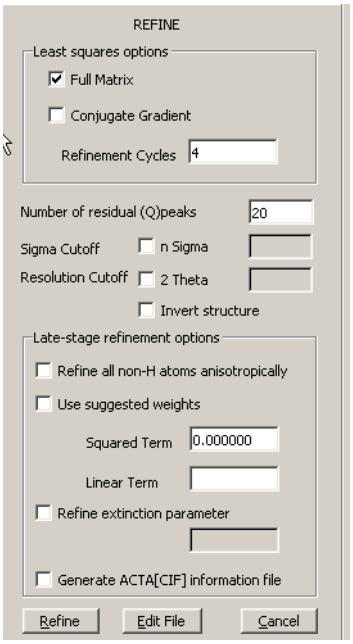


Figure 9.21 – The Refine menu

After a few seconds, a Refine window will open with output results for the calculation.

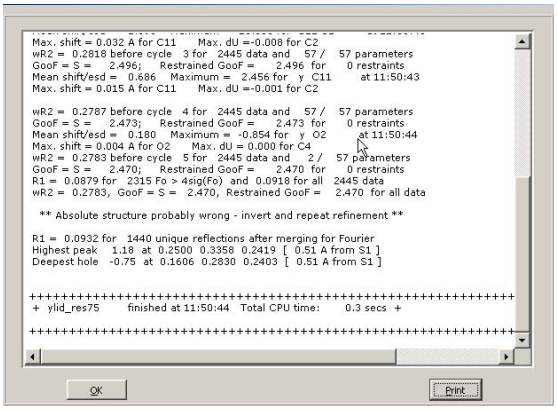


Figure 9.22 – Isotropic refinement output

In the output displayed above, note that the R1 value is 0.09. This is typical for a preliminary isotropic refinement for an organic molecule with data to a resolution of 0.75 and no hydrogen atoms included. The refinement program also indicates that the model needs to be inverted to get the correct absolute structure.

NOTE: This indication of the absolute configuration is usually reliable, but all assignments of absolute structure should be confirmed later in the refinement process by including TWIN and BASF cards in the instruction input.

3. Click **OK** to return to XShell. The molecule display refreshes with the results of the least-squares calculations.

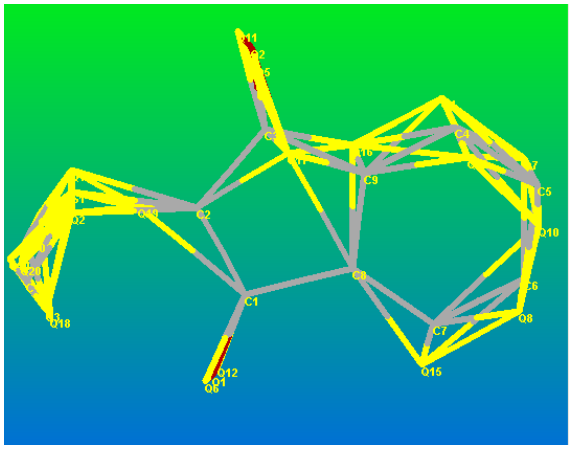


Figure 9.23 – The model after isotropic refinement

The peaks in the diagram represent difference in the electron density between the refinement model and the experimental electron density as defined by the measured data. Many of these difference peaks are near the sulfur and oxygen atoms. Refinement of all atoms anisotropically should improve the model.

4. In the refinement box, click **Invert Structure** and **Refine All Non-H Atoms Anisotropically** as in Figure 9.24.

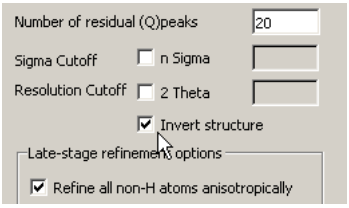


Figure 9.24 – Inverting and choosing anisotropic refinement

5. Click the **Refine** button to launch the least-squares refinement program. The output window opens and a summary of the results of individual cycles of refinement appears.

```
GooF = S =    1.487;    Restrained GooF =     1.487  for     0 restraints
Mean shift/esd =  1.718   Maximum =  13.893 for  U13 S1        at 19:05:00
Max. shift = 0.009 A for C5     Max. dU = 0.003 for C5

wR2 =  0.1407 before cycle   3 for   2389 data and   127 /   127 parameters
GooF = S =    1.184;    Restrained GooF =     1.184  for     0 restraints
Mean shift/esd =  0.422   Maximum =  2.423 for  U11 C6        at 19:05:00
Max. shift = 0.004 A for C5     Max. dU = 0.002 for C6

wR2 =  0.1391 before cycle   4 for   2389 data and   127 /   127 parameters
GooF = S =    1.167;    Restrained GooF =     1.167  for     0 restraints
Mean shift/esd =  0.101   Maximum =  -0.688 for  U13 C3        at 19:05:00
Max. shift = 0.002 A for C5     Max. dU = 0.000 for C5
wR2 =  0.1389 before cycle   5 for   2389 data and     2 /   127 parameters
GooF = S =    1.164;    Restrained GooF =     1.164  for     0 restraints
R1 =  0.0459 for   2262 Fo > 4sig(Fo)  and  0.0476 for all   2389 data
wR2 =  0.1389,  GooF = S =   1.164,  Restrained GooF =    1.164  for all data
R1 =  0.0498 for   1418 unique reflections after merging for Fourier

Highest peak    0.47  at  0.8147  0.9219  0.2317  [  0.97 A from C10 ]
Deepest hole   -0.27  at  0.2028  0.5207  0.0249  [  1.27 A from C6 ]

++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  ylid_0m      finished at 19:05:00   Total elapsed time:      0.6 secs  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

Figure 9.25 – Least-squares refinement summary

The value for R1, 0.0459, is typical for an initial anisotropic refinement with no hydrogen atoms included. Since the mean shift/esd is greater than 0.1, the model is still changing. Since the highest peak in the difference map has a height of 0.47 and is 0.97Å from C10, there are certainly some hydrogen atoms to be included.

Examination of the difference peaks in the resulting difference electron density map shows that the top difference peaks all correspond to hydrogen atoms.

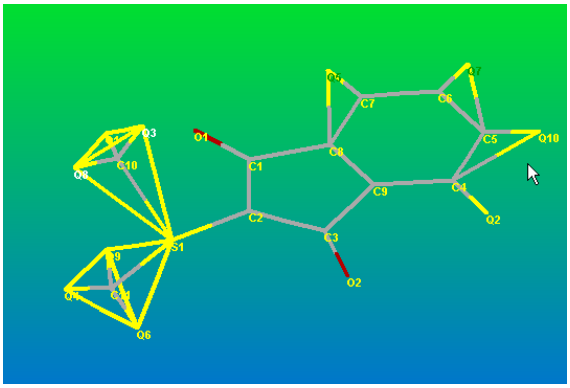


Figure 9.26 – Difference peaks correspond to expected hydrogen atom positions

9.3.3 View Atomic Displacement Parameters (Thermal Ellipsoids)

It is generally good practice to examine the displacement parameters at this point in structure determination.

1. Remove difference peaks with the slider tool.

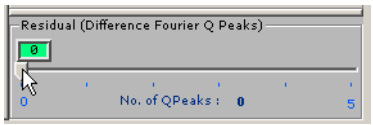


Figure 9.27 – Removing all Q-peaks with the slider bar

2. Right-click on the background and select **Thermal Ellipsoids**.

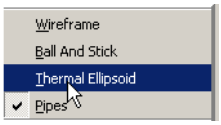


Figure 9.28 – Selecting Thermal Ellipsoids

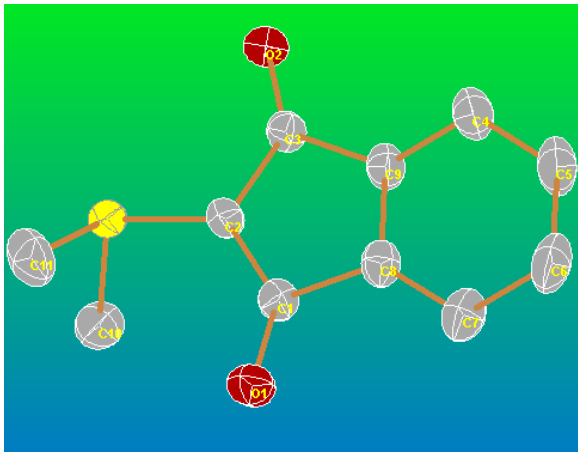


Figure 9.29 – Thermal Ellipsoid plot

Alternatively, a list of Ueq's (equivalent isotropic displacement parameters) can be generated by requesting information on all of the atoms. Examining these values for outliers is good practice.

3. Right-click on the background and click **Information on All Atoms**.

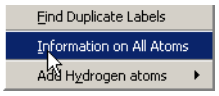
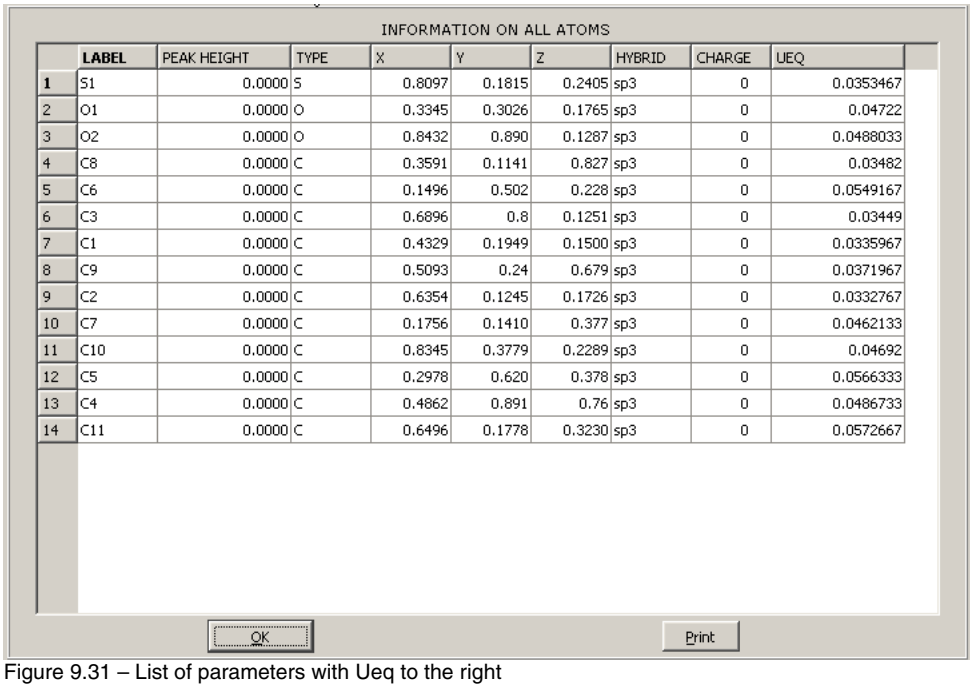


Figure 9.30 – Selecting Information on All Atoms

This produces the table in Figure 9.31.



INFORMATION ON ALL ATOMS

	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.8097	0.1815	0.2405	sp3	0	0.0353467
2	O1	0.0000	O	0.3345	0.3026	0.1765	sp3	0	0.04722
3	O2	0.0000	O	0.8432	0.890	0.1287	sp3	0	0.0488033
4	C8	0.0000	C	0.3591	0.1141	0.827	sp3	0	0.03482
5	C6	0.0000	C	0.1496	0.502	0.228	sp3	0	0.0549167
6	C3	0.0000	C	0.6896	0.8	0.1251	sp3	0	0.03449
7	C1	0.0000	C	0.4329	0.1949	0.1500	sp3	0	0.0335967
8	C9	0.0000	C	0.5093	0.24	0.679	sp3	0	0.0371967
9	C2	0.0000	C	0.6354	0.1245	0.1726	sp3	0	0.0332767
10	C7	0.0000	C	0.1756	0.1410	0.377	sp3	0	0.0462133
11	C10	0.0000	C	0.8345	0.3779	0.2289	sp3	0	0.04692
12	C5	0.0000	C	0.2978	0.620	0.378	sp3	0	0.0566333
13	C4	0.0000	C	0.4862	0.891	0.76	sp3	0	0.0486733
14	C11	0.0000	C	0.6496	0.1778	0.3230	sp3	0	0.0572667

OK Print

Figure 9.31 – List of parameters with Ueq to the right

Examining the values here is difficult because the atoms are not in order.

9.3.4 Sort Atoms

1. Right-click on the background and click **Sort…** to sort the atoms into a sensible order.

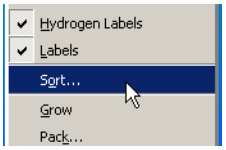


Figure 9.32 – Select the Sort option

An Atom List box appears to the right of the molecule display. Since S1, O1 and O2 are in a reasonable order, only the carbon atoms need to be ordered.

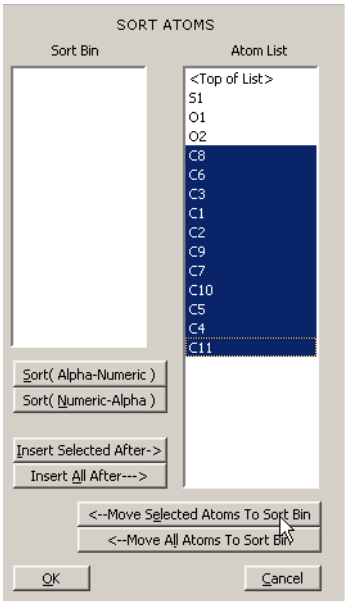


Figure 9.33 – Selecting carbons for sorting

NOTE: Generally, it is easier to sort and number atoms before adding hydrogen atoms.

2. Highlight the atoms to be sorted (i.e., left-click and drag over the atoms) and move them to the Sort Bin (i.e., click **Move Selected Atoms to Sort Bin**).

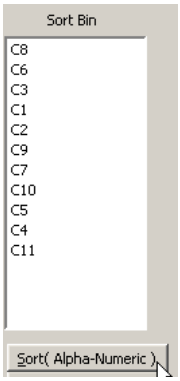


Figure 9.34 – Carbon atoms in Sort Bin

3. After the atoms are in the Sort Bin, click **Sort(Alpha-Numeric)** to get them into a normal order.

4. Highlight O2 and click **Insert All After** as in Figure 9.35.

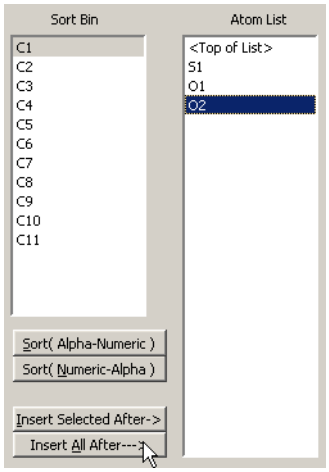
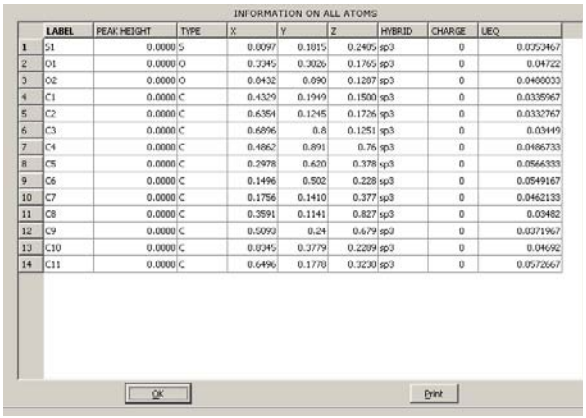


Figure 9.35 – Sorted atoms with “insert after” atom (O2) selected.

5. Click **OK** to accept the sorting.

Figure 9.36 – OK button

The resulting list of atom information is easier to examine.



	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.0097	0.1015	0.2405	sp3	0	0.0353467
2	O1	0.0000	O	0.2345	0.3026	0.1765	sp3	0	0.04722
3	O2	0.0000	O	0.0432	0.090	0.1287	sp3	0	0.0406033
4	C1	0.0000	C	0.4329	0.1949	0.1500	sp3	0	0.0335967
5	C2	0.0000	C	0.6354	0.1245	0.1726	sp3	0	0.0332767
6	C3	0.0000	C	0.6896	0.8	0.1251	sp3	0	0.03449
7	C4	0.0000	C	0.4862	0.891	0.76	sp3	0	0.0486733
8	C5	0.0000	C	0.2978	0.620	0.378	sp3	0	0.0566333
9	C6	0.0000	C	0.1496	0.502	0.228	sp3	0	0.0549167
10	C7	0.0000	C	0.1756	0.1410	0.377	sp3	0	0.0462133
11	C8	0.0000	C	0.3591	0.1141	0.827	sp3	0	0.03482
12	C9	0.0000	C	0.5093	0.24	0.679	sp3	0	0.0371967
13	C10	0.0000	C	0.0345	0.3779	0.2289	sp3	0	0.04692
14	C11	0.0000	C	0.6496	0.1770	0.3230	sp3	0	0.0572667

Figure 9.37 – Sorted atoms

The Ueq values are reasonable if there are:

- lower values for the sulfur atom and the five-member ring
- higher values for the terminal atoms — methyls and carbonyls
- sensible numbers for the six-member ring — lower for C8 and C9, higher for C5 and C6, and in between for C4 and C7.

9.3.5 Add Hydrogen Atoms

At this point, refine the hydrogen atoms either by renaming the Q-peaks using the Labelling tool as before and then including them in the refinement, or include the hydrogen atoms in fixed idealized positions riding on the atoms to which they are attached.

In this User Manual we will illustrate the latter fixed idealized method.

NOTE: In some cases (e.g., hydrogen atoms on hetero atoms or on geometrically strained carbon atoms), it is better to refine the hydrogen atom positions. A general rule is to refine if there is some doubt about the position of the hydrogen atom and there is difference electron density present.

---

1. Using the slider tool, remove all of the Q-peaks.

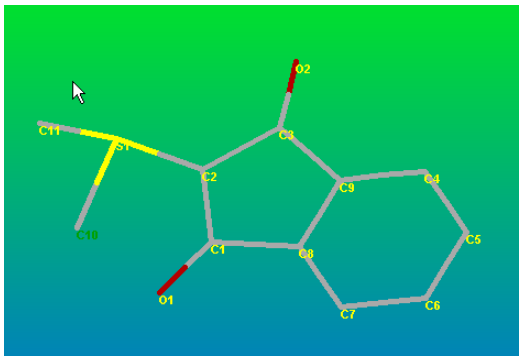


Figure 9.38 – Refined model with difference peaks removed

2. Right-click on the background and click **Add Hydrogen atoms > Hybridize All**.

Figure 9.39 – Hybridize button

The colors of the atom labels change to indicate the atom hybridization.

NOTE: If the atom hybridizations are hard to see, right-click on the background and open the Information on All Atoms window.

---

3. Right-click in the background and click **Add Hydrogen atoms > Calculate Hydrogens**.

Figure 9.40 – Calculate Hydrogens button

4. A warning appears indicating that the hydrogen atoms have been provisionally added and that the final addition with naming will take place when the least-squares calculations are started (Figure 9.41). Click **OK**.

Figure 9.41 – Hydrogen atom warning

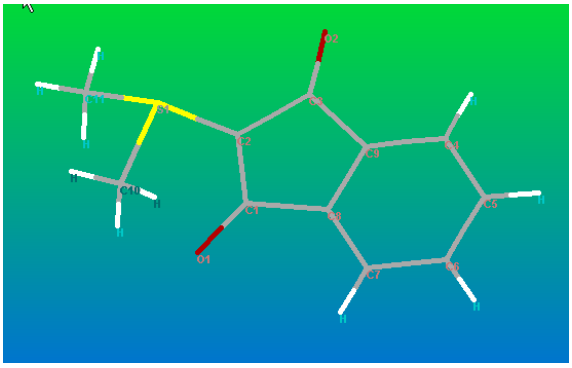


Figure 9.42 – Model with added hydrogen atoms

5. Click the **Refine** button to launch the least-squares refinement program. The output window opens, and a summary of the results of individual refinement cycles appears.

```
GooF = S =    0.726;    Restrained GooF =    0.728  for     0 restraints
Mean shift/esd =   0.693   Maximum =  -3.778 for   y  C11       at 19:16:48
Max. shift = 0.013 A for H11B     Max. dU = 0.000 for C4

wR2 =  0.0841 before cycle   3 for   2389 data and   129 /   129 parameters
GooF = S =    0.701;    Restrained GooF =    0.701  for     0 restraints
Mean shift/esd =   0.104   Maximum =  -0.681 for  U13 C4       at 19:16:48
Max. shift = 0.005 A for H10B     Max. dU = 0.000 for C8

wR2 =  0.0840 before cycle   4 for   2389 data and   129 /   129 parameters
GooF = S =    0.701;    Restrained GooF =    0.701  for     0 restraints
Mean shift/esd =   0.043   Maximum =   0.265 for   y  C11       at 19:16:48
Max. shift = 0.002 A for H11A     Max. dU = 0.000 for C4

wR2 =  0.0840 before cycle   5 for   2389 data and     2 /   129 parameters
GooF = S =    0.700;    Restrained GooF =    0.700  for     0 restraints
R1 =  0.0281 for   2262 Fo > 4sig(Fo)  and  0.0296 for all   2389 data
wR2 =  0.0840,  GooF = S =   0.700,  Restrained GooF =    0.700  for all data
R1 =  0.0289 for   1418 unique reflections after merging for Fourier
Highest peak    0.23  at  0.8218  0.5646  0.1540  [  0.68 A from C2 ]
Deepest hole   -0.19  at  0.1976  0.5182  0.0223  [  1.26 A from C6 ]

++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  ylid_0m       finished at 19:16:48   Total elapsed time:       0.9 secs  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
```

Figure 9.43 – Refinement results

The value for R1, 0.0281, is typical for an anisotropic refinement with riding hydrogen atoms and a data set measured to 0.75Å. Since the mean shift/esd is less than 0.01, the model is not changing significantly. Since the highest peak and the deepest hole in the difference map are similar, there are probably no missing atoms.

The resulting difference electron density map appears rather ugly. This is because the number of difference peaks to be displayed has been left at 20.

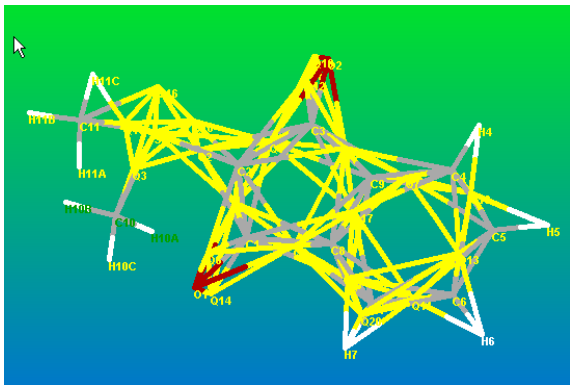


Figure 9.44 – Difference peaks in a well-refined model

9.3.6 Final Refinement for Publication

Obtain a view that is much easier to interpret by reducing the number of difference peaks in the **Refine** menu (see Figure 9.45).

1. For the final cycles of refinement, reduce the number of peaks to be displayed to five and turn on “Use Suggested Weights” and “Generate ACTA(CIF) Information File” as shown in the next two figures.

NOTE: If the suggested weights vary significantly from 0.06 and 0.00, then change the values to 0.06 and 0.00.

Figure 9.45 – Reduce the number of difference peaks

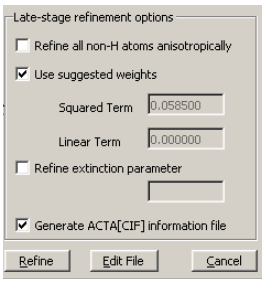


Figure 9.46 – Select weights and request ACTA output

Figure 9.47 shows a difference map with the number of peaks set at five.

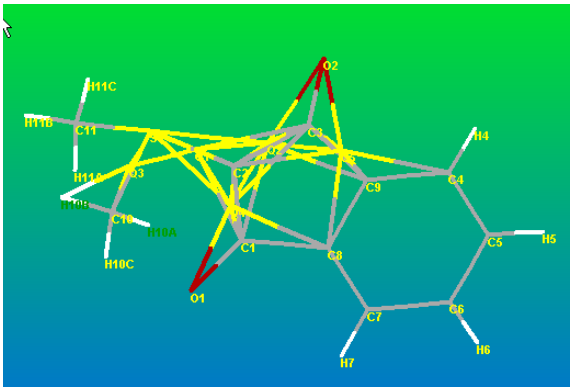


Figure 9.47 – Difference peaks

2. Click **Edit File**, choose *.INS and click **OK** to open the instruction file for XL. This allows you to add instructions that are not directly available in the GUI.

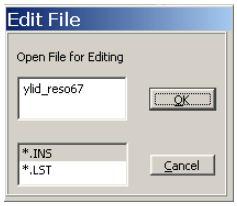


Figure 9.48 – Open the .ins file for editing

3. Check that the temperature card is correct.

4. If torsion angles are of interest, add a CONF (confirmation) card (see Figure 9.49).

```
TEMP 23
conf
mpla 12 c1 c2 c3 c4 c5 c6 c7 c8 c9  s1  o1 o2 c10 c11
mpla 6 c4 c5 c6 c7 c8 c9 c1 c2 c3  o1 o2 s1 c10 c11
mpla 5 c8 c9 c1 c2 c3 c4 c5 c6 c7  o1 o2 s1 c10 c11
mpla 10 c1 c2 c3 c4 c5 c6 c7 c8 c9  s1  o1 o2 c10 c11
```

Figure 9.49 – Add instructions in the .ins file

5. If planarity and angles between planes are of interest, add mpla cards.

9.3.7 Generate an Atomic Displacement (Thermal Ellipsoid) Plot

1. Remove difference peaks with the slider tool.
2. Right-click on the background and select **Thermal Ellipsoids**.

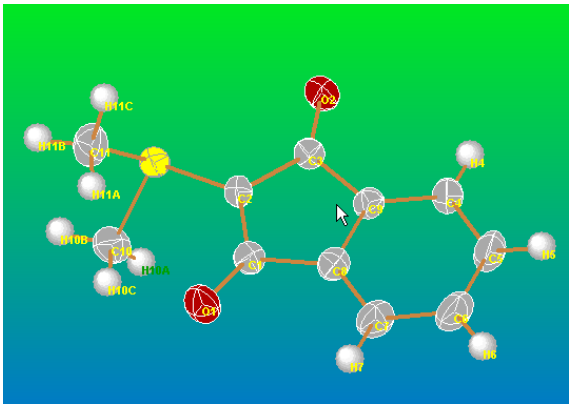


Figure 9.50 – Thermal ellipsoids with large hydrogen atoms

3. If the hydrogen atoms in Figure 9.50 are large, change their sizes by going to **Preferences > Atom Preferences...** in XShell's Menu Bar.

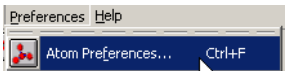


Figure 9.51 – Atom Preferences tool

At the top of the box that is opened, there are three values that can be changed to adjust the size of atoms and the bonds to them.

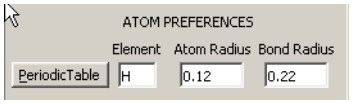


Figure 9.52 – Default atom preferences

4. Change the hydrogen radius to 0.08 and the bond radius to 0.15 to make the hydrogen atoms smaller. Click **Apply**.

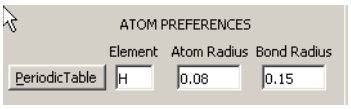


Figure 9.53 – Modified atom preferences

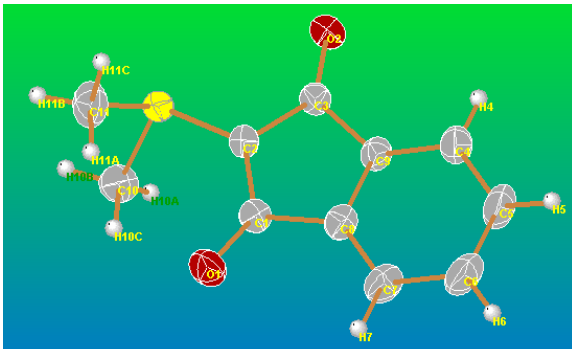


Figure 9.54 – Thermal ellipsoid plot with smaller hydrogen atoms

5. Position atom labels by right-clicking on the atom and choosing **Position Label**.

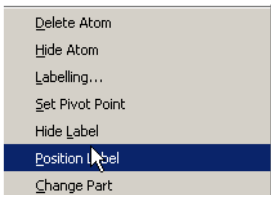


Figure 9.55 – Position Label menu

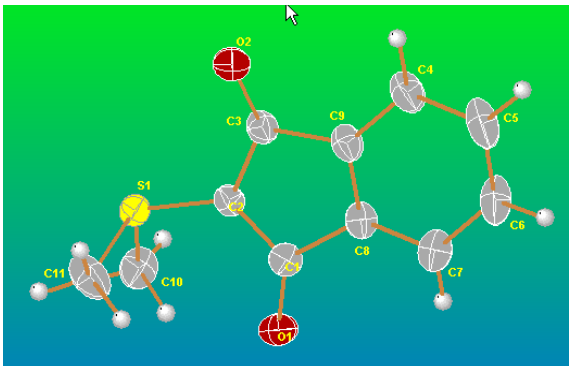


Figure 9.56 – Positioned labels

6. Convert this image into a high-quality .jpg file for inclusion into reports by clicking **Render** in the Windows Tool Bar and choosing **Color > To High Quality JPEG File**.

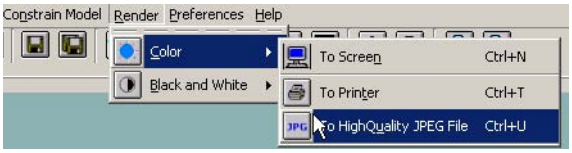


Figure 9.57 – Generate JPEG

NOTE: A message may appear that the font file needs to be defined: “Please set your font directory for the first time for opengl rendering.” If this happens, click **Preferences > OpenGL Preferences** and click on the TrueType font For.OpenGL.

In Windows, browse to C:\bn\xshell\fonts\

In Linux, navigate to /usr/local/bin/xshell/fonts. Choose a font.

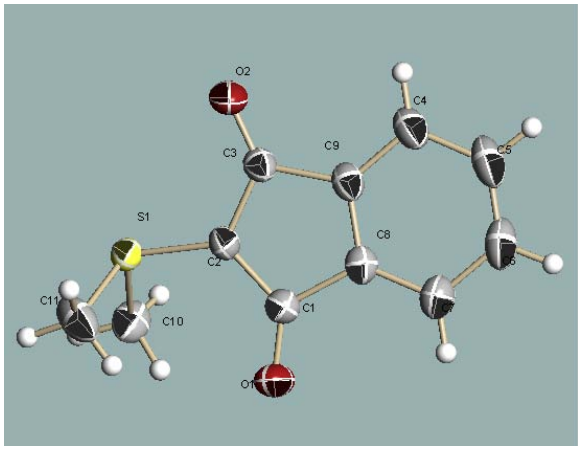


Figure 9.58 – The final rendered thermal ellipsoid/atomic displacement parameter plot

Appendix A RLATT

RLATT is a very powerful tool for viewing the harvested reflections in a reciprocal lattice. This Appendix covers only a few of its features. More information on RLATT is available in M86-Exx045 RLATT User Manual.

This example has over 3300 reflections. This is not the typical case; generally only 300 or 400 reflections are examined. Using extra reflections will make the examples easier to see.

A.1 Open RLATT

1. Under **Evaluate** in APEX2's Task Bar, click **Reciprocal Lattice Viewer**. RLATT (The Reciprocal Lattice Viewer module) opens.

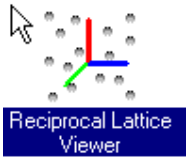


Figure A.1 – RLATT icon

2. RLATT will automatically read in the reflections from the current project and display them as a reciprocal space plot.

Figure A.2 is a typical unoriented view after import. The gray circles are more intense reflections, i.e., those with higher I/sigma(I). The black dots represent less intense reflections. The intensity key slider at the bottom of the window can be used to change the selection criterion.

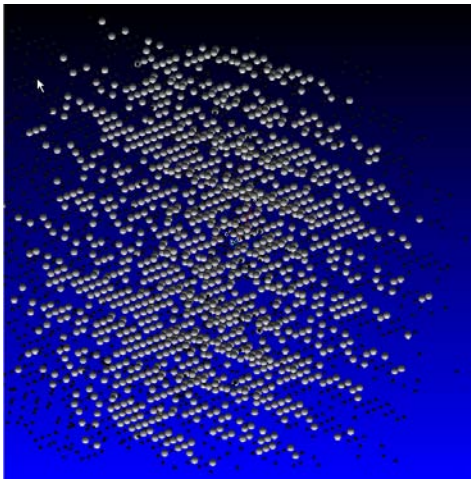


Figure A.2 – Unoriented RLATT view

Other tools can be accessed by right-clicking on the background to give the “Quick Tools” menu (Figure A.3) and by clicking on RLATT in the menu bar (Figure A.4).

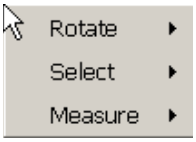


Figure A.3 – Quick Tools menu

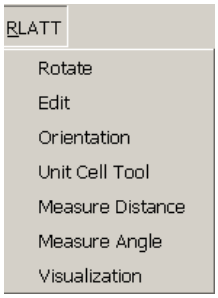


Figure A.4 – Full menu tools

Additional context-sensitive information is available by left-clicking on the “What’s This” help arrow in the menu bar.

Figure A.5 – The “What’s This“ help arrow

For example, clicking on the help arrow and then clicking in the work area (the gray area to the right of the lattice display) gives the display of shortcuts shown in Figure A.6. Clicking on the blue background gives the hints shown in Figure A.7.

RLATT Keyboard Commands

Rotate & Zoom:

UP / DOWN	= rotate along x
LEFT / RIGHT	= rotate along y
INS / DEL	= rotate along z
+ / -	= zoom
hold SHIFT	= 4x speed
hold CTRL	= 1/4 speed
hold SHIFT + CTRL	= 16x speed

Selection Tools:

SHIFT + left-click	= add spots to current selection
CTRL + left-click	= remove spots from current selection
UP, DOWN, LEFT, RIGHT	= move selection tool
hold SHIFT	= 4x speed (during movement only)
hold CTRL	= 1/4 speed (during movement only)
hold SHIFT + CTRL	= 16x speed (during movement only)
+	= add slice (lattice tool only)
-	= remove Slice (lattice tool only)
PGUP	= add Extension (lattice tool only)
PGDN	= remove Extension (lattice tool only)
ALT + left-click	= lock onto spot (lattice tool only)

Orientations:

F1	= 1 0 0 (along a*)
F2	= 0 1 0 (along b*)
F3	= 0 0 1 (along c*)
F4	= unoriented (identity matrix)
F5	= user-defined orientation 1
F6	= user-defined orientation 2
F7	= user-defined orientation 3
F8	= user-defined orientation 4
SHIFT + F5	= store user-defined orientation 1
SHIFT + F6	= store user-defined orientation 2
SHIFT + F7	= store user-defined orientation 3
SHIFT + F8	= store user-defined orientation 4

Distance Measurement:

+	= add slice
-	= remove Slice
PGUP	= add Extension
PGDN	= remove Extension
hold SHIFT	= action x 10
ALT + left-click	= lock onto spot

Angle Measurement:

ALT + left-click	= lock onto spot (locking onto all three calculates the angle in 3D)

Misc:

[	= adjust intensity slider
]	= adjust intensity slider
A	= change axis modes
C	= change colorization method
I	= invert selection
M	= toggle distance measurement during lattice selection tool
N	= toggle Null Resolution Mode
S	= change 2D Projection Scale mode
U	= toggle Unit Cell Tool

Figure A.6 – Shortcut keys and commands

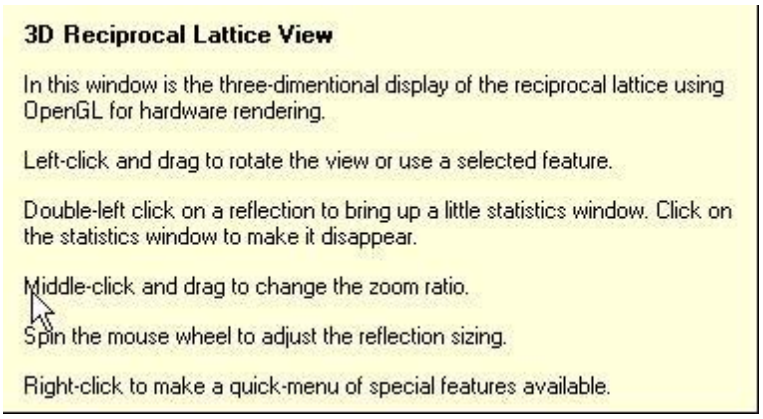


Figure A.7 – Help for the 3D display obtained by clicking the "What's This" arrow on the blue display background

A.2 Orienting Views

Move the mouse to rotate the RLATT display. It is possible to easily see rows and non-fitting peaks (see Figure A.8).

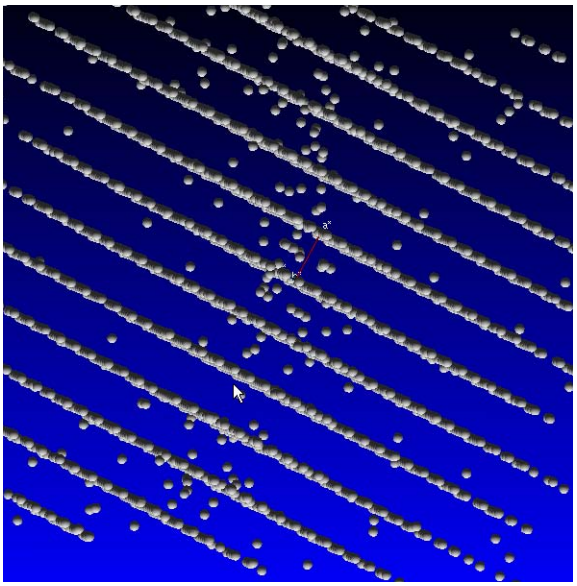


Figure A.8 – RLATT display looking down layers of reflections

By sliding the intensity key at the bottom of the display, it is easy to deselect weak reflections. In Figure A.9 the reflections between the layer lines all go black when the intensity filter is moved to the left.

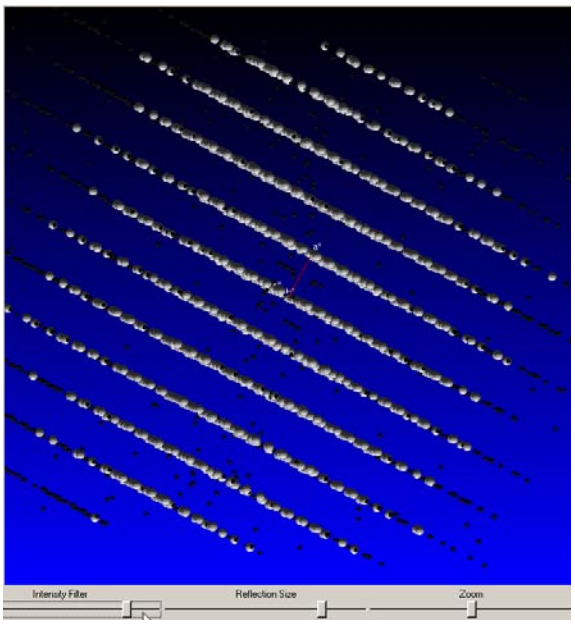


Figure A.9 – RLATT display looking down layers of reflections. Weaker reflections, i.e., those with smaller I/sigma(I), are black because the “Intensity Filter” slider (bottom left) has been moved slightly to the left.

With additional rotations, the layers of reflections can be further oriented so that stacks of reflections become visible. Sometimes it is quite useful to right-click on the background and choose Z-rotations from the rotation options. The 2D profiles on the top and to the left are a valuable aid in this process. With practice, the 2D profiles can be organized into clusters. As shown, the 2D profiles are counting intensity. The counting mode can be changed to spot count or turned off completely using the Visualization menu (**RLATT > Visualization**) or by pressing the [s] key to cycle through the counting modes.

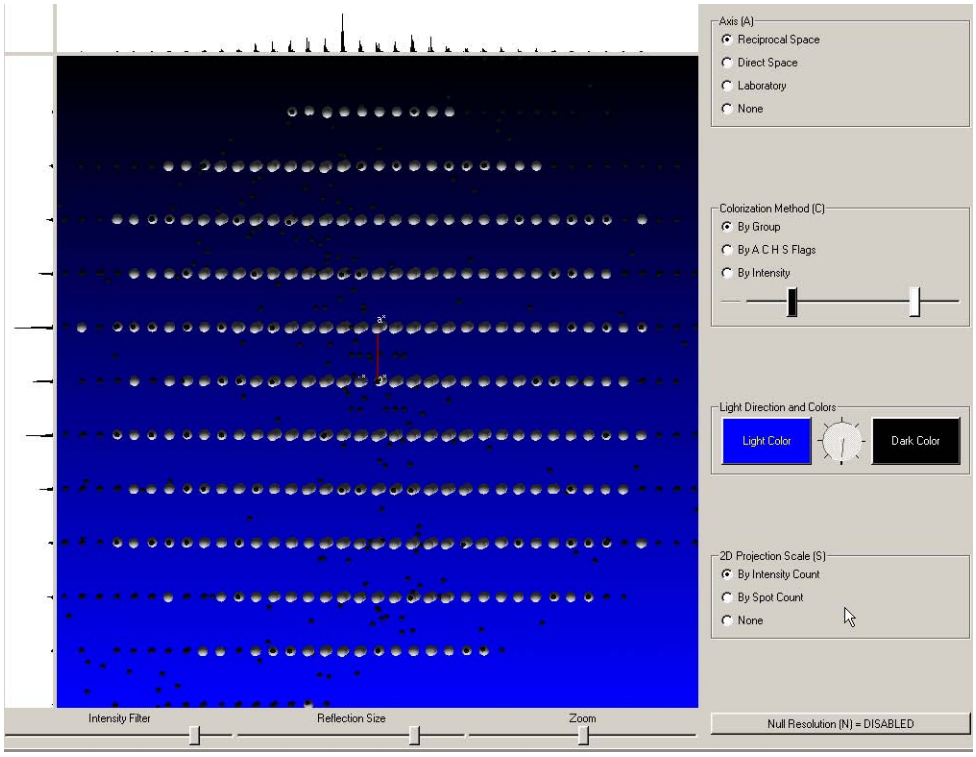


Figure A.10 – An oriented lattice view. The Visualization menu is to the right. The 2D profiles are to the left and to the top.

The distance between layers can be measured by clicking on one of the clusters of lines in the 2D view and then dragging to the next cluster. If you drag over three clusters, then the distance as measured would need to be multiplied by three.

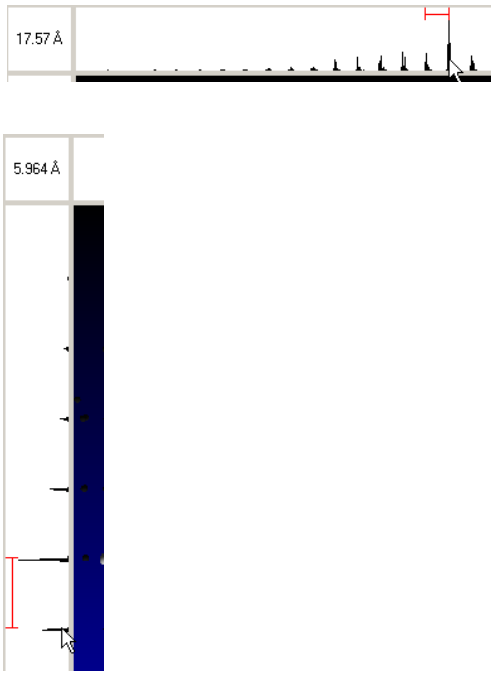


Figure A.11 – RLATT display with measured distances in the 2D views.

The Orientation menu allows easy orientation if the cell is indexed. Press the [F1], [F2], or [F3] key or click the appropriate button.

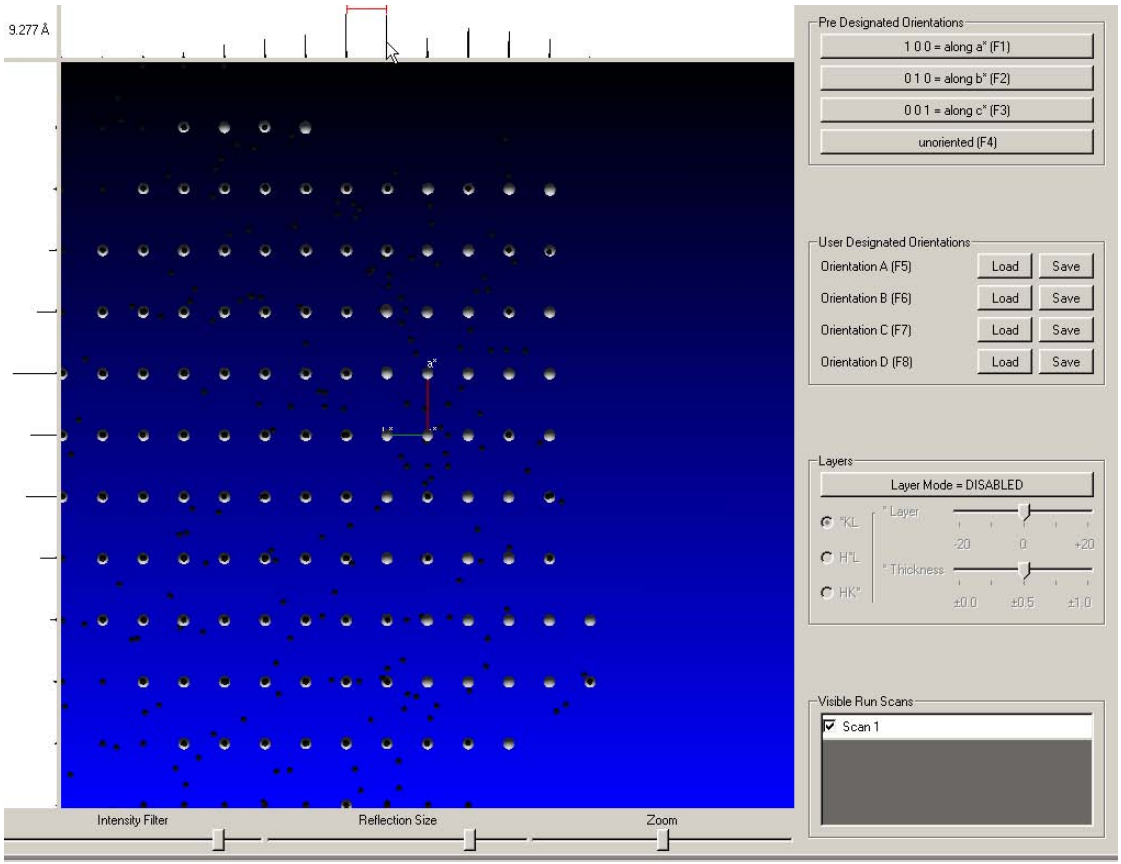


Figure A.12 – An aligned image with the Orientation menu to the left

3. Select reflections with the Lattice tool.

The Lattice tool is one of the most useful editing tools. Activate it from the Edit menu or by clicking on the background and choosing **Select > Lattice**. When initially activated, nothing appears. Hold down the [Alt] key to lock on a centroid and then left-click and hold on a spot. Now drag the mouse and line up the line that is tied to your spot on a row. Choose a longer row and align carefully. Now, while still holding the left mouse button, drag the second line to another row as shown in Figure A.13. Let go of the button. If you want to start over, click on another spot and repeat the process. Using the [Alt] key is optional, but it makes alignment of the lines easier and more accurate (Linux users may find that the [Alt] key has operating system functions).

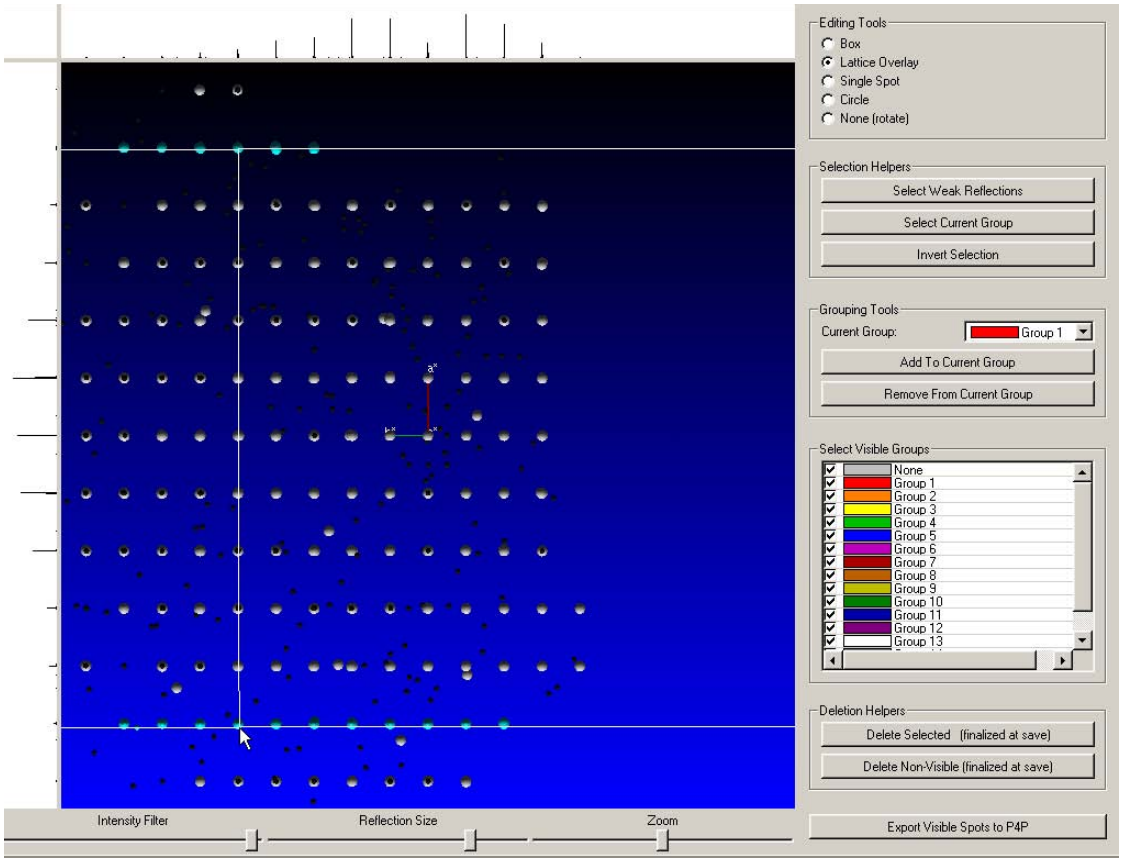


Figure A.13 – Two lattice lines selected

Press the [+] key to add lines between the two you have marked. Press the [-] key to remove lines.

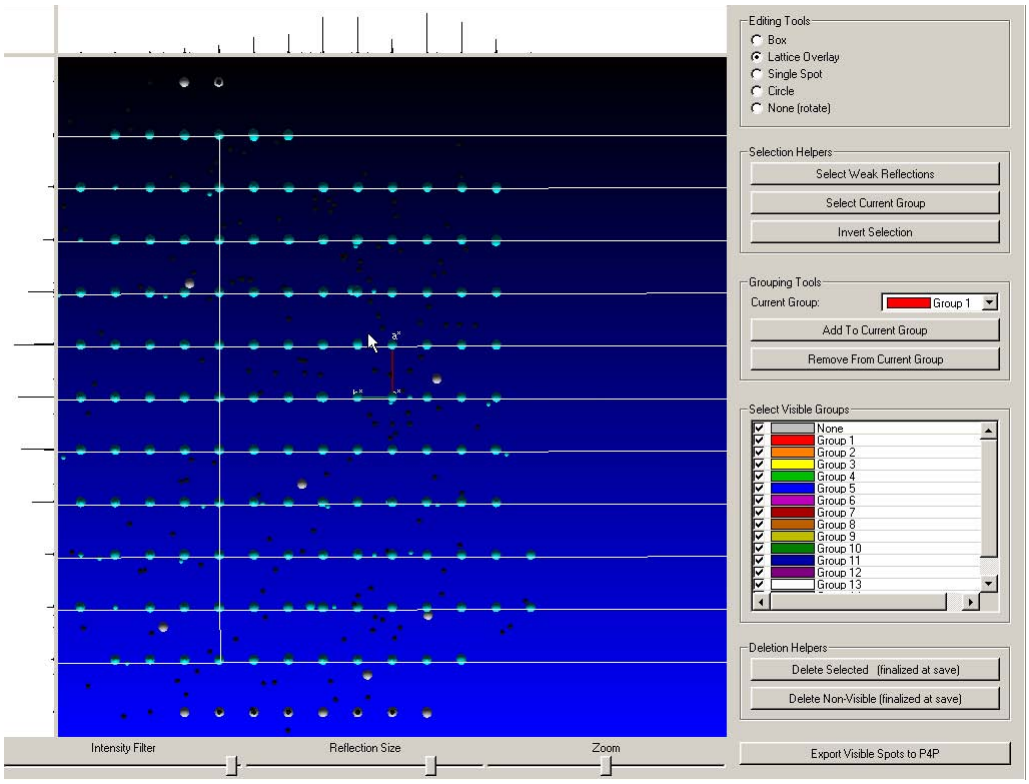


Figure A.14 – View with most of the lattice lines selected.
Lines to the top and bottom are not selected.

Use the [Page Up] key to add lines to the outside of the previously selected lines (in this example, the top and bottom). [Page Down] removes lines from the outside. Now all visible lines are selected. Note that the selected reflections are turquoise.

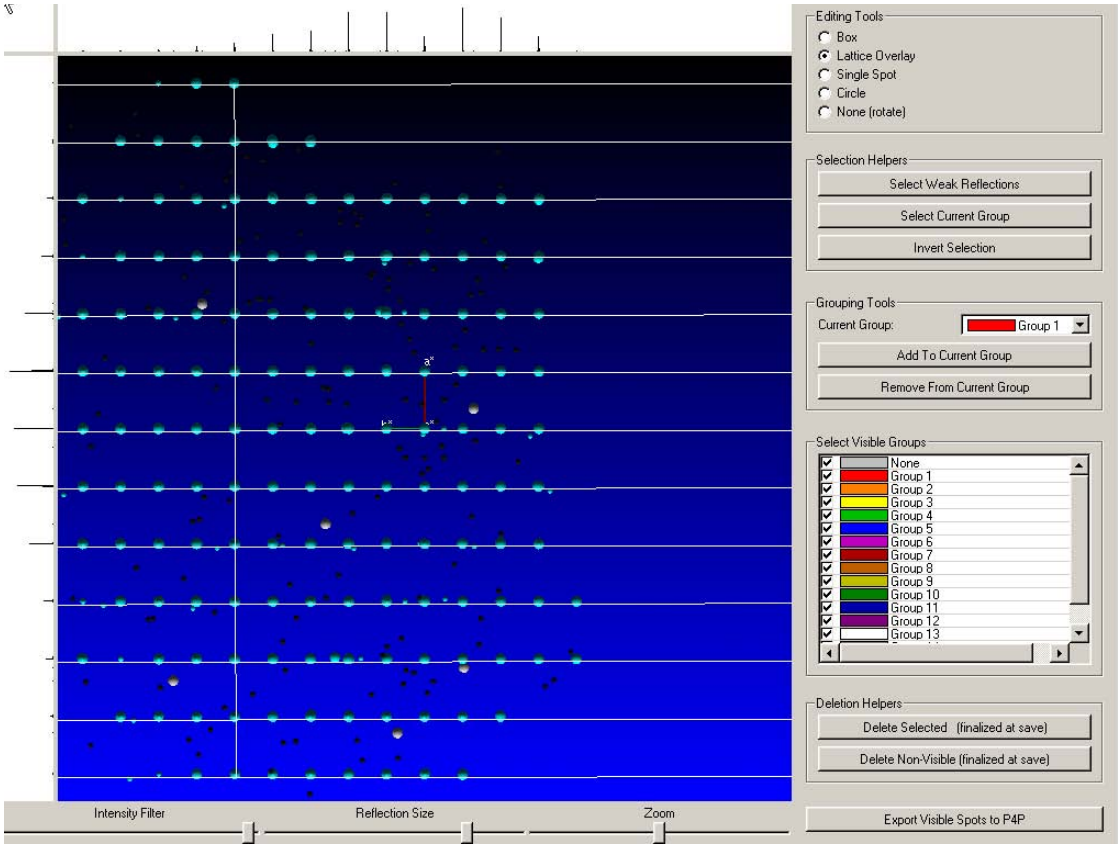


Figure A.15 – A view with all layers selected

Click **Invert Selection** (in the “Editing Menu” (**RLATT > Edit**) under “Selection Helpers”). All of the spots that do not touch the layer lines are now selected.

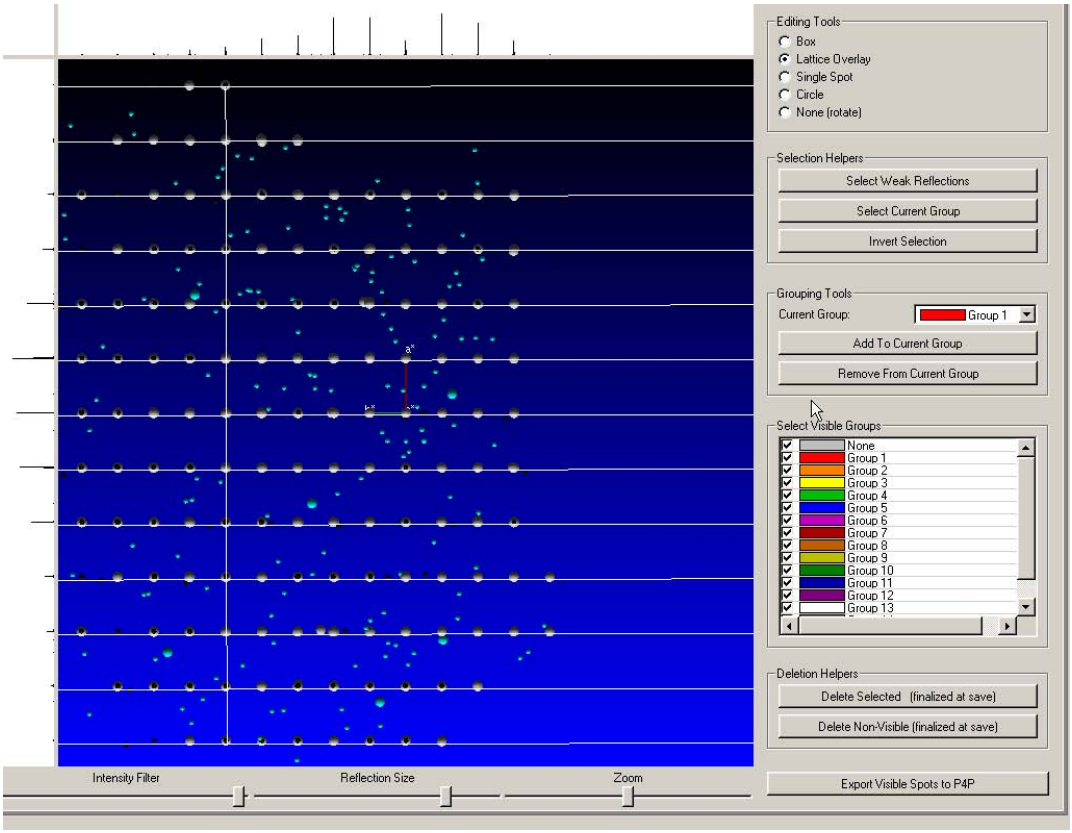


Figure A.16 – The selected spots are now the little ones between the layers. The spots in the layers are grey and the selected spots are turquoise.

Click **Delete Selected (finalized at save)** to mark the selected reflections to be deleted (and turned black). Alternatively, these selected reflections can be added to a group and then hidden using “Select Visible Groups.”

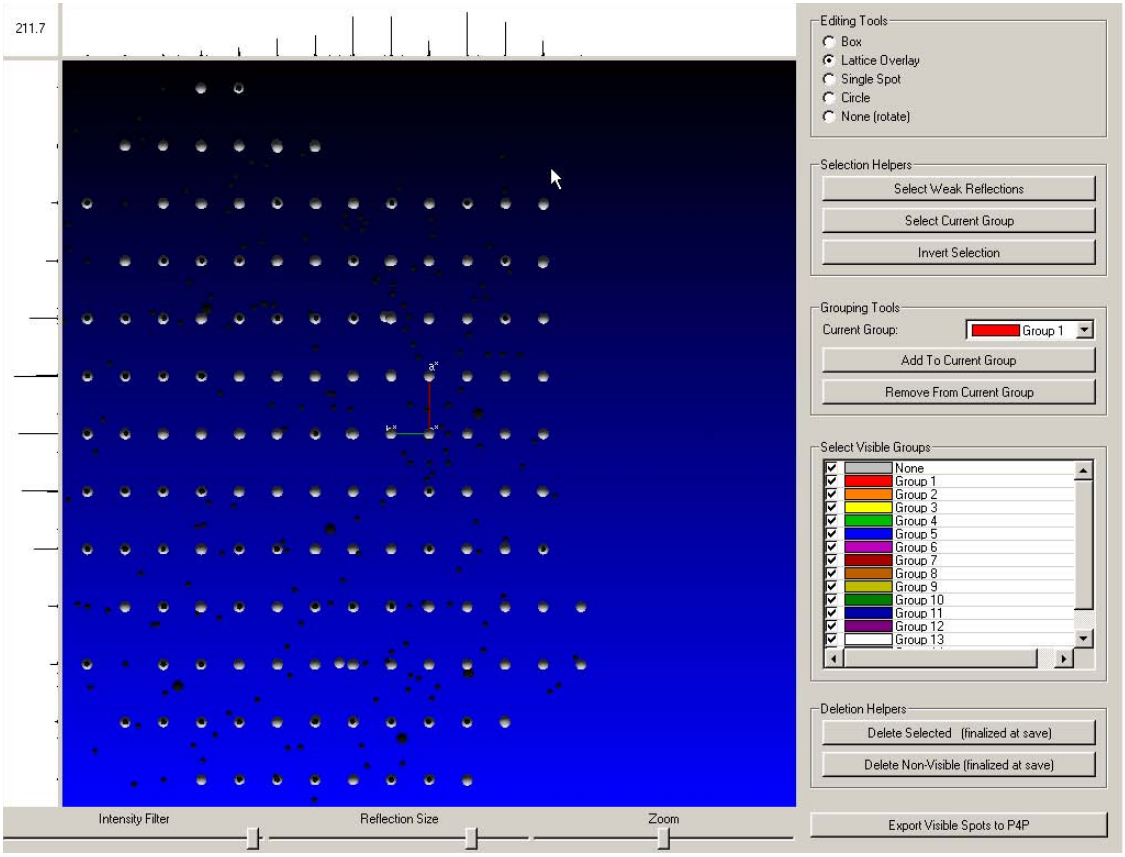


Figure A.17 – Selected reflections are blacked out

Save the file by clicking the disk icon (Figure A.18) or use **File > Save** to remove the reflection from the reflection list. Hiding a group with “Select Visible Groups” does not remove it from the reflection list. Either way, the resulting RLATT image is much cleaner and easier to interpret (Figure A.19).

Figure A.18 – Save File icon

Enable the Unit Cell tool to put a colored box in the view. This allows you to see if reflections are actually falling on the corners of the box (see Figure A.19 and Figure A.20). There are multiple options in the Unit Cell tool. “Select a Visible Plane” displays individual planes. “Select Plane Size” determines the boundaries of the planes and the number of unit cells displayed. Grid Planes mode displays planes as grids with spacings determined by the lattice.

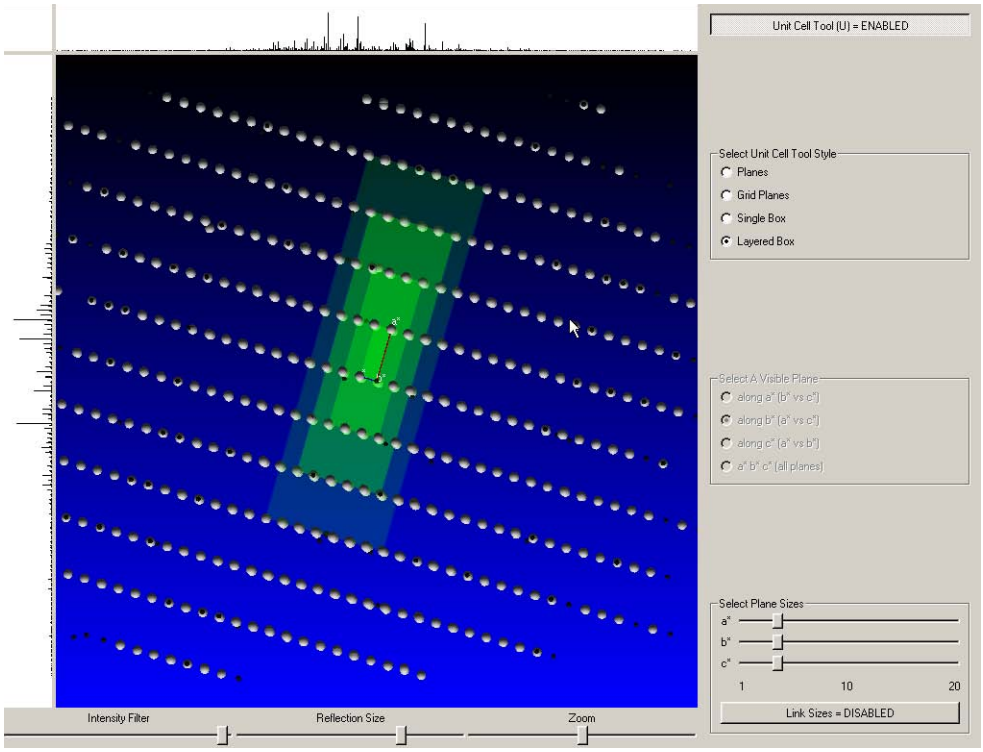


Figure A.19 – A view of the reciprocal lattice after the non-fitting reflections were deleted and with the Unit Cell tool enabled.

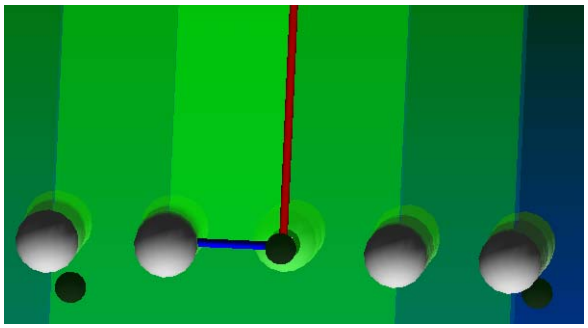


Figure A.20 – A view that zooms in on the Unit Cell tool. Almost all spots lie on the lines and planes defined by the unit cell. To the left and right are two weaker reflections (black dots) that do not fit.

A.3 Defining Groups

Selected reflections can be grouped. This tool is most useful for examining twinned, split, ingrown and otherwise problematic crystals. For Figure A.21, alternate rows were selected with the Lattice Selection tool. In the Grouping Tools section of the tool boxes to the right, the current group was set to Group 1 (red). Click “Add to Current Group” to turn these rows red. Then, the other rows and Group 13 (white) were selected and added. The box tool was used to select the reflection to the top left and these were put in Group 3, the yellow group. Finally, some of the reflections to the top and bottom were selected and added to Group 5, the blue group. This example of using the color groups generates a flag-type display. For twinned crystals, etc., the groups would be used to denote different components (see Figure A.22).

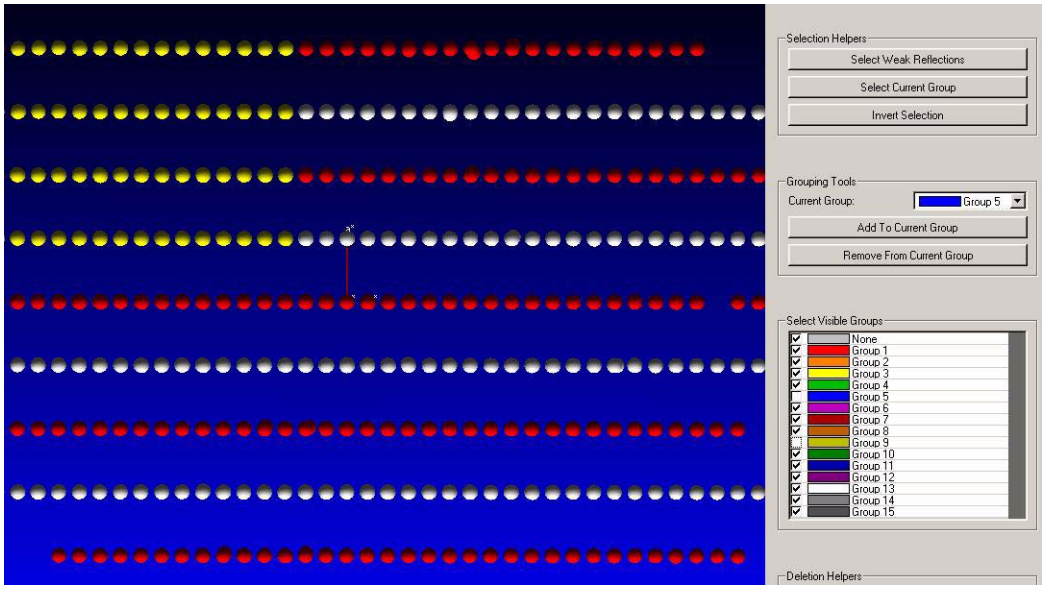


Figure A.21 – Reflections selected for different groups

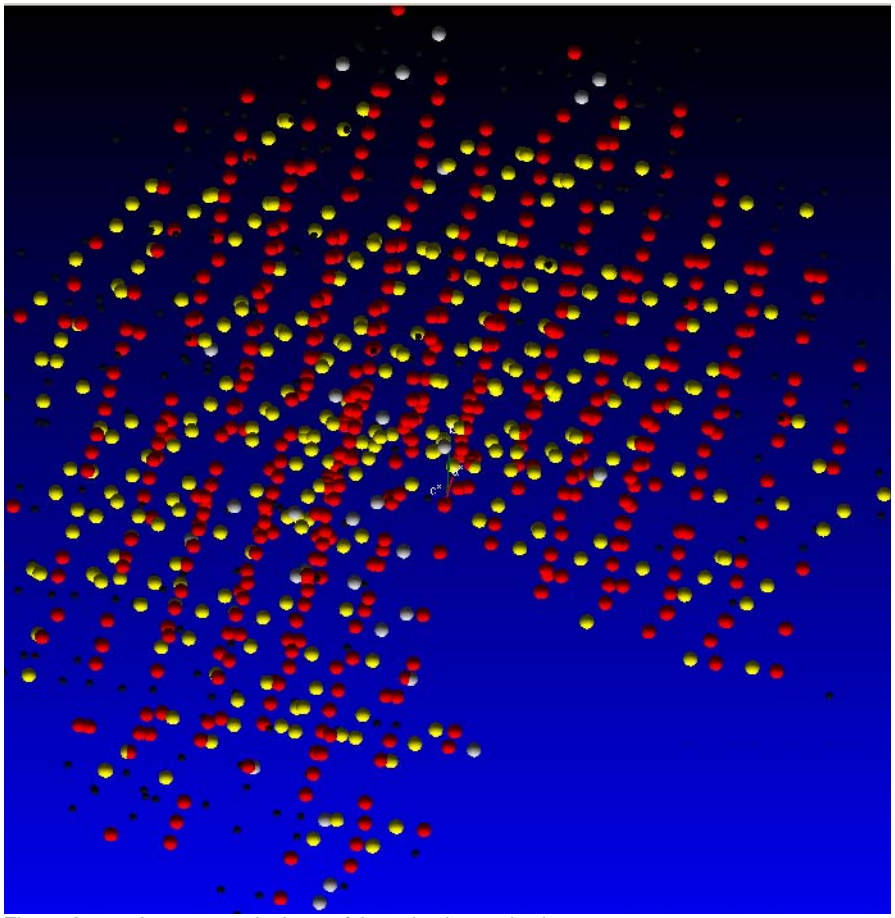


Figure A.22 – A more practical use of the selection and color groupings in RLATT. The two components of a rotational twin are colored yellow and red.

A.4 Measuring Distances and Angles

Right-click on the background and choose **Measure** to get tools for measuring distances and angles.

The Measure Distance tool gives two lines, much like the lines in the Lattice Selection tool, that can be oriented and dragged to get lattice spacings (see Figure A.23). Use the [Alt] key to lock on spot centers for easier orientation and more accurate measuring.

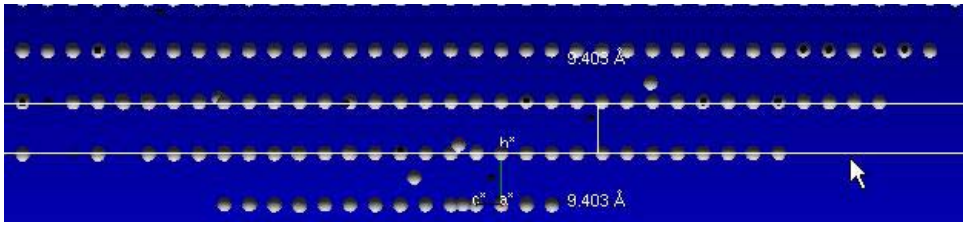


Figure A.23 – Using the Measure Distance tool

With the Measure Angle tool, left-click, hold, and drag a line on a lattice layer ending on the spot that will become the vertex of the angle. When the mouse is released, that point becomes the pivot point for a new line. Moving the mouse with no buttons depressed gives angle measurements (see Figure A.24). As described, the angle measurements will be done in 2D mode (i.e., the angle between the two lines displayed). To measure an angle in 3D mode, hold the [Alt] key while selecting spots as above. The tool will then lock on to spot centroids and the angle will be calculated using spot coordinates. This eliminates errors that might result from measuring using the 2D projection of the current orientation.

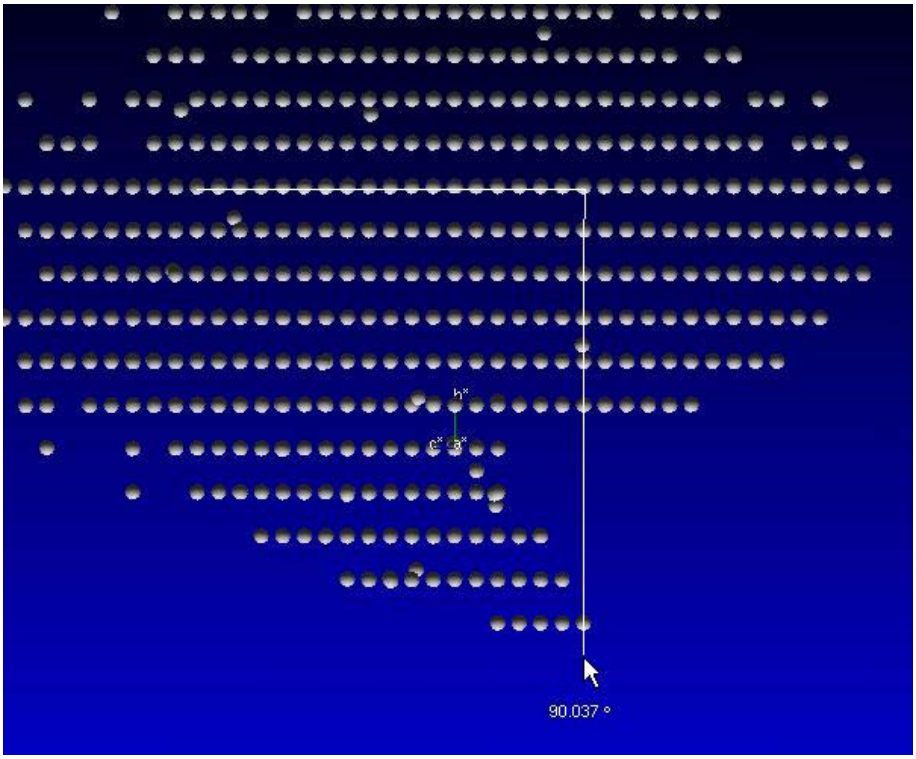


Figure A.24 – Using the Measure Angle tool

A.5 Writing a .p4p File

At the bottom of the Edit menu, there is a button for exporting all **visible** reflections to a .p4p file. The .p4p file is a text file of crystal, instrument, and reflection information. If the cell and crystal orientation is known, that information is included also (otherwise, dummy values are included). The sequence of cleaning up a set of reflections using RLATT and then exporting the results for input to CELL_NOW is a useful tool for dealing with hard-to-index crystals.

Appendix B Face Indexing

In order to correct intensity data for absorption numerically, the crystal's faces must be indexed. This allows a very exact crystallographic description of the crystal's shape.

Once you have a unit cell for your crystal, the Face Indexing module allows you to define faces directly over images of the crystal. As you define more and more faces, APEX2 automatically determines the dimensions of the crystal.

The examples in this Appendix use a large crystal for which the unit cell has already been determined.

B.1 Start the Crystal Faces Module

1. From the **Scale** category in APEX2's Task Bar, select **Crystal Faces**.

B.2 Collect a Video Zip Stream

To index the faces, first collect a *video zip stream*, which is a series of images taken with the video microscope as the crystal is rotated through 360° about the phi axis.

NOTE: For the best results, make sure that lighting conditions are optimal for the crystal you are viewing. If possible, try to illuminate the crystal for optimum contrast between the crystal and the background. Also, it may desirable to use multiple light sources to illuminate several faces simultaneously.

1. If no video zip stream has been collected for this crystal, a dialog appears prompting you to acquire a new video zip stream or load an existing one.

Figure B.1 – Video Source prompt

2. Because we have not yet taken a video zip stream for this crystal, click **Acquire New**. A save dialog box (defaulted to your current project directory) appears.

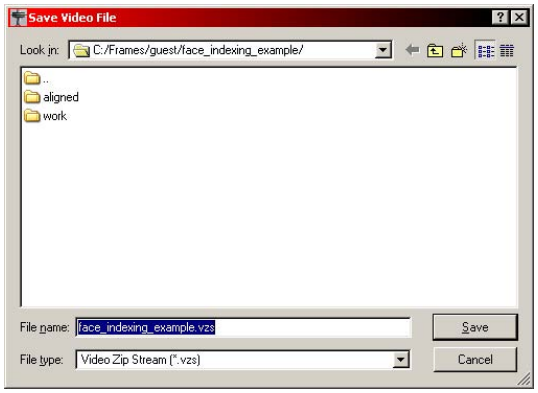


Figure B.2 – Save Video File dialog

3. Click **Save**. If VIDEO is not open, it will open with a progress bar showing the video zip stream collection. When the video zip stream is fully collected, the Face Indexing initial view will appear (Figure B.4).

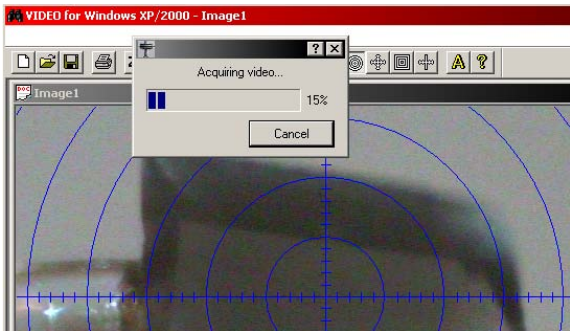


Figure B.3 – Progress bar

B.3 Using the Face Indexing Module

The Face Indexing module uses a dial at the bottom of the screen to step through the images of the crystal, and a tool called the T-tool to define faces. The Face List, shown on the right, displays the faces you have defined for the crystal and their distance from the instrument center.

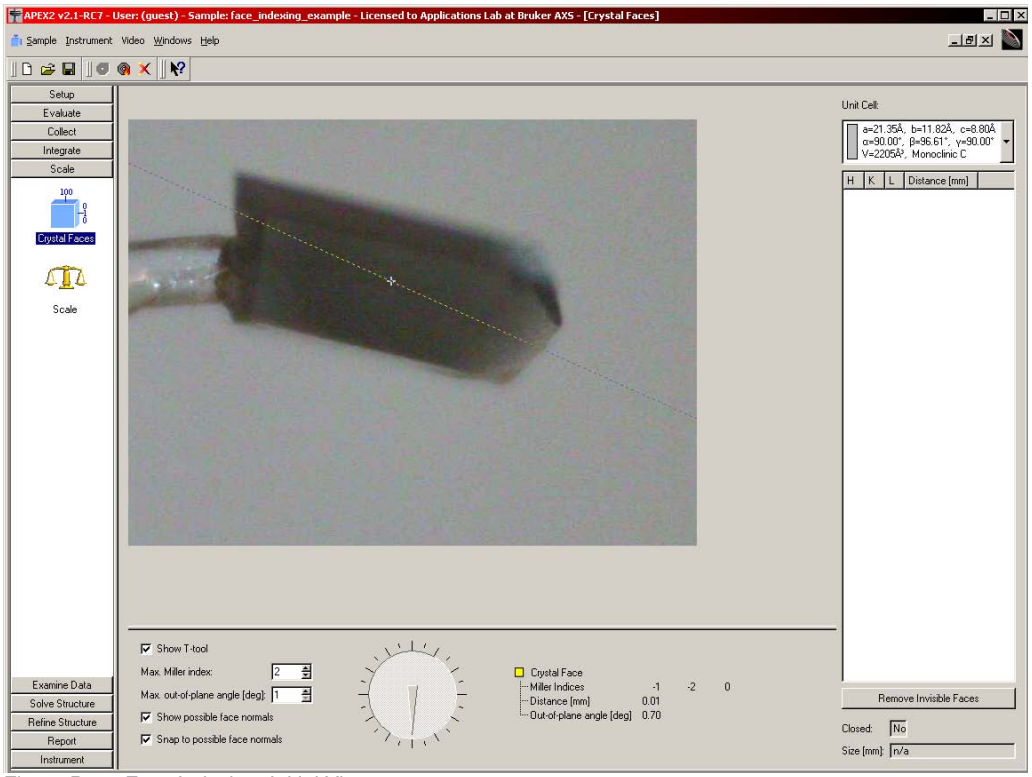


Figure B.4 – Face Indexing: Initial View

Item	Description
Show T-tool	Show and hide the T-tool for a better view of the crystal faces.
Max. Miller index:	The highest Miller index shown as face normals in the overlay.
Max. out-of-plane angle (deg):	When the T-tool snaps to a face normal, the plane you define may differ from the specified index by a maximum of this amount.
Show possible face normals	APEX2 suggests face normals.
Snap to possible face normals	The T-tool snaps to the suggested face normals.
Dial (or mouse wheel)	Use the dial to rotate around 360° of phi. The right-click menu lets you rotate phi by 180° to define parallel faces.
Crystal face information	Shows the Miller indices of a given face, the face's distance from the instrument center, and the out-of-plane angle.

Table B.1 – Face indexing controls

Item	Description
Unit Cell	Displays the unit cell that APEX2 uses to display faces in the overlay.
H,K,L	Miller indices of the normal to the specified face.
Distance (mm)	The specified face's distance to the center of the instrument.
Remove Invisible Faces	Faces obscured by the body of the crystal are removed from the Face list.
Closed:	Displays whether the faces you have defined encapsulate a closed volume.
Size (mm):	Dimensions of the crystal, calculated according to the defined faces.

Table B.2 – Face list

B.3.1 Set View Options

Depending on the color and reflectivity of the crystal and background, you may want to adjust the display colors.

1. Right-click in the image display area and select **Configure Overlay** from the menu. The Overlay Configuration menu appears (Figure B.5).

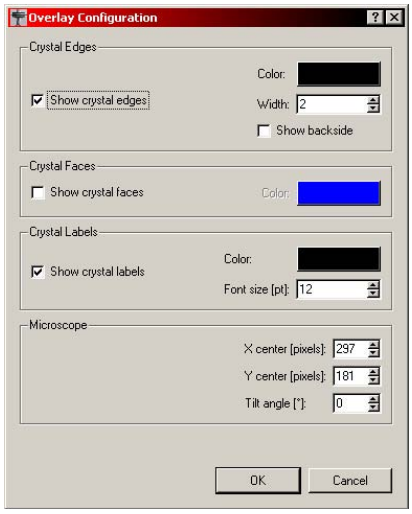


Figure B.5 – Overlay Configuration menu

B.3.2 Specify Crystal Faces

1. Use the dial control at the bottom of the screen (or use the mouse wheel) to rotate the crystal until one of the faces is parallel to the microscope axis as in Figure B.6. Note that, because **Show T-Tool** and **Show possible face normals** are selected, APEX2 suggests possible faces (whose indices are within the **Max. Miller Index** parameter) with dotted lines.

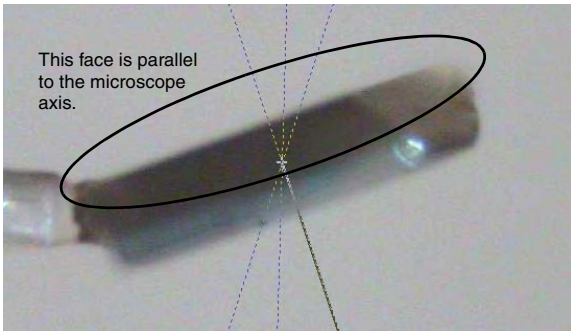


Figure B.6 – Face parallel to microscope axis

2. Move the mouse cursor to the dotted line that is perpendicular to the face. The mouse cursor snaps to the possible face normal.
3. A line appears to show the possible face. This line defines a plane along the microscope axis, which should be parallel to the crystal face. Use the mouse to move the line until it touches the crystal face.
4. Click to fix the face. An entry appears in the Face List showing the Miller Indices of the crystal face, along with the face's distance from the instrument center.

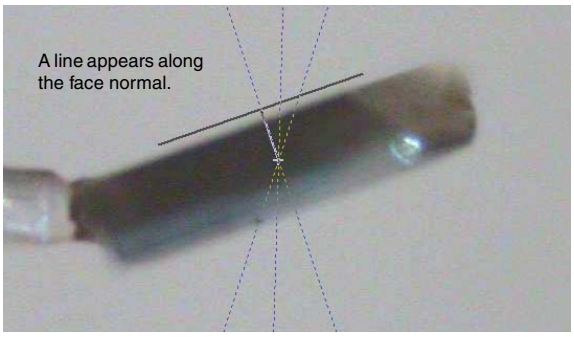


Figure B.7 – Face normals

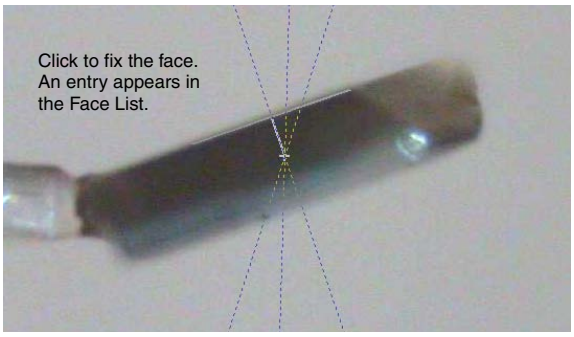


Figure B.8 – Fix the face

5. Repeat all steps in this section to specify all of the crystal's visible faces. If APEX2 does not suggest a face normal for a certain face, you can increase the value in the **Max. Miller index** field to show more suggested normals.
6. As you define more faces, APEX2 will begin to display its geometric model of the crystal, superimposed over the video images (Figure B.9). Continue until all faces are defined.

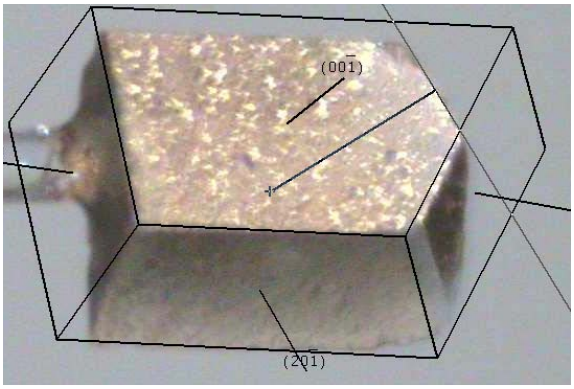


Figure B.9 – Some, but not all, faces defined

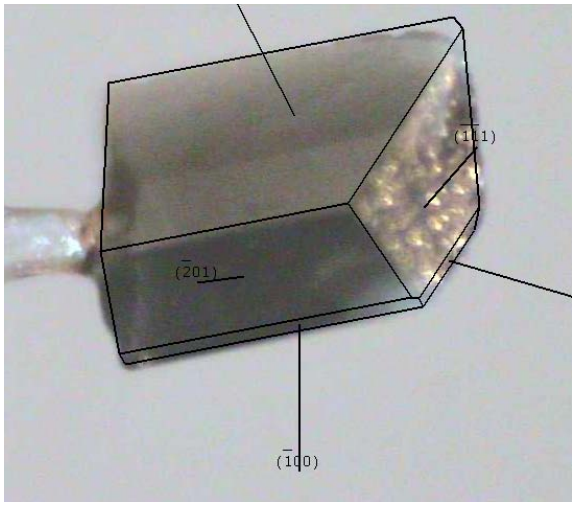


Figure B.10 – All faces defined

B.3.3 Editing the Face List

Consider Figure B.11. The face (1 3 -2) does not appear to define a face correctly. Removing this face will give a more accurate model of the crystal.

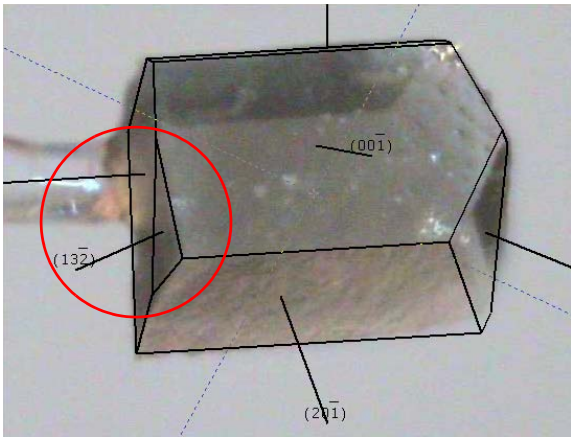


Figure B.11 – Face (1 3 -2) is incorrect

1. In the Face List, select the HKL (1 3 -2).
2. Right-click and select **Remove**. The face is removed from the list and the model is improved (Figures B.13 and B.13).

NOTE: Alternatively, you can select **Clear List** to start over, or **Add** and **Edit** to define your own faces and see where they appear on the crystal.

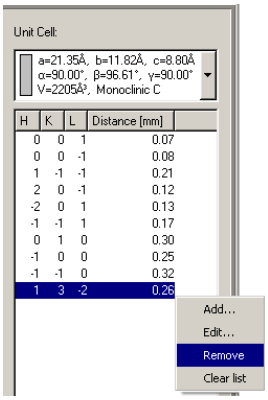


Figure B.12 – Face List right-click menu

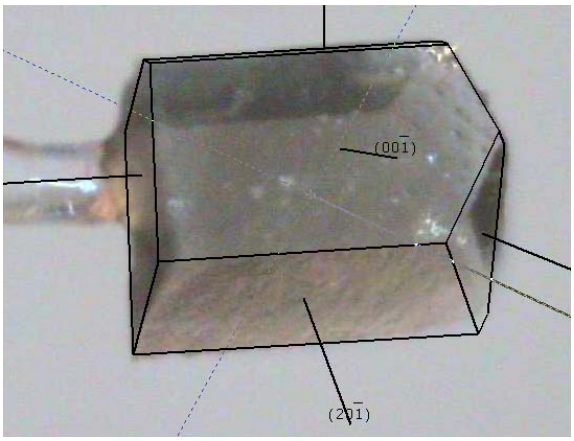


Figure B.13 – Face (1 3 -2) removed

B.4 Scaling With Face Indices

1. When all faces are defined, APEX2 has accurate crystal dimensions (as shown in the bottom right-hand corner). At this point, you can exit the Face Indexing module and proceed with scaling.
2. In the Scaling module, be sure to select **Numerical Absorption Correction (From Face Indices)** under the Setup tab (see Section 7.3.1).

Appendix C Using CELL_NOW

CELL_NOW is an extremely powerful tool for determining unit cells for difficult crystals. It analyzes a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other problems.

In initial search mode, the program tries to find sets of reciprocal lattice planes that pass close to as many reflections as possible. The corresponding real space vectors are sorted on a figure of merit (1.0 being a perfect fit). After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP.

Once a cell is found, it may be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

If CELL_NOW fails to suggest a sensible cell, either something is seriously wrong with

the reflection list (e.g., a wrong detector distance) or a cell axis is longer than the given search range.

C.1 Running CELL_NOW

CELL_NOW must be run from the command line. It requires a list of reflections exported from the APEX software (a .p4p file).

1. Open a Command Prompt window.
2. Change directories to the folder containing the exported reflection list.
3. For Windows or Linux, enter the command:

```
cell_now
```

After the command is entered, the terminal will print out an explanation of the program. Read this as it has useful information about what the program is doing:

CELL_NOW analyses a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other junk. In initial search mode the program tries to find sets of reciprocal lattice planes that pass close to as many reflections as possible. The corresponding real space vectors are sorted on a figure of merit (1.0 would be a perfect fit). In the output these are followed by the percentages of reflections that fit within 0.1, 0.2 and 0.3 times the interplanar separation, the components a1, a2 and a3 of the vector, the angles to previous vectors and a cross figure of merit to previous vectors. The latter should be larger for reflections belonging to the same twin component. Cosines of angles between vectors a and b can also be calculated from the components by (a1*b1+a2*b2+a3*b3)/(a*b).

After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP (without an .hkl file) taking the lattice type found with CELL_NOW into account. If necessary this conventional cell may be reinput in 'specified cell' search mode to find the orientation matrix. If CELL_NOW fails to suggest a sensible cell, either something is seriously wrong with the reflection list (e.g. a wrong detector distance) or a cell axis is longer than the given search range.

In specified cell search mode the program tries to find the best cell within the specified ranges. The reflections that fit this cell within a specified fraction of all three interplanar spacings may be flagged as indexed, and a new .p4p or .spin file written in which they have the 'H' flag so that they can be displayed in a different color with RLATT. Then the cell may be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

** WARNING: the exhaustive search employed in this program is VERY SLOW **
** so a CPU clock frequency of AT LEAST 3GHz is strongly recommended   **

4. CELL_NOW requests the name of the input file and suggests a name for the output file. Enter the name of the input file (For the output file, press [Enter] to accept the default).

```
Full name of .p4p, .spin or .drx file to read: example.p4p
   999 reflections read in

Listing file [example._cn]:

   514 reflections read in
Listing file [example._cn]:
```

5. Press [Enter] to start a general search.

```
Initial search (<Enter>) or specified cell search (S):
```

6. If the default superlattice threshold is suitable, press [Enter].

```
Superlattice threshold: an axis will be rejected if less than this percentage
of reflections has indices not equal to 2n or 3n resp. [10]:
```

7. If the default minimum and maximum distances are acceptable, press [Enter] again.

```
Minimum and maximum allowed values for cell edge [5 40]:
```

NOTE: Usually CELL_NOW is used to search generally for a cell, but if the cell is known, choose the specified cell option and enter the cell dimensions. CELL_NOW will then search for a matching cell.

After some period of time, a list of real space vectors sorted on a figure of merit (1.0 being a perfect fit) will be output. These are followed by the percentages of reflections that fit within 0.1, 0.2 and 0.3 times the interplanar separation, the components a1, a2 and a3 of the vector, the angles to previous vectors and a cross figure of merit to previous vectors. The latter should be larger for reflections belonging to the same twin component. Cosines of angles between vectors a and b can also be calculated from the components by (a1*b1+a2*b2+a3*b3)/(a*b).

```
Length, fom, #hits, %(0.1), %(0.2), %(0.3), components
 Followed by angle/correlation to previous vectors:

   12.692  0.902    30    94.493  99.999  99.999   -0.475    5.493  -11.432

   11.016  0.891    20    99.098  99.698  99.899    0.491   -9.912   -4.782
  90.0
 0.798

   16.802  0.836    24    93.793  99.398  99.698   -0.011    4.417   16.211
 139.0 131.0
 0.754 0.755

   16.805  0.819    25    90.189  99.498  99.899    0.953  -15.402    6.654
 139.1  49.1  81.9
 0.749 0.739 0.688

   27.693  0.741    19    80.379  93.692  98.898   -0.446    1.111  -27.668
  23.4  66.6 162.4 115.7
 0.675 0.657 0.638 0.616

   27.701  0.736    43    78.477  92.091  97.296    1.396  -20.915   18.109
 156.6  66.6  64.4  17.5 133.2
 0.687 0.659 0.620 0.645 0.569

   25.428  0.722    45    77.376  98.497  99.598   -0.496   14.332   20.999
 119.9 150.1  19.1 101.0 143.3  83.5
 0.651 0.674 0.638 0.600 0.557 0.533

   18.381  0.688    35    72.271  86.285  91.791  -15.128    9.256    4.827
  89.3 127.1  67.3 113.8 103.2 104.5  58.9
 0.596 0.605 0.556 0.544 0.480 0.484 0.487

    6.329  0.686     4    65.465  65.465  65.465    0.248   -2.729    5.705
 179.9  90.1  40.9  41.0 156.7  23.5  60.0  90.7
 0.574 0.573 0.532 0.524 0.459 0.469 0.465 0.426

   14.648  0.684    18    73.873  85.484  91.090  -14.633   -0.652    0.025
  89.1  90.3  90.5  90.9  89.3  90.9  90.2  36.8  91.1
 0.591 0.603 0.556 0.539 0.475 0.477 0.490 0.539 0.424
.
.
.
-------------------------------------------------------------------------------
```

After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP (without an .hkl file) taking the lattice type found with CELL_NOW into account. If necessary, this conventional cell may then be input in 'specified cell' search mode to find the orientation matrix.

```
The following cells would appear to be plausible, but should be checked using
XPREP because they are not necessarily the conventional cells.

FOM, % within 0.2, a..gamma, volume and lattice type for potential unit-cells:

  1 1.000  85.3   12.692  11.016  14.648   89.70   90.95   90.00    2047.7  P

--------------------------------------------------------------------------------

Cell for domain  1:   12.692   11.016   14.648    89.70    90.95    90.00

Figure of merit: 0.619   %(0.1):  68.3   %(0.2):  85.3   %(0.3):  91.0
```

CELL_NOW has identified a domain that fits 85.3% of the data with a tolerance of 0.2.

The output shows a suitable cell with its orientation matrix, figure of merit and the deviations of the input reflections from integer indices. This deviation is used to determine which reflections fit this cell and which are left for further searches for new domains.

```
Orientation matrix:  0.00164338 -0.00357404 -0.06816319
                    -0.03417112  0.08170117 -0.00385047
                     0.07098872  0.03940338  0.00098036

Maximum deviation from integer index [0.25]:
```

8. Enter the maximum deviation from integer index cutoff (Typically the default is reasonable). Press [Enter].

```
Percentages of reflections in this domain not consistent with lattice types:
A: 50.1,  B: 49.9,  C: 45.0,  I: 48.9,  F: 72.5,  O: 64.3  and  R: 65.8%
```

Lattice information (given as % of relevant reflections that do not fit a particular lattice) should be used in XPREP to determine the conventional cell. Typically, for a lattice to be present the % inconsistent should be less than 10, but problem crystals can make the value higher.

```
Percentages of reflections in this domain that do not have:
h=2n: 36.3,  k=2n: 49.8,  l=2n: 49.3,  h=3n: 69.1,  k=3n: 67.2,  l=3n: 66.4%
```

This percentage information is useful for identifying supercell problems. Typically, the percentages should be 30% or higher for the 2n values and 50% or higher for the 3n values, but problem crystals or crystals with heavy atoms in special positions can make the values lower. Low values for a particular class of reflections can suggest that an axis is double (2n) or triple (3n) its correct length.

```
New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]: A
```

9. Enter A or just press [Enter] to accept this cell. The program asks for a name for a .p4p file. Enter the name of the .p4p file for this solution (in this example, domain1.p4p).

```
.p4p or .spin file to write domain to: domain1.p4p

 RLATT color-coding employed in file: domain1.p4p
 White: indexed for first domain
 Red: not yet indexed

   877 reflections within 0.250 of an integer index assigned to domain  1,
   877 of them exclusively;   122 reflections not yet assigned to a domain
```

122 reflections, approximately 1/10 of the data, have not yet been indexed (assigned to a domain). This is more than might be expected for random noise peaks. A search for an additional domain makes sense. The domain 1 cell will be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

```
Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
new cell from list (enter number) [S]: S
```

10. Enter S to search for another orientation of the newly chosen cell.

```
 Cell for domain  2:   12.692   11.016   14.648    89.70    90.95    90.00

 Figure of merit: 0.643   %(0.1):  69.7   %(0.2):  97.5   %(0.3):  98.4

 Orientation matrix:  0.00598966  0.00402327  0.06807401
                     -0.03367313 -0.08180340  0.00480114
                      0.07099302 -0.03914713 -0.00221035

 Rotated from first domain by 179.7 degrees about
 reciprocal axis  1.000  0.001 -0.032  and real axis  1.000  0.002 -0.010

 Twin law to convert hkl from first to      0.999   0.004  -0.019
 this domain (SHELXL TWIN matrix):          0.003  -1.000  -0.004
                                           -0.064   0.006  -0.999
```

CELL_NOW has identified an additional domain that fits 97.5% of the data with a tolerance of 0.2. The relationship between this domain and the first domain and the SHELXL TWIN matrix are reported. This example is for a rotational twin about the a* axis.

The output shows a suitable cell with its orientation matrix, figure of merit and the deviations of the input reflections from integer indices. This deviation is used to determine which reflections fit this cell and which are left for further searches for new domains.

11. Enter the maximum deviation from integer index cutoff (typically the default is reasonable).

```
Maximum deviation from integer index [0.25]:
```

12. Enter the name of the .p4p file for this solution (in this example domain2.p4p).

```
.p4p or .spin file to write domain to: domain2.p4p

 RLATT color-coding employed in file: domain2.p4p
 White: indexed for first domain
 Green: current domain (but not in a previous domain)
 Red: not yet indexed

   691 reflections within 0.250 of an integer index assigned to domain  2,
   119 of them exclusively;     3 reflections not yet assigned to a domain

 Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
 new cell from list (enter number) [Q]
```

13. Enter <q> to exit CELL_NOW.

```
C:\struc\guest\twin>
```

NOTE: At this point CELL_NOW has indexed a two-component twin. The orientation matrices for the two components are in the domain2.p4p file discussed below.

C.2 CELL_NOW output

In this example, in addition to the terminal output described above and the example._cn listing file written to disk, CELL_NOW has written two .p4p files, domain1.p4p and domain2.p4p. The second of these is a multicomponent .p4p for a two-component twin. It is shown below.

The following entries appear twice: once for each domain.

```
CELL, ORT1, ORT2, ORT3, ZEROES, ADCOR
CELL2, ORT12, ORT22, ORT32, ZEROES2, ADCOR2
```

This will eventually trigger SAINT to treat this data set as a twin.

```
FILEID SAINT        V6.43A        4.00        06/19/03 09:36:24 mraa
SITEID RufM                                Ruf
TITLE  [RCpCrCl2.nTHF]n
CHEM   C40H58Cr2Cl4O2
CELL     12.6918   11.0161   14.6480   89.7011   90.9494   90.0010   2047.695
CELLSD    0.0025    0.0022    0.0029    0.0300    0.0300    0.0300     1.024
ORT1        0.001643376    -0.003574040    -0.068163186
ORT2       -0.034171116     0.081701167    -0.003850471
ORT3        0.070988722     0.039403379     0.000980361
ZEROS   0.0000000  0.3249754 -0.0041494    0.0000    0.0000    0.0000
ADCOR        2.9302     -0.0047      0.0003     -0.8959      0.0123      0.0937
CELL2    12.6918   11.0161   14.6480   89.7011   90.9494   90.0010   2047.695
CELLSD2   0.0025    0.0022    0.0029    0.0300    0.0300    0.0300     1.024
ORT12       0.005989663     0.004023272     0.068074010
ORT22      -0.033673126    -0.081803404     0.004801138
ORT32       0.070993021    -0.039147127    -0.002210349
ZEROS2  0.0000000  0.3249754 -0.0041494    0.0000    0.0000    0.0000
ADCOR2       2.9302     -0.0047      0.0003     -0.8959      0.0123      0.0937
SOURCE Mo     0.71073   0.70930   0.71359   2.00000   50.00   40.0
```

NOTE: SAINT+ users should be aware that APEX2 Version 2 can import .p4p files created with CELL_NOW and the Integrate Images module will handle the multiple orientation matrices correctly. There is no longer a need to use the standalone SAINT+ program.

Appendix D Configuring APEX2

The APEX2 Suite includes the ability to customize many features of the program. Most customizations should be left to the site administrator, but some introduction is appropriate. The example below is a color-coded example of an actual Python file. It gives an introduction to configuring the program.

The following is a collection of examples of possible changes to the configuration files for APEX2 and PROTEUM2. These files control the look and feel of the GUI.

D.1 Global, User, and Sample-specific Variables

Configuration variables the system administrator wants to set globally for all users go into a file called **bn-config.py** that resides in C:\ for Windows or /usr/local/lib for Linux.

Individual users can set their own preferences for most configuration variables in a file in their home directory. This file is named bn-config.py for Windows and .bn-config for Linux (Under Windows, at a command prompt type `echo %UserProfile%` to find out where the user version of bn-config.py should be located.)

Additionally, configuration variables can be set for a specific sample using a file in the sample directory. This file is bn-config for Windows and Linux.

These files are read in the order given above, so values set globally are replaced by individual values and global and individual values are replaced by sample-specific values.

D.2 Python Code Examples

In the following text,

- Red denotes comments that result from # comments.
- Green denotes comments between triple quotes (""").
- Black denotes actual Python code.

```
# Examples of configuration changes-
"""
```

A few notes about Python comments.
Three quotation marks in a row on a line (“““) start a comment section
The next three quotation marks (“““) end the section
The text you have just read is in a comment section
"""
#end of commenting out a section with triple "'s
Pound or Number starts a simple comment. This can be anywhere on a line.
#
#
The default color scheme for the image display:
#
colorramp='Colored_SCD.ICM' # This is the typical BAXS orange display
#
"""
The most frequently used other color choices are:

Black_On_White_SCD.ICM
White_On_Black_SCD.ICM
The_Blues_ANY.ICM

Other color ramps are in:

Windows: C:\bn\src\gui\intensitycolormaps
Linux: /usr/local/bn/src/gui/intensitycolormaps
"""
#
This is how user positions can be defined:
#
userpos1=
{'name':'Zero@dx60','type': 'n',
'dx': 60.0,
'theta': 0,
'omega': 0,
'chi': 0,

```
            'phi': +0.0 }
#
#If you want angles other than zero, the import projtls and del projtls must be used:
#
import projtls
userpos2=
   {'name':'Chi45@dx90','type': 'n',
             'dx': 90.0,
             'theta': -10.0*projtls.degrees,
             'omega': -5.0*projtls.degrees,
             'chi': 45.0*projtls.degrees,
             'phi': 180.0*projtls.degrees  }
del projtls
#
# Acceptable CCD temperature:
#
ccdtemperaturerange=(-65,-55)
#
# Auto connect to the instrument when needed:
#     (This is a protected variable that is controlled by the System Administrator)
#
autoconnect=1
#
# The following five values define matrix runs:
#
perpendicular_matrix_runs_2theta=30
# Set the 2-theta for the matrix measurement:
perpendicular_matrix_runs_distance=41
# Set the detector distance for the matrix measurement:
perpendicular_matrix_runs_nr_sweeps=3
# Do three sweeps or runs:
perpendicular_matrix_runs_sweep=6
# Each run will be 6 degrees long.
perpendicular_matrix_runs_frametime=None
```

```
# The frame time is left to be the default.
#
# Setup to get some debug messages for scans.  This is useful if resume is failing.
#
#debug=('goniometer.scan',)
#debug=('expt.findresolution',)
#
#
# Hardware profile used by the data collection strategy planner:
bishost='bruker-server'
hardwareprofile='Kappa-APEX-II-euler.hrd'
#bishost='smart-apex'
#hardwareprofile='SMART-APEX-II.hrd'
#
#
# IP address or DNS name of the file server used for storing images,
# followed by the name of a network shared folder on that server.
# Add additional path segments as comma-separated and quoted strings.
# To find out the DNS name:
# For Linux type:     hostname       at the command prompt
# For Windows type:      echo %computername%      at the DOS prompt.
#
fileserver=('x8-client','frames')

# Supported file name protocols. Currently these are 'smb' for
# files that are accessed over a MS Windows network and 'file' for
# local files.
universalfnprotocols=('smb','file')
#
```

```
Configuration of the APEX & PROTEUM programs
============================================

The appearance and working of this suite of programs
can be modified by a configuration file. The programs read the
a series of configuration files in following order.  As described below, a
specification in a later file
overwrites a setting in an earlier file.

On unix these files are:
 1 a file /usr/local/lib/bn-config.py
 2 a file .bn-config in your home directory (Please note
   the initial "." character, which renders this file invisible
   for the normal "ls" command).
 3 a file bn-config in your current directory.

On Windows these files are:
 1 bn-config.py in the "SystemDrive" folder
 2 bn-config.py in the "USERPROFILE" folder
 3 bn-config in the current folder

If none of these files is present (the situation when the program has
just been installed) all parameters are using built-in defaults.  To
change a single configuration parameter, creates or edits
one of the three files and put in a value for the parameter
to be customized.

You should put a parameter in (1) if it should be the system default for
all users; put it in (2) if it is your own default, and other people might
want to use other values; put it in (3) if it is only required for a single
project.

The configuration files are read as "Python" programs, and therefore
syntax is very important. The best way to use it is to only use
variable assignments or function calls, as described below. If required,
you can comment out lines from your configuration file by preceding them
with a '#' character.
```

```
Note:

 - Leading space is significant. Start all assignments in the first column.
 - Variable names are case sensitive.

Functionality groups
====================

Sometimes a complete group of options need to be changed to get
a specific functionality. For these cases there are function calls
that change a number of variables at once.

Due to the "default-like" nature of these function calls, they should
probably be used only in the site-wide configuration files, and even
there only at the beginning [but this is not enforced].

The available functions are:

default_protein()
  This switches from the default "small molecule" parameters to more
  protein-like parameters. At this time, the following parameters are
  set:
    chiralonly=1
    autochiralpointgroup=1
    resomode=1
    resolution=2.5
default_noprotein()
  This sets the defaults back to small molecule values
The following variables, in alphabetic order, can be set in a configuration file.

* anglemargin, type=floating point, default=1.0
   Do not change

* autochiralpointgroup, type=boolean, default=0
   This selects whether point groups are preferably chiral. This can be useful in a
    protein setting.
```

```
* autoconnect, type=boolean, default=0
   If this is set to 1, the connection to the BIS server will be created
   automatically without user-intervention whenever required. This variable
   can only be changed in the system-wide configuration file (see
    ”protectedvariables”)

* axcrit, type=floating point angle, default=0.2 degrees
   This is the criterion used by the automatic cell reduction algorithm to decide
    whether two axes coincide. Do not touch.

* badcolor, type=tuple of three integers, default=(255,190,190)
   Color in the GUI that is associated with ’bad’ situations

* busycolor, type=tuple of three integers, default=(240,240,96)
   Color in the GUI that is associated with ’busy’ situations

* bgdone, type=tuple of three integers, default=(96,240,96)
   Color in the GUI that is associated with ’done’ situations

* bgerror, type=tuple of three integers, default=(240,96,96)
   Color in the GUI that is associated with ’error’ situations

* bgimpossible, type=tuple of three integers, default=(240,240,96)
   Color in the GUI that is associated with ’impossible’ situations

* bgpossible, type=tuple of three integers, default=(96,240,96)
   Color in the GUI that is associated with ’possible’ situations

* bgtodo, type=tuple of three integers, default=(96,240,96)
   Color in the GUI that is associated with ’todo’ situations

* bgwarning, type=tuple of three integers, default=(240,240,96)
   Color in the GUI that is associated with ’warning’ situations

* bishost, type=string, default is not set
   The name or IP address of the BIS server computer
```

```
* ccdtemperaturerange, type=tuple of 2 integers, default=(-65,-35)
   Acceptable range (minimal,maximal) of temperatures in deg C for the detector.
    This is used only to indicate a red or a green status light for the CCD
    temperature in the status window.

* chiralonly, type=boolean, default=0
   If set to 1, only chiral point groups will be used at any moment. Do not touch.

* colorramp, type=file name, default='Colored_SCD.ICM'
   Default color ramp used by the image display tool

* communicationlog, type=file name, default='communication.log'
   File name used for logging all communication between application and BIS. If not
    set, no logfile will be kept.

* component1color, type=string (color name), default='green'
   The component#color variables are obsolete. Use groupcolors instead.

* component2color, type=string (color name), default='blue'
   The component#color variables are obsolete. Use groupcolors instead.

* component3color, type=string (color name), default='yellow'
   The component#color variables are obsolete. Use groupcolors instead.

* datcoldx, type=floating point, default=35.0
   Default sample-to-detector distance for data collections.

* dbtype, type=string, default='postgresql'
   Type of database engine used. Do not touch.

* dbhost, type=string, default='127.0.0.1'
   Name or IP address of the server running the database. The default is to
   run the database on the same host as the rest of the software, hence the
   default that refers to the local host.
```

```
* dbport, type=string, default='5432'
   IP port to use to address the database. Normally dependent on database
    installation.

* dbname, type=string, default='BAXSdb'
   Name of the database. Do not touch.

* debug, type=tuple of strings, default=()
   This is a list of all modules that are to be debugged. This should
   not be touched except if a developer asks you to add something here to
   help track a problem.

* defaulthardwaretype, type=integer, default=4
   If different hardware types are supported, this can be used to select which one
    should be used. 4=BIS. Do not touch.

* detectorsizemargin, type=floating point, default=0.1
   Do not touch.

* eventlogfile, type=file name, default='bn-event.log'
   File in which important events are logged during program runs.

* fontsize, type=integer, default=None
   GUI font size in points

* groupcolors, type=tuple of color names, default=('gray75',
          'red','green','yellow','blue','magenta','cyan','white',
          'gray25','brown','forest green',
          'khaki','midnight blue','violet','turquoise','gray50')

* guicolor, type=tuple of three integers making a color, default=0xd4,0xd0,0xc8
   Overall background color of user interface.

* guistyle, type=string, default='windows'
   The style of the GUI. Do not touch.
```

* hardwareprofile, type=file name, default=None
  The hardware profile to be used by COSMO for strategy calculations.

* hashfilepattern, type=string, default='_##_####'
  Do not touch

* headerdisplay, type=?, default=not set
  If present, this variable defines the name of a file containing the default image header items that should be displayed in the table underneath the image display.

* imageextension, type=string, default='.sfrm'
  Do not touch

* imagemask, type=int,default=0
  value of a mask used in viewing powder frames

* inhibitgc, type=boolean, default=0
  Inhibit garbage collection. Do not touch.

* localfilemap, type=map, default=None
  A map of network drives, translating local file names to equivalent network names. On Windows machines, this is set automatically. On unix, this should represent a copy of the relevant parts of the samba configuration.

* lowcurrent, type=floating point, default=5
  Tube current in mA to set when an experiment finishes.

* lowvoltage, type=floating point, default=20
  Tube voltage in kV to set when an experiment finishes.

* mailfrom, type=string, default=Not set
  The "From" address of E-mails sent by the software (e.g. bug reports).

* mailfromdomain, type=string, default=Not set
  The domain of the From address of E-mails sent by the software. This will only be used if ''mailfrom'' is not set. The user name will be pre-pended.

```
* matrixstrategy, type=list of scans, default=3 specific scans
   The measurement strategy that is to be used to find a unit cell.

* minimalpowerfraction, type=floating point, default=0.5
   The minimal generator power as fraction of the maximal power that will be
    considered as sufficient to make a measurement

* mosaicity, type=floating point angle, default=0.4
   Expected normal width of a reflection, in degrees.

* nominalpowerfraction, type=floating point, default=0.9
   The normal generator power as fraction of the maximal power that the generator
    should be set to before a measurement

* nominalvoltage, type=map, default={'MO':60,'CU':45,'AG':60}
   A definition of the nominal tube voltage in kV as a function of the
   target element.

* orientdx, type=floating point, default=-5
   The default crystal to detector distance for the "aligned images" module. If
    given as a negative number, it is counted from the back of the dx-sled.

* orientdegreetime, type=floating point, default=10.0
   Default scan time in seconds per degree for an aligned image.

* orientscanwidth, type=floating point, default=5.0
   Default scan angle in degrees for an aligned image.

* overlapcolor, type=color string, default='red'
   Color used for overlapping predicted spots

* overlaycolor, type=color string, default='green'
   Color used for predicted spots

* peakradiusfactor, type=floating point, default=2.2
   How many times the single-sigma-spot radius should be used as integration radius.
    Do not touch.
```

```
* planes, type=string, default='0kl h0l hk0'
   List of precession planes that is suggested as default planes for the
    calculations

* planethickness, type=floating point, default=0.1
   Default thickness of precession images

* precessionmargin, type=integer, default=1
   How many pixels at the outer edge of the detector that is to be ignored in
    precession calculations

* prefereulerian, type=boolean, default=1
   1 = prefer eulerian geometry over kappa geometry
   0 = prefer kappa geometry over eulerian geometry
   This should not be changed after installation.

* preferreddirection, type=integer, default=-1
   (default rotation direction of axes)

* productline, type=string, default='ANY'
   A selection of the features in the program suite based on the type of
   application we're running:
    'ANY' or 'ALL' = all products,
    'SCD' = single crystal,
    'XRD' = powder,
    'XRF' = fluorescence

* protectedvariables, type=map, default={'protectedvariables':1,'autoconnect':1}
   A selection of variable names and a limit to which configuration files can be
    used to change them. This can be used to make sure users do not overwrite system
    defaults, like is done by default for the autoconnect variable.

* resolution, type=floating point, default=0.77
   Default resolution to go for in an experiment. 0.77 is reasonable for standard
    small-molecule experiments.
```

```
* scanrepr, type=integer, default=-1
   Determines how scans are represented in text:
    -1: scan angle from x to y length y-x
     0: scan angle length y-x starting from x
     1: scan angle from x to y

* scanwidthtolerance, type=floating point angle, default=0.003
   Tolerance in degrees that determines whether two scans are equal. Do not touch.

* fileserver, type=tuple of strings, default=()
   Where experiment data files should be stored by default. First element in the
    tuple is the host name, second the share name, and the rest are successive
    folder names inside the share.

* fileserverprotocol, type=string, default='smb'
   The protocol that should be used to address the file server. Should be either
    'file' (local file) or 'smb' (network drive)

* smbroot, type=string, default='/smb'
   Unix only. Where samba will mount shares made available from other hosts.

* smtphost, type=string, default='localhost'
   Host of a machine that can be addressed via SMTP to send E-mails.

* statuslines, type=integer, default=1000
   Maximum number of lines displayed in status window

* temperaturerampspeed, type=integer, default=240
   The speed to use for ramping the temperature of a low-temperature device, in K
    per hour.

* universalfnprotocols, type=tuple of strings, default=('smb','file')
   Allowed protocols to exchange files with the BIS. Should be
   ('smb','file') when BIS is running on the same machine, and ('smb',)
   if BIS is running remotely.
```

```
* userpos1, type=scan, default=Not set
   User-defined instrument axis position. See the example on page D-2.

* userpos2, type=scan, default=Not set
   User-defined instrument axis position. See the example on page D-2.

* userpos3, type=scan, default=Not set
   User-defined instrument axis position. See the example on page D-2.

* userpos4, type=scan, default=Not set
   User-defined instrument axis position. See the example on page D-2.

* calibweaklevel, type=integer, default=10
   Minimal I/sigma for peaks to be used in calibration routines.
```

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