X-Seed 4.1

A program for supramolecular crystallography

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Contents

1. Introduction

1.1 What is X-Seed?

X-Seed is a crystallographic tool with three primary functions: (i) to serve as a graphical user interface (GUI) to the well-established *SHELX* programs for crystal structure solution and refinement, (ii) to facilitate the exploration of crystal packing, and (iii) to produce high-quality molecular graphics *via* *POV-Ray*. By now there are several other computer programs that serve similar functions; these include *WinGX*, *ShelxLE*, *Olex²*, *Mercury*, and others. Despite significant overlap in functionality, each of these programs also possesses unique features and the crystallographer would be wise to consider all of these programs as a complete toolbox rather than to settle on using only one of them exclusively.

    X-Seed is a *Microsoft Windows* program. It was coded in Pascal using Borland's *Delphi 5* Integrated Development Environment and Version 4 consists of approximately 120,000 lines of code. Development of *X-Seed* began in 1998 and the first stable version was distributed in 1999 as commercial software. Over the past 17 years *X-Seed* has undergone many minor upgrades but the *SHELX* interface remained specific to *SHELX-97* during all that time. In 2013 *SHELX* was upgraded and extended, and regular minor updates have been released since then. The author of *X-Seed* never anticipated that his software would still be in widespread use by 2016, and so he allowed the web site URL to lapse, stopped updating the manual and also stopped making any significant changes apart from those that he felt were personally useful. However, many long-time *X-Seed* users have continued to use *X-Seed* despite the author's inertia to add support for the new commands and sub-programs of *SHELX*. After much pressure from these users over the past few years, the author has finally revisited the code in order to modernize *X-Seed*. This occurred mainly over the New Year period of 2017, and most of the upgrades involve support for the 2016 version of *SHELX* and a minor facelift of some of the *X-Seed* controls. *X-Seed* has been free since 2010 and will remain so.

    Throughout this manual, programs and files are indicated using *bold italic lettering*. References to program controls (e.g. menu items, dialog boxes, buttons, control panels, etc.) are in green and mouse/keyboard operations in purple.

1.2 Credits

X-Seed was developed and continues to be maintained by:

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1.3 Installing X-Seed

Run the setup program *XSeed400.EXE* to install *X-Seed* into any folder of your choice. **Note:** the various versions of **Microsoft Windows** implement different security measures that may encumber optimal usage of *X-Seed*. It is therefore advisable to install *X-Seed* as an Administrator.

1.3.1 Setting up the ancillary programs

In order to gain the full benefit of **X-Seed's** ability to communicate with other programs such as *SHELXS*, *SHELXD*, *SHELXT*, *SHELXL* and *POV-Ray*, these programs must first be gathered from their various sources as described below. A recompiled and slightly modified version of the third-party program *LAZY PULVERIX* is included in the *X-Seed* installation. For *X-Seed* to be able to invoke the ancillary programs, it also needs to be informed of their respective locations by means of the Preferences | Program Settings menu. These programs are not required to share the same folder with *X-Seed*.

1.3.2 *SHELX* programs

SHELXS, SHELXD, SHELXT and *SHELXL* were developed by Professor George M. Sheldrick and must be obtained from him. If you do not already have copies of these programs, details of how to obtain the *SHELX* software suite can be found at http://shelx.uni-ac.gwdg.de/SHELX/.
Note: it is highly recommended that you download the December 2017 (or later) build of the *SHELX* programs.

1.3.3 *LAYER*

The program *LAYER* was developed by *X-Seed's* author and is distributed as part of the *X-Seed* package.

1.3.4 *MSRoll*

MSRoll installs with X-Seed Version 4. The MSRoll executable can be downloaded from http://academic.sun.ac.za/barbour/Software.html. Permission to distribute the executable file is given here: https://web.archive.org/web/20160315232228/http://biohedron.drupalgardens.com/content/pricing

1.3.5 POV-Ray

POV-Ray can be obtained as Freeware at www.povray.org and it has its own installation protocol. The *POV-Ray* executable file is called *pvengine.exe* and is usually located in the *bin* folder under the *POV-Ray* installation folder.

1.3.6 LAZY PULVERIX

A modified version of *LAZY PULVERIX* is distributed with *X-Seed* by kind permission of Professor Erwin Parthe of the University of Geneva. The executable files *lazy.exe* and *pulv.exe* *must* be in the same folder (*X-Seed's* installation procedure takes care of this).

1.3.7 XRDPLOT

XRDPlot is a part of the *X-Seed* program suite and is used to display powder patterns calculated by *LAZY PULVERIX*. The executable file *XRDPlot.EXE must* reside in the same folder as the *LAZY PULVERIX* executable programs *lazy.exe* and *pulv.exe* (The *X-Seed* installation program will usually take care of this).

1.3.8 POVLabel

POVLabel is part of the *X-Seed* program suite and is used to edit and position the atomic labels of *POV-Ray* images. *POVLabel* can be invoked from within *X-Seed* and it is merely a utility program for labeling images.

1.3.9 PNG2AVI

Using *POV-Ray*, *X-Seed* is able to produce files representing the frames of an animation. *PNG2AVI* can be called from within *X-Seed* to stitch the frames together to produce a single animated file.

1.4 How does X-Seed work?

X-Seed is a GUI to the various ancillary programs. All of these programs are number crunchers that require instructions and data to be input as text files, and they similarly output their results as text files. *X-Seed* provides the use with a near-seamless way of interacting with these programs by means of a point-and-click protocol rather than extensive text editing. So the user manipulates a crystal structure model graphically and provides the necessary additional instructions by means of dialog boxes with a standardized *MS Windows* look and feel. *X-Seed* utilizes the *Microsoft Windows* design philosophy of making extensive use of a suitable pointing device (mouse or trackball), drop-down menus and dialog boxes to intuitively perform various operations on the crystallographic model.

   Although its other features are generally quite useful, *X-Seed's* primary purpose is to interface with *SHELX* for structure solution and refinement such that it should be possible to solve and refine a well-behaved crystal structure with minimal use of the keyboard. The structural model is always displayed and can be manipulated extensively for the purposes of solution/refinement, exploring the extended structure and producing publication-quality molecular graphics.

1.4.1 Program features

Below is a brief list of the program's features. The list is by no means comprehensive and only serves as a general overview. Further details of how each feature is implemented can be found elsewhere in this manual.

1.4.2 Interfacing with the following programs:
1. *SHELXS* (classical small-molecule structure solution)
2. *SHELXD* (dual-space small-molecule structure solution)
3. *SHELXT* (small-molecule structure solution)
4. *SHELXL* (crystal structure refinement)
5. *MSROLL* (calculation of molecular surfaces)
6. *POV-Ray* (rendering of publication-quality molecular graphics)
7. *POVLabel* (placement of atomic labels on high-quality POV-Ray images)
8. *LAYER* (simulation of precession photographs using HKL data)
9. *Lazy Pulverix* (simulates powder diffraction patterns from single-crystal data)
10. *OPiX* (calculates lattice energies – written by Prof. Angelo Gavezzotti)

1.4.3 Display and manipulation of the model:
- Rotate, translate, zoom in and out using the mouse or keyboard
- Various display features (atom labels, electron density peaks, hydrogen atoms, unit cell, stereo)
- View and edit atom properties
- Point-and-click sequential labeling of atoms
- Grow using space group symmetry information
- Locate hydrogen bonds
- Easily define *SHELX* connectivity, constraints and restraints
- Measure distances as well as bond and torsion angles
- Easily transform atomic coordinates of the asymmetric unit atoms
- Move the asymmetric unit around the unit cell using symmetry information
- Import and export various file formats
- Browser for *Cambridge Database DAT* files containing multiple structures
- High-quality molecular graphics via the *POV-Ray* interface

1.5 Terminology

Throughout this manual, certain terms are used of which the meaning may not be immediately obvious to the reader. Some of these terms are be explained or defined below.

ASU	asymmetric unit
CSD	Cambridge Database
CCDC	Cambridge Crystallographic Data Centre
dpi	dots per inch
GUI	graphical user interface
pixel	picture element (a single dot on the screen)
CPU	central processing unit
focus	a window has the "focus" when it is the active window on the computer's desktop - i.e. will respond to the keyboard
fragment	a set of atoms where each atom in the set bonds to at least one other atom in the set

2. User Guide

2.1 Program execution

It is assumed that the user is generally familiar with the *Windows* operating system. These are several ways to invoke *X-Seed*:

1. Normal execution: select *X-Seed* from the start menu or the taskbar.
2. Drag-and-drop icons: if you have a shortcut to *X-Seed* on your desktop then an icon representing a structure file (i.e. a *SHELX RES* or *INS* file, or a *Cambridge Database DAT)* file can be dragged and dropped onto the *X-Seed* icon. The program will then start up and automatically load the structure file.
3. Drag-and-drop icons: If *X-Seed* is already running, a structure file (**.RES*, **.INS*, **.CIF*, **.DAT*) can simply be dragged and dropped onto its main window. This is equivalent to loading the structure file using the main menu.

4. If X-Seed is associated with *SHELX INS* or *RES* files, then double-click the *SHELX* file icon to run *X-Seed* and to load the structure automatically.

2.2 The program Window

X-Seed's program window can be divided into the following five distinct regions:

2.2.1 Main menu

The main menu provides access to a number of options, many of which are also available *via* the control panel, popup menu and keystroke shortcuts. Details of the function of each of the main menu selections are given in **Chapter 3** of this document.

2.2.2 Main Toolbar Ribbon
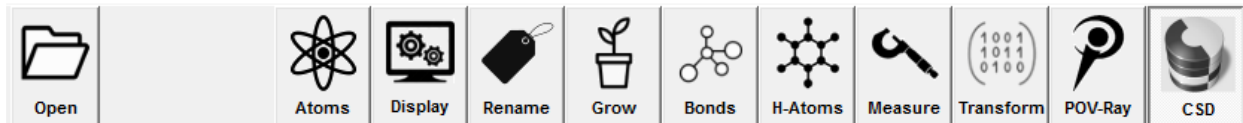
The main toolbar ribbon provides a button to open an existing structure and several additional buttons for accessing the various pages of the control panel (see below).

2.2.3 Control Panel

The control panel is attached to the left-hand side of the main window and contains several pages. The most powerful features of *X-Seed* are accessed *via* this control panel and navigation through its pages is facilitated by the array of buttons on the toolbar. Details relating to the functionality of the control panel pages are given in **Chapter 4** of this document. In total, the control panel consists of 13 separate pages. Each page typically consists of a large number of loosely related options grouped together, and some pages have additional tabbed pages.
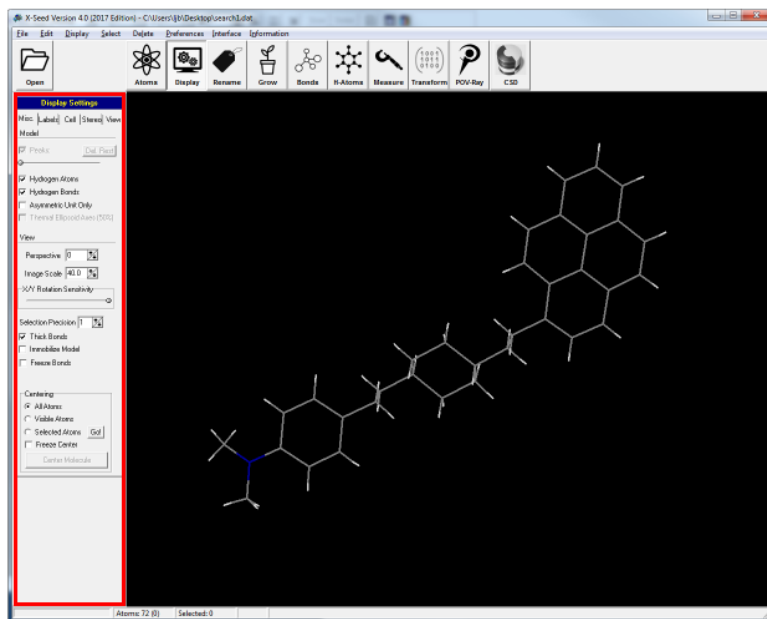
2.2.4 Model Display Area

This is where the structural model is displayed as a wireframe object that can be rotated and translated in real-time. Details of how the model is displayed and manipulated are given in the sections below.

Question: why did the author decide not to use *OpenGL* to display the model? *OpenGL* is a protocol for rendering 3D graphics that can be applied cross-platform.

Answer: it is true that *OpenGL* would have been more aesthetic and it would also have allowed a much better display of atoms as thermal ellipsoids. However, the simple line graphics used by *X-Seed* is much faster to render as one rotates the model using a mouse. While the speed difference is not that noticeable for a few hundred atoms, it would become very noticeable for a few tens of thousands of atoms. The author is of the opinion that the simple line graphics of the *X-Seed* interface provides a clean, crisp display that is more practical for large numbers of atoms.
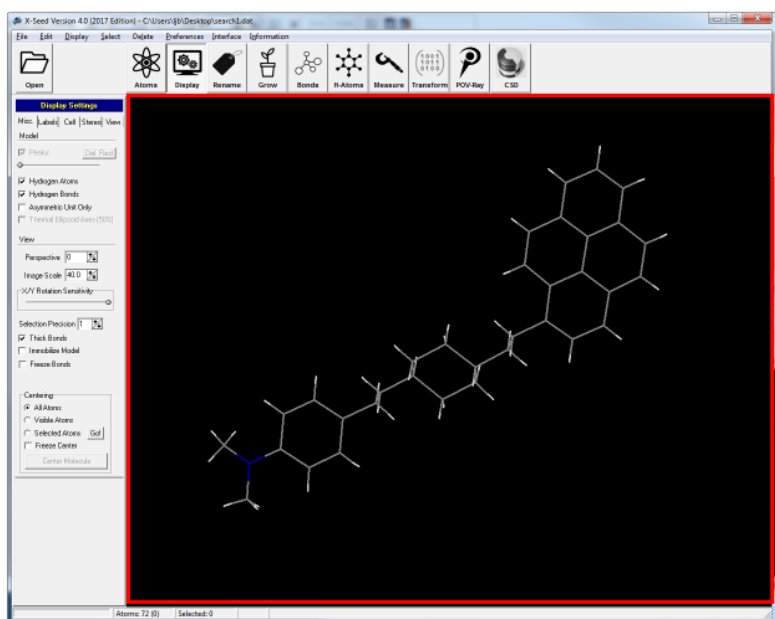
2.2.5 Status bar

The status bar is the narrow bar at the bottom of the window. It provides information regarding the number of atoms present in the model (number of hidden atoms given in parentheses), the name of the atom closest to the mouse cursor, and the length of the bond currently being pointed to. The first subdivision of the status bar will show the progress of operations that may be lengthy (e.g. sorting the atom list, generating a packing diagram, locating hydrogen bonds, etc.)

2.3 Model display

When a structural model containing one or more atoms has been loaded, the structure is displayed in the Model Display Window.

2.3.1 Connectivity
The connectivity of the model is determined using the same bonding criteria used by *SHELX*. Since it is highly desirable that the user sees the connectivity as *SHELX* "sees" it, there is no provision for changing the bonding radii of the elements. These have been hard-coded into the program. However, *X-Seed* provides means to easily add and remove bonds from the connectivity list and will communicate these changes to *SHELX* by means of `BIND` and `FREE` commands.

2.3.2 Atom colors
The colors used for the different element types can be customized *via* the Element Settings dialog box. These colors are referred to as the CPK colors by *X-Seed*. Bonds between two atoms of different CPK color are shown split into the respective colors and the position of the color boundary is proportional to the bonding radii of the atoms involved.

2.3.3 The screen origin
For the purposes of this manual, the screen is referred to a right-handed Cartesian coordinate system with its origin at the center of the model display area, *x* increasing to the right, *y* increasing upwards and *z* perpendicular to the plane of the screen pointing towards the viewer. By default, *X-Seed* places the model such that its centroid is on the screen origin. The screen origin can be moved in the *xy* plane using Shift-Ctrl-Drag (see **Section 2.5**) and can be repositioned at its default location using the Center Molecule option of the Display menu.

2.3.4 Atom labels
The manner in which *X-Seed* handles atomic labels may be confusing at first. Each atom is able to "own" a label. The label can be either its *SHELX* name, or a number representing its peak height (if it's an electron density peak), `PART` number, U_{iso} or S.O.F. `FVAR` number, `HFIX` number or `AFIX` number. By default, when a model is loaded, each asymmetric unit atom owns a label, and the label is its *SHELX* name. When symmetry-related atoms are generated,

they do not own labels by default. Turning the display of atom labels on will result in only the labels of atoms that actually own labels to be displayed. The label ownership and format of any atom can be set using the Labels tabsheet of the Display Settings control panel.

2.3.5 Selecting atoms

Many actions taken by *X-Seed* are performed on the selected atoms *only*. See **Sections 2.4 and 2.5** of this document for descriptions of how to (de)select atoms.

2.3.6 Hiding atoms

If you wish to suppress the display of selected atoms *without deleting them*, you can hide them (see the View/Edit Atoms control panel). Once hidden, atoms will not be displayed until they become "unhidden" again. Hiding atoms is *not* the same as choosing not to display certain atom types (e.g. hydrogen atoms, peaks, non-ASU atoms). For example, when you choose not to display hydrogen atoms, these atoms are simply not displayed, but are not really hidden. If a hydrogen atom has specifically been hidden, it will not be unhidden simply by the action of choosing to display hydrogen atoms. Many operations that are performed on the model are performed only on non-hidden atoms (e.g. exporting, *POV-Ray* images, selection, and many more).

> *Example:*
> You have completed the structure of an inclusion compound and wish to make a *POV-Ray* image of only the host molecule, but don't want to delete the guest just for this purpose. If any atoms are selected, press the spacebar to clear selection. Ctrl-click on the guest molecule to select all of its atoms, right-click to bring up the pop-up menu, and then click on Hide Selected Atoms to hide the guest (the Ctrl-H shortcut key combination performs the same function). When you render the *POV-Ray* image, only the atoms displayed on screen will be included in the picture. Reinstate the guest molecule by selecting Unhide All Hidden Atoms button from the pop-up menu (or using the hot-key Ctrl-U).

2.3.7 Showing thermal ellipsoid axes
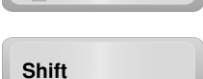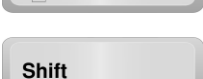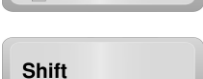
This option can be turned on and off by pressing e on the keyboard, or *via* the Display Settings control panel. The principal axes of the 50% thermal ellipsoids of anisotropic atoms of the ASU will be displayed. Non-positive definite atoms will be encircled. This feature serves as a useful quick check that the thermal ellipsoids are reasonable.

2.4 Using the Keyboard

A number of keystrokes can be used as shortcuts to various options when a structural model is displayed. These keystrokes are not case-sensitive and are listed below.
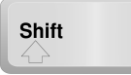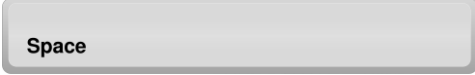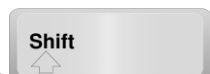
2.4.1 Rotate the model on screen

Although using the mouse is preferable, these cursor keys rotate the model by an exact angle.

↑	−5° rotation about the *x* axis
↓	+5° rotation about the *x* axis
←	−5° rotation about the *y* axis
→	+5° rotation about the *y* axis
Page Up	− rotation about the *z* axis (the angle depends on X/Y Rotation Sensitivity; see <u>4.2.1 Misc. Tab</u>)
Page Down	+ rotation about the *z* axis (the angle depends on X/Y Rotation Sensitivity; see <u>4.2.1 Misc. Tab</u>)
Shift ↑	−90° rotation about the *x* axis
Shift ↓	+90° rotation about the *x* axis
Shift ←	−90° rotation about the *y* axis
Shift →	+90° rotation about the *y* axis
Shift Page Up	−90° rotation about the *z* axis
Shift Page Down	+90° rotation about the *z* axis

2.4.2 Other keystrokes

Delete	delete selected atoms/bonds
Space	select or unselect all atoms*
Ctrl Space	select or unselect all bonds*

Shift **Space**		toggle selected atoms
F2		invoke a popup menu that will allow you to change the element type assigned to the selected atoms
Ctrl **H**		hide selected atoms
Ctrl **U**		unhide selected atoms
Ctrl **C**		create a centroid using the selected atoms
Ctrl **R**		render the current model using *POV-Ray*, even when the *POV-Ray* control panel is not displayed
Ctrl **Q**		convert all visible electron density peaks to carbon atoms
Ctrl + **L**		label the selected atoms
Ctrl + **Shift** **L**		unlabel the selected atoms

* if no atoms/bonds are currently selected, all will be selected. If at least one atom/bond is currently selected, the selection will be cleared.

2.4.3 Resize the image (using the mouse is preferable)

+ **=**	zoom in
– **-**	zoom out

2.4.4 Toggle switches for specific display options

A	show or hide asymmetric unit atoms only
C	show or hide unit cell

Key	Description
E	show or hide thermal ellipsoid axes
H	show or hide hydrogen atoms
L	show or hide atomic labels
Q	show or hide electron density peaks
P	show or hide ***POV-Ray*** colors
R	reset number to 1 when renaming atoms
S	stereo view on or off
T	reset suffix to A when renaming atoms
) 0 .. **(9**	toggle display of atoms with the corresponding PART number

2.5 Using the Mouse

Within the model display window, the mouse is used to perform several different functions. In the text below, left-drag means holding down the left mouse button while dragging the mouse cursor around the screen. Ctrl-left-drag means holding down the Ctrl key while performing this action, etc...

2.5.1 Pointing
When you point at an atom or a bond, the name of the atom or the bond length is displayed on the status bar. If the View/Edit Atoms control panel is displayed, the properties of the closest atom are displayed on the panel.

2.5.2 Rotating, translating and zooming
Left-drag	rotate the model about the *x* and *y* axes
Shift-left-drag	rotate the model about the *z* axis
Ctrl-left-drag	zoom in and out

Shift-Ctrl-drag   translate the model in the *xy* plane (this action actually translates the screen origin)

2.5.3 Selecting atoms/bonds*

Left-click   (un)select the atom/bond closest to the cursor

Ctrl-left-click   (un)select all atoms/bonds of the entire fragment

Shift-left-click   (un)select all atoms/bonds of the same type

Alt-left-click   (un)select all atoms of the same `PART` number

Shift-Ctrl-click   combination of Ctrl-click and Shift-click

Ctrl-Alt-click   (un)select all instances of this atom/bond (i.e. ASU *and* symmetry-generated)

Alt-drag   "lasso" atoms to be selected

Ctrl-Alt-drag   "lasso" parts of fragments to be selected

Shift-Alt-drag   "lasso" atoms to be <u>un</u>selected

Shift-Ctrl-Alt-drag   "lasso" parts of fragments to be <u>un</u>selected

* By default, the cursor must be within 0.6 Å of the atom or bond (in the plane of the screen) for the selection to take place. This default setting can be changed on the Display Settings control panel (see Selection Precision). The distance of the cursor to the bond is measured to the *center* of the bond. Either an atom or a bond can be selected, depending on which is closer when the mouse button is clicked. Selected atoms are displayed as filled circles and selected bonds are shown as thick lines. If the atom or bond clicked on is already selected, it will become "un-selected". If selection requires a group of atoms/bonds to be (un)selected (e.g. Ctrl-left-click), the selection/unselection of the entire group will depend on the current selection status of the clicked-on atom/bond.

2.5.4 Popup menu

If you right-click when the mouse cursor is over the model display area, a popup menu will appear. This menu has several options that are also available either on the control panel or *via* the main menu, or both. Being able to invoke the popup menu means that options can become available to you with minimal movement of the mouse cursor. Extensive mouse movement can become annoying if required often. In addition, the desired control panel may not always be visible, and the popup menu allows you to access options for which you might otherwise have to change the currently-displayed control panel page. The options available on the popup menu will usually depend on the selection status of the model (e.g. if no atoms are selected, the Delete Atom(s) menu item will not be present).

2.5.5 Double-click on an atom

This action activates the following dialog:
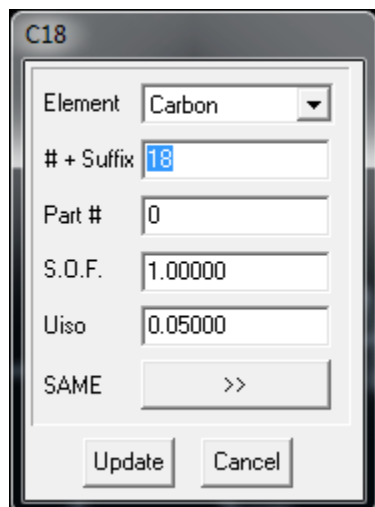
Using this dialog, you can change various properties and parameters belonging to an atom. Press the Enter key or click on the Update button to accept the changes you make. Press the Esc key or click on the Cancel button to discard the changes. Even if the atom on which you double-clicked is a symmetry-generated instance of an asymmetric unit atom, it will be the properties of the parent (i.e. the asymmetric unit atom) that will be altered. All symmetry-generated instances of this atom will be updated too.

To make renaming an atom easy, when the dialog appears the atom name is automatically selected. So to rename an atom you can simply double-click on it, type the new name and then press Enter.

2.6 Using the toolbar ribbon buttons
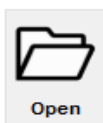
Read a crystal structure model in various supported formats. When the Open dialog box appears, select the file type using the Files of type dropdown list box.
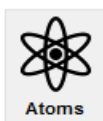
Activate the View/Edit Atoms control panel.
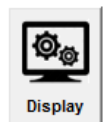
Activate the Display Settings control panel.
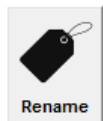
Activate the Atom Names control panel.
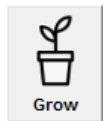
Activate the Grow control panel.
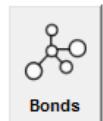
Activate the Add/Remove Bonds control panel.
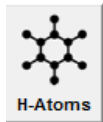
Activate the Generate H-Atoms (HFIX) control panel.

Activate the Measure control panel.

Activate the Transformations control panel.
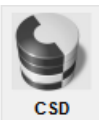
Activate the CSD Browser control panel.

Activate the POV-Ray control panel
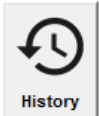
Activate the SHELX History panel

2.7 Space groups

X-Seed uses the file *space groups.lib* for all information relating to the space groups. This library file *must* be in the same folder as *X-Seed*. A *SHELX INS* or *RES* file does not actually contain the name of the space group - it uses `LATT` and `SYMM` instructions to specify the symmetry operators. When it reads such a file, *X-Seed* generates the full list of symmetry operators and checks these against the entries in the library file in order to determine the space group. The library file contains all settings of the 230 space groups - however, if the structure is centrosymmetric, the origin *must* lie on an inversion center. In the Crystal Data dialog, you can specify the space group by typing the short standard Hermann-Mauguin symbol. For monoclinic systems, the full Hermann-Mauguin symbol can also be used – if the short symbol is used, it is assumed that the *b* axis is unique.

3. Main Menu Options

Overview

The main menu of the program is dynamic - i.e., only options that are applicable according to the current status of the model will be presented or enabled. Thus, although the actions of all the possible menu options are described here, they may not always be accessible.

Many of the options available on the main menu are also available *via* shortcut keystrokes (see **2.4 Using the Keyboard**), the main toolbar (**2.6 Using the Toolbar**), the model display area popup menu (**2.5 Using the mouse**) and the control panel (**Chapter 4, Control Panel Overview**).

3.1 File

3.1.1 Open File
Read a **SHELX INS** or **RES** file, or a **Cambridge Database DAT** file. When the Open dialog box appears, select the file type using the Files of type dropdown list box.

3.1.2 Export Structure
Allows you to export the current structure in various formats:

INS	A **SHELX** formatted file will be saved as a rather minimalist **INS** file, which contains only essential structural data. No special **SHELX** instructions regarding solution or refinement procedures will be added to this file. Only the asymmetric unit is saved.
XYZ	Cartesian coordinates of *all currently displayed atoms*, including those generated by symmetry
XYZC	As above, but including the unit cell parameters
PDB	Export *all the currently displayed atoms* to a minimalistic **PDB** formatted file that contains enough information for **PDB**-oriented programs such as **RasWin**.
PDB for SHELXD	Export currently displayed atoms to a **PDB** file that can be used by **SHELXD** to search for a fragment.
CIF	A rudimentary **CIF** file that can be read by programs such as **CrystalExplorer** and **OPiX**.
INS (all atoms)	Export *all currently displayed atoms* to a **SHELX INS** file in P1.

Note: more file formats might be added to this list in future upgrades.

3.1.3 Exit
Closes the current structure and exits X-Seed.

3.2 Edit

Options shown below with blue headings are also available on the View/Edit Atoms control panel and *via* the popup menu (see **2.5 Using the Mouse**).

3.2.1 Crystal Data
Activates the Crystal Data dialog box, which allows you to alter various parameters relating to the same.

3.2.2 Change Element
Allows you to change the element of the selected atom(s). A list of elements constructed from the current molecular formula is presented.

3.2.3 Add Hydrogens
Activates the Generate H-Atoms (HFIX) control panel.

3.2.4 Make Anisotropic
Alters the properties of the selected atoms such that *SHELXL* will refine them *anisotropically* in the next run (facilitates the generation of `ANIS` cards in the next *INS* file).

3.2.5 Make Isotropic
Alters the properties of the selected atoms such that *SHELXL* will refine them *isotropically* in the next run. When this option is applied to anisotropic atoms, the starting value of U_{iso} is calculated from the current U_{ij} values.

3.2.6 Change Q's to C's
If carbon atoms are present in the molecular formula, this option will assign all the *currently displayed* peaks as carbon atoms. This option is especially useful after the solution of an organic structure where most of the peaks may represent carbon atoms.

3.2.7 Generate Centroid
If two or more atoms are selected, this option generates a non-weighted centroid using the atomic positions of the selected atoms. If necessary, the centroid can be assigned an element type and can subsequently be refined as an atom (if chemically applicable, of course).

3.2.8 Locate All H-Bonds
Locates all possible hydrogen bonded contacts according to the criteria set in the Program Settings dialog box. If necessary, symmetry generation of atoms will occur. This option is also available of the Add/Remove Bonds control panel.

3.2.9 Sort Atom List
Sorts the asymmetric unit atoms using criteria based on their names. Realistically, this is only of consequence with regard to the generation of the next *SHELX INS* file (and when the model is exported). This option is also available on the Atom Names control panel.

3.2.10 Restrain

Activates the Restrain (SHELXL) control panel, where you can define `DFIX` and `DANG` instructions to be added to the *SHELXL INS* file. Bonds to hydrogen atoms can also be normalized to a preferred value here.

3.2.11 Transform Coordinates

Activates the Transformations control panel.

3.2.12 Transform Unit Cell

Activates the Transform Unit Cell dialog, which allows you to input a matrix with which to transform the unit cell. You can also provide the new space group (Warning: transforming space groups and space group settings can be tricky – you should know what you're doing!). The dialog also offers the option of transforming the HKL file and changing the base name of the file. The unit cell will be transformed using the transformation matrix **M**, while the atomic coordinates will be transformed by applying the inverse of the transpose of **M**: $(\mathbf{M}^T)^{-1}$.

3.2.13 Duplicate Atoms

Allows the user to create duplicates of the selected atoms.

3.2.14 Add New Atom

Allows the user to create a new atom to be added to the model.

3.2.15 Hide Selected Atoms

Hides the selected atoms. See Model display for details.

3.2.16 Unhide All Atoms

Unhides all hidden atoms.

3.2.17 Macro

Activates the Macro dialog box. See Section 5.18 Macro and Appendix A – Macro Commands

3.3 Display

Many of the following options are available as shortcut keystrokes (**2.4 Using the keyboard**), on the Display Settings control panel and via the popup menu (see **2.5 Using the mouse**).

3.3.1 Stereo

Toggle stereo display. Advanced stereo settings can be adjusted on the Display Settings control panel.

3.3.2 Unit Cell

Toggle unit cell display. Advanced unit cell display settings can be adjusted on the Display Settings control panel.

3.3.3 Hydrogen Atoms
Toggle display of hydrogen atoms.

3.3.4 Peaks
Toggle display of electron density peaks.

3.3.5 Atom Labels
Toggle display of atomic labels.

3.3.6 Hydrogen Bonds
Toggle display of hydrogen bonds.

3.3.7 ASU Only
Toggle between displaying *only* the asymmetric unit atoms or *all* atoms, including those that are symmetry generated.

3.3.8 Thermal ellipsoid axes
Toggle display of axes of the 50% probability ellipsoids of anisotropic ASU atoms.

3.3.9 Center Molecule
If the model has been translated on screen using Shift-Ctrl-drag (see Using the mouse), selection of this option will once again place the overall centroid on the screen origin.

3.3.10 Reset Rotation Matrix
Rotation of the model on the screen orients it according to a rotation matrix. This option resets the rotation matrix to give the default view.

3.3.11 SHELX History
Display the SHELX History control panel (see Section 4.13).

3.3.12 Animate
Activates the Animate control panel.

3.3.13 Molecular Volume
Performs a molecular volume calculation using the selected atoms. Note that this option is not as credible as using *MSRoll*.

3.3.14 Cartesian → Cell Coordinates
If you have the Cartesian coordinates of a point, you can then convert these to unit cell coordinates. You will also be presented with the option of placing an atom at this position. This feature is useful when *MSRoll* provides you with the Cartesian coordinates of the centroid of a molecular cavity.

3.4 Select

This menu provides the user with several different ways to select or unselect atoms according to certain criteria.

3.4.1 Clear Atom Selection
Unselects all selected atoms. Pressing the spacebar has the same effect. This option *does not* clear bond selection. Use Ctrl-spacebar to do this.

3.4.2 Clear Bond Selection
Unselects all selected bonds. Pressing Ctrl-spacebar has the same effect.

3.4.3 Select All...
This should be self-explanatory.

3.4.4 Deselect All…
This should be self-explanatory.

3.4.5 Toggle All...
This should be self-explanatory. The selection status of atoms that fall into the category chosen will be toggled.

3.4.6 Allow Bond Selection
Turns bond selection on and off. At times it is not desirable to select bonds and it can become rather annoying when, in attempting to select atoms, you also end up selecting bonds owing to inaccuracy in your hasty pointing and clicking. When such a time arises, you can disable the bond selection feature. You cannot turn off atom selection because this is the most commonly used selection feature by far.

3.5 Delete

Atoms present in the model can be deleted. When deleting a series of atoms, symmetry-generated atoms are deleted without warning. However, if you attempt to delete atoms belonging to the asymmetric unit, you will be warned and prompted to either continue with, or cancel this operation. Symmetry-generated atoms are dependent on their asymmetric unit 'parents' for their positional and other information. If you delete an asymmetric unit atom of which there are symmetry-generated instances, the latter will also be removed from the model as they will no longer have 'parents'. Once deleted, asymmetric unit atoms can be recovered, but their symmetry-related instances will have to be re-generated.

3.5.1 Undelete Atoms
Activates the Undelete Atoms dialog to allow recovery of deleted ASU atoms.

3.5.2 Selected Atoms
Deletes all selected atoms.

3.5.3 Selected Bonds
Deletes all selected bonds

3.5.4 All Peaks
Deletes all electron-density peaks.

3.5.5 All Residual Peaks
Deletes all 'residual' peaks. Residual peaks are peaks that are too close to established atoms to be indicative of atoms missing from the model. Is it especially common for such peaks to appear close to 'heavy' atoms after refinement. Using the Program Settings dialog, you can set the program's distance criteria for deciding what 'too close' means. After a *SHELXL* refinement run, this menu option allows you to quickly 'clean' a structure that contains numerous peaks that are obviously not candidates for missing atoms. Be aware that residual peaks may be indicative of disorder in some cases.

3.5.6 All Hydrogen Atoms
Deletes all hydrogen atoms.

3.5.7 All Symmetry-generated Atoms
Deletes all atoms not belonging to the asymmetric unit (excluding peaks and centroids).

3.5.8 Clear BIND List
Removes all bonds that were manually added to the connectivity list.

3.5.9 Clear FREE List
Restores all bonds that were manually removed from the connectivity list.

3.5.10 Clear Hydrogen Bonds
Removes all hydrogen bonds from the connectivity list.

3.5.11 Inter-SymmOp Bonds (Selected Atoms)
Removes all bonds between selected atoms where those atoms have been generated using different symmetry operators.

3.6 Preferences

3.6.1 Program Settings
Activates the Program Settings dialog.

3.6.2 Element Settings
Activates the Element Settings dialog.

3.6.3 Display Settings
Activates the Display Settings control panel.

3.6.4 Control Panel
Shows or hides the control panel.

3.7 Interface

The interface menu is the gateway to the programs with which **X-Seed** cooperates. Some of the programs (i.e. **LAYER** and **POVLabel**) are invoked immediately while others first require further fine-tuning of their instructions *via* an appropriate dialog box or control panel (e.g. **Lazy Pulverix**). The interfacing programs are all described in further detail in subsequent section of this help file.

3.7.1 SHELXS
Activates the SHELXS dialog for structure solution.

3.7.2 SHELXT
Activates the SHELXT dialog for structure solution.

3.7.3 SHELXD
Activates the SHELXD dialog for structure solution.

3.7.4 SHELXL
Activates the SHELXL dialog for structure refinement.

3.7.5 MSRoll
Activates the MSRoll dialog to calculate cavity/molecular volumes and surfaces.

3.7.6 POV-Ray
Activates the POV-Ray control panel.

3.7.7 POV-Label
Runs the program **POVLabel**.

3.7.8 Layer
Runs the program **LAYER** using the current structure's **HKL** file as input to that program. See the relevant topic describing the program **LAYER** below.

3.7.9 Lazy Pulverix
Activates the Lazy Pulverix dialog.

3.7.10 OPiX

Saves the structure and runs *OPiX*

(*OPiX* was developed by Prof. Angelo Gavezzotti – see http://www.angelogavezzotti.it/).

3.8 Information

3.8.1 About X-Seed

Displays *X-Seed's* splash screen.

3.8.2 X-Seed Manual

Loads this file.

3.8.3 X-Seed Shortcuts

Loads a PDF file that shows all the X-Seed keyboard and mouse shortcuts for performing various useful operations.

3.8.4 X-Seed Home Page

Loads the X-Seed home page into your web browser.

3.8.5 SHELX Home Page

Loads the SHELX home page into your web browser.

3.8.6 About LAZY PULVERIX

Shows the credits for the *LAZY-PULVERIX* software.

4. Control Panels

Overview

Much of the capability of **X-Seed** is accessed *via* the control panels. In most cases, these panels are activated using the buttons on the toolbar.

Important: please keep in mind that one of the primary functions of **X-Seed** is to *construct* a **SHELX INS** file. Therefore, certain operations performed via the control panels do not take effect immediately – they are just coded into the next **INS** file and will take effect after **SHELX** has processed the relevant commands. For example, applying the `ANIS` setting to an atom means that **SHELX** will be instructed to refine the atom anisotropically in the next set of refinements.

4.1 View/Edit Atoms

This is the **default** control panel. It is activated when a new structure is loaded (**unless** the structure is embedded in a **CSD DAT** file) or when the Atoms button is pressed on the toolbar. The upper half of the panel is intended for informative purposes only. The parameters relating to the atom currently pointed at are shown here. The lower half of the panel allows you to perform various operations on selected atoms. Each of the lower-half controls is explained in turn below. For further elaboration of some of the concepts referred to below, see the **Section 2.2.4**. In order to apply certain operations to atoms, the atoms *must first be selected*. When you click on one of the control panel buttons, the relevant operation is immediately applied to the selected atoms and the atom selection will also be cleared.

Select atoms by Uiso drag the left-hand slider to select atoms with Uiso smaller than the slider position. Similarly, drag the right hand slider to select atoms with U_{iso} larger than the slider value. This function allows you to easily check which atoms have unusually low or high U_{iso} values.

Find Atom type the name of an atom and either press Enter or click on the Find Atom button. The atom name is not case-sensitive. If an atom of that name exists, it will become selected.

ANIS Insert `ANIS` commands into the next **SHELX INS** file to cause the selected atoms to be refined anisotropically. Unless the `NEUT` command is specified in the Crystal Data dialog box (for neutron diffraction data), any selected hydrogen atoms will be ignored. No

action is taken for selected atoms that are already anisotropic, or for Q peaks.

Isotropic If the selected atoms are *anisotropic*, this selection will flag them for *isotropic* refinement during the next **SHELXL** refinement. **X-Seed** will also *immediately* regard these atoms as being isotropic and will calculate their equivalent U_{iso} values from their current anisotropic thermal parameters.

Element You can select the new element type to assign to the selected atoms or peaks from a popup menu list, which is dynamically, constructed using the information contained in the current molecular formula (i.e. the `SFAC` instruction). The keyboard shortcut F2 performs the same function.

To ASU This option will assign the selected atoms as *asymmetric unit* atoms. If the structure has been extended using Grow, and multiple instances of the same atom are selected, the final instance of that atom in the program's internal list will become the asymmetric unit atom. This operation is useful for manually assigning which instances of the atoms should be regarded as the asymmetric unit. Remember, only the asymmetric unit atoms are written to **SHELX INS** files for refinement of the structure.

Centroid Generate a *centroid* of the currently selected atoms. The non-weighted average position of the selected atoms is calculated and a special atom of type 'Centroid' is created at this position. The centroid 'atom' can be treated as any other atom but will not be written to any files as long as it is assigned as a centroid. A centroid 'atom' will not bond naturally to any atom, but you can manually add bonds to it if you like (e.g. for producing images). You can reassign a centroid as an atom at any time, in which case it will become part of the asymmetric unit.

Apply (S.O.F., Uiso, AFIX, PART, Fix/Unfix Atom's Position): this function allows you to apply the checked settings to multiple atoms at the same time. SOF, Uiso and `PART` are atomic properties. Fix Atom's Position will add 10 to all three of the atomic coordinates of each selected atom, which instructs **SHELX** not to refine these parameters (Unfix will reverse this). If AFIX is checked, then an `AFIX` instruction with the given value will be inserted before the selected atom(s) in the next **INS** file. In particular, applying SOF and PART collectively to selected atoms is extremely useful when defining a disordered model.

4.2 Display Settings

Use this control panel to change various display settings. Most of the settings on this panel are self-evident and will thus only be described briefly below. In order to accommodate the many display settings that can be altered, this control panel contains five pages:

4.2.1 Misc. Tab

Display Settings

Misc. | Labels | Cell | Stereo | View

Model

☑ Peaks: 20   Del. Rest
                           ○ 0.1

☑ Hydrogen Atoms
☑ Hydrogen Bonds
☐ Asymmetric Unit Only
☐ Thermal Ellipsoid Axes (50%)
    ☐ Iso atoms too

View

Perspective   0   ⬍
Image Scale   30.0   ⬍

X/Y Rotation Sensitivity
━━━━━━━━━━━━━━○

Selection Precision 1 ⬍
☑ Thick Bonds
☐ Immobilize Model
☐ Freeze Bonds

Centering
  ◉ All Atoms
  ○ Visible Atoms
  ○ Selected Atoms   Go!
  ☐ Freeze Center
    Center Molecule

The first page contains *miscellaneous* display setting options. You can suppress the display of electron density peaks, hydrogen atoms, hydrogen bonds, symmetry-generated atoms and thermal ellipsoid axes. Note that there are hotkeys that also perform most of these functions. **Peaks** When the display of electron density peaks is not suppressed, you can set the maximum number of peaks to be displayed (alternatively, roll the mouse wheel). Peaks are stored in the program's internal atom list in descending order of intensity. Thus, if you only choose to display 10 peaks, these will be the 10 strongest peaks in the list. Use the Del. Rest button to delete only the peaks that are further down the list than the maximum number of peaks chosen for display. Usually, the current model is displayed with its non-weighted center of mass on the screen origin (i.e. at the center of the model display area). When the display of certain types of atoms is suppressed, the centroid is *not* recalculated for just the displayed atoms. If this were to occur, the model would appear to 'jump' around the screen when you, for example, turn off the display of hydrogen atoms. This would quickly become annoying if all you want to do is turn various atoms on and off for clarification of the structure. See Centering below if you *want* the molecule to be re-centered. See the subtopic **Section 2.2.4** for further details relating to how the model is displayed. The Perspective value represents the angle in degrees between the left- and right-hand edges of the model display area and the viewpoint. This perspective value is also used by **POV-Ray** when the image is ray traced. A perspective value of 0 is best for most images intended for publication. The Image Scale setting can be used to adjust the size of the model precisely - using the Ctrl-mouse-drag method is not precise. Use this setting if you would like to compare two structures, displayed at the same scale, using two instances of **X-Seed** on your desktop (or if you would like to render two images of different structures to the same scale). The X/Y Rotation Sensitivity slide bar can be used to adjust the sensitivity of rotation about the screen *x* and *y* axes to the movement of the mouse whilst left-dragging. Selection Precision allows you to specify how close the mouse pointer needs to be to an atom in order to select it. Thick Bonds (default) displays the wireframe model with thicker lines that are easier to see. When this setting is turned off the wireframe model is shown using lines with a thickness of one pixel. The response to on-screen rotation is significantly faster for the thin lines, especially if the displayed model consists of > 10,000 atoms. Use the Immobilize Model setting to 'freeze' the model in its current orientation so that it cannot be rotated accidentally.

Centering: here you can select various criteria for placement of the model on the screen origin. These options are mostly self-explanatory. If Freeze Center is checked, then the current centering position will remain locked, even if you grow the model. If the model has been

translated away from the screen origin using Shift-Ctrl-left-drag (see **2.5 Using the mouse**), it can be re-centered using the Center Molecule button. This button is only enabled if the molecule is not currently centered on the screen origin.

4.2.2. Labels Tab
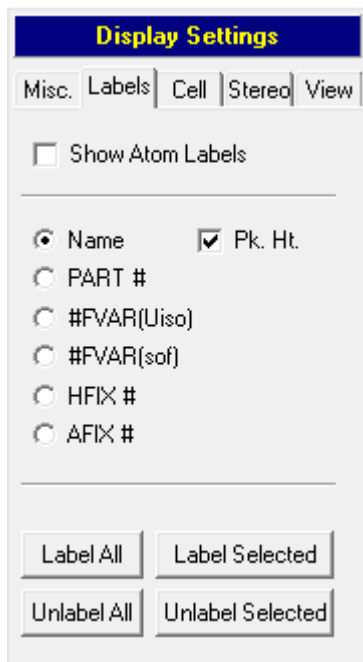
Using the second page, you can turn the display of *atom labels* on and off. Just turning labels on *does not* necessarily mean that all atoms will be labeled, but merely that atoms that actually own labels will have their labels displayed. By default, asymmetric unit atoms and electron density peaks own labels. The labels of the former are their names while the labels of the latter are their peak heights as determined by **SHELX**. Also by default, symmetry-generated atoms do not 'own' labels. However, any atom can be provided with a label. An atom's label can consist of its name, peak height (in the case of peaks), **PART** number, the number of the free variable to which its U_{iso} or site occupancy factor is tied, or the **HFIX** or **AFIX** number currently assigned to the atom. The four buttons on this page allow you to (un)label all the atoms or just the selected ones according to the status of the radio buttons. If Name and Pk. Ht. are both checked, peak names will be their heights as described above. If Pk. Ht. is not checked, the peaks will be assigned the names assigned to them by **SHELX**.

4.2.3 Cell Tab
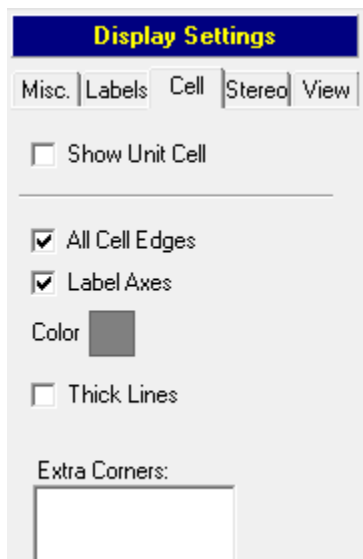
Use this page to turn the display of the unit cell on and off. You can customize the color of the unit cell edges by clicking on the color panel and selecting a color from the standard **Windows** color dialog. Additional features include the display of all 12 cell edges, or just the three principal edges as well as the cell axis labels. You can also select whether the unit cell should be displayed as thick or thin lines.

Extra Corners**:** to display additional unit cells, enter a series of lines where each line consists of three integer numbers that specify an additional cell. These numbers represent the corner furthest from the origin of the additional unit cell. For example, the default the unit cell does not need to be specified, but its furthest corner is at 1 1 1 (i.e. *x/a* = 1, *y/b* = 1 and *z/c* = 1). The line 2 1 1 represents the next unit cell over in the direction of the *a* axis. After typing the line, it drawing of the unit cell(s) will take effect once the mouse pointer places the focus back in the model display area (i.e. click in the model window).

4.2.4 Stereo Tab

Two stereo modes are available. If you choose to display the model in stereo, it can be shown in red/blue (or other colors of your choice) or side-by-side stereo mode. When using the red/blue mode, you will need special stereo glasses with a red filter over the left eye and a blue or green filter over the right eye. Both the view angle and separation can be customized. The view angle represents the rotation angle in degrees of the left-eye model relative to the right-eye model. A mid-range value of about 6 degrees is usually considered to be optimum. The stereo separation is based on an arbitrary scale and can be selected according to personal preference. Click on the color panels to select new colors. Side-by-side stereo images will be rendered as such in POV-ray.
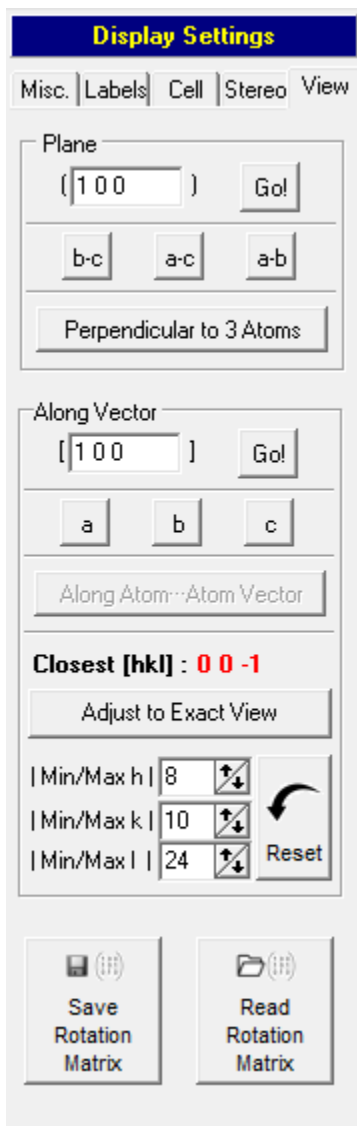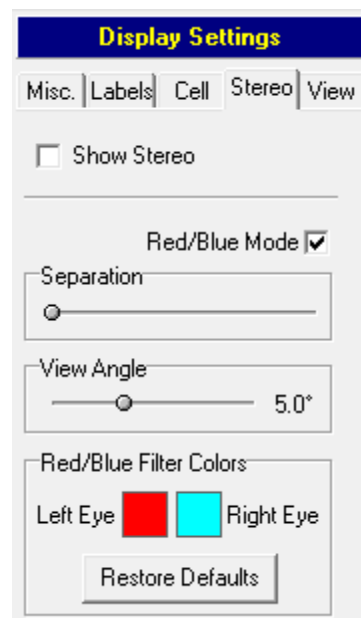
4.2.5 View Tab

Plane: orient the model such that your view is perpendicular to a particular crystallographic plane as specified using the Miller indices. For convenience, buttons are provided for viewing perpendicular to the *bc*, *ac* or *ab* planes: (100), (010) or (001), respectively. You can also select three atoms to define a plane perpendicular to the desired viewing direction. The View Perpendicular to 3 Atoms button will only be enabled if exactly three atoms are selected.

Along Vector: Orient the model such that the view is parallel to [*hkl*] as specified. For convenience, buttons are provided to view along the *a*, *b* or *c* axes: [100], [010] or [001]. If exactly two atoms are selected, you can view Along Atom…Atom Vector, which sets the view direction parallel to the vector linking the two atoms.

As you rotate the model freely using the mouse or cursor keys, the closest view direction [*hkl*] is displayed in red. Since the values of *h*, *k* and *l* can, in principle, range from -∞ to +∞, we need to impose reasonable limits. By default, the limits for |*h*|, |*k*| and |*l*| are set to the rounded values of *a*, *b* and *c*, respectively, but you can also select your own limits. If you change the limits, the "Reset" button will restore the default values. X-Seed calculates the [*hkl*] direction that is closest to the current view direction, but this might not be exactly down [*hkl*]. Pressing the "Adjust to Exact View" will result in the view being *exactly* along [*hkl*].

The buttons "Save Rotation Matrix" and "Read Rotation Matrix" will save and retrieve the current view direction, respectively. This is useful if you want to produce several images with the same view direction.

4.3 Atom Names
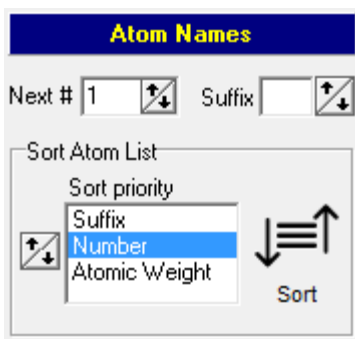
X-Seed considers the name of an atom to consist of three parts - i.e. the atomic symbol, a number and a suffix. For example, if an atom's name is **Na3B,** the element is sodium**,** the number is 3 and the suffix is "B". The only part of the name that is essential is the atomic symbol. The number and suffix, while optional, are generally required to distinguish atoms from one another. Indeed, *SHELX* *requires* that *all* of the names of the atoms of the asymmetric unit be unique (barring the use of **RESI** commands, which are currently not supported by *X-Seed*). Therefore, it is logical to include numbers in the atom names. In addition to numbers, it may also be logical to add an alphabetic suffix to the name in cases where the model contains separate molecular entities. *SHELX* only allows atoms names of up to four characters in length. Since *X-Seed* is largely based on cooperating with *SHELX*, this constraint is adhered to very strictly. Although *SHELX* does not require the atomic symbol to be in the atom's name, *X-Seed* insists on it since this is just good practice. Nevertheless, *X-Seed* will correctly recognize atoms read from a structure file that do not conform to this naming convention, but, if renamed, these atoms will be forced to conform. When you assign an atom name using *X-Seed*, the first character (or two if the symbol consists of two characters) of the name will consist of the atomic symbol of the assigned element. This leaves either two or three characters for the number and suffix. *X-Seed* will also insist that the suffix be an uppercase character. When peaks are assigned as atoms by changing the element type, these newly created atoms will assume names based on their atomic symbol and the original peak number. At some stage during refinement, you will probably want to rename all the atoms to conform to some naming convention of your preference. Generally, such naming conventions involve sequentially numbered atoms with or without a suffix. When you activate this control panel, atom selection is switched off and "re-naming" mode is turned on. The mouse cursor changes to indicate that when you click on an atom, its name will change according to the settings on the control panel. You can set the starting number and current suffix in the Next # and Suffix fields, respectively. When you click on an atom its new name will consist of the *element symbol* plus the *current number* and *suffix*. The Next # field will increment by 1 automatically. Since the atom name is tied only to the parent ASU atom, it doesn't matter whether you click on ASU or symmetry-generated atoms. It will be the name of the parent atom that changes. It is advisable to have atom labels displayed when performing this operation so that you can immediately see the changes taking effect. Whilst in re-naming mode, you can quickly reset the Next # field to 1 by pressing R on the keyboard. If the current suffix is in the range "A" ... "Y", pressing T will increment the character to the next letter of the alphabet. This can also be achieved using the up/down spin button control to the right of the Suffix field.

**Note**: An atom's name may be no longer than 4 characters in length! If you click on, for example, a sodium atom when the current number is **56** and the suffix is **B**, the new name of the atom will be **Na56** and not **Na56B**.

When you click on the Sort button, **X-Seed** will sort the asymmetric unit atom list. The effects of this operation are only of consequence to **SHELX INS** file creation. When sorting the atom list, the highest priority is assigned to the atom's suffix. If two atoms have the same suffix, the next priority is the atom's number. When two atoms have the same number *and* suffix, the higher priority is the atomic weight. If atomic positions are riding on parent atoms (e.g. hydrogen atoms in calculated positions), this is usually indicated by a non-zero `AFIX` parameter. **X-Seed** tries very hard to keep these atoms together, using only the name of the parent atom as the sort criterion for the entire AFIX'ed series.

Owing to the many parameters that need to be considered, sorting might be a lengthy process for very large structures. During sorting, **X-Seed** suspends all other operations and displays the progress of the sorting procedure on the program's status bar.

Recommendation: to avoid potential unreliability of keeping `AFIX` instructions in their correct places during sorting, it would be better to name the atoms of the model systematically before adding hydrogen atoms and applying other `AFIX` commands to non-hydrogen atoms.

4.4 Grow
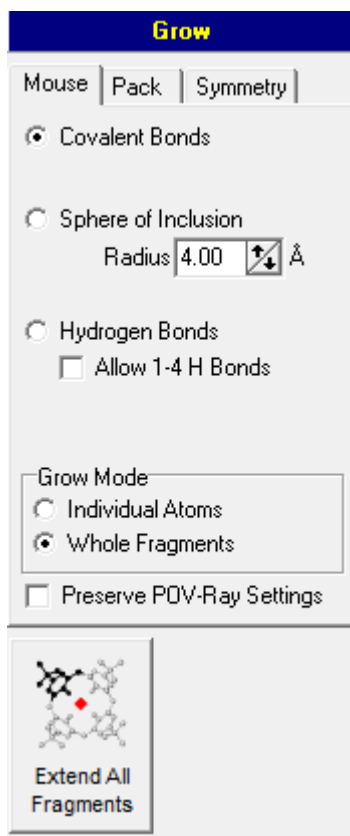
X-Seed provides a number of powerful routines for extending the structure using space group symmetry. When the Grow control panel is activated, atom selection is turned off and the mouse cursor changes to indicate that grow mode is active. In this mode, clicking on an atom (i.e. the *target* atom) will cause **X-Seed** to extend the structure according to the current settings on the control panel. The radio controls Individual Atoms and Whole Fragments are used to specify what should be attached. If the Individual Atoms radio button is active, only individual atoms that meet the growth criteria are generated. If Whole Fragment is active, an entire instance of the relevant part of the asymmetric unit will be generated if at least one of its atoms meet the bonding criteria.

The Preserve POV-Ray Settings checkbox allows you to retain settings such as atom color and radius for newly generated atoms based on these settings for the asymmetric unit atoms.

Important Note: See **Section 2.3.1** for details of how **X-Seed** evaluates whether or not two atoms should be bonded.

There are three tabs that let you select how to grow the structure:

4.4.1 Mouse Tab

When in Covalent Bonds connection mode, one can click on any atom in order to extend the fragment from the target atom. **X-Seed** will then use space group symmetry information to generate all symmetry-related instances of asymmetric unit atoms that can be attached to the target atom. If "Sphere of Inclusion" mode is active, **X-Seed** will generate all atoms within the specified radius (in Å) of the target atom. Using Locate H-Bonds mode, you can click on a target atom to locate any atoms that are defined in the Program Settings dialog as hydrogen-bond donors or acceptors (of course the target atom must also be in the donor/acceptor list). If H-Bond participants are 1-3 connected within a fragment, a hydrogen bond will not be generated (e.g. the oxygen atoms of a nitrate anion). Generally, 1-4 connected hydrogen bonds *should* not be generated either. However, in some cases (e.g. resorcinol) 1-4 donors and acceptors may be hydrogen bonded, and for such cases, you can allow 1-4 H-bonds by checking the appropriate box on the control panel. The criteria for evaluating possible hydrogen bonded contacts are specified in the Program Settings dialog – depending on how you specify the parameters, the hydrogen bond may be generated either between the donor and acceptor, or between the hydrogen atom and the acceptor. Note that **X-Seed** does not know which atom is the donor and which is the acceptor – i.e. the hydrogen bonds generated by **X-Seed** will not end up as **HTAB** instructions in the **INS** file.

4.4.2 Pack Tab
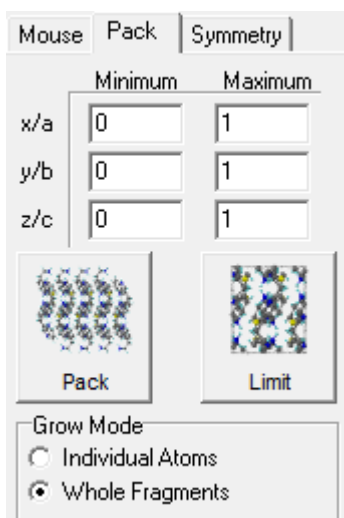
The Pack Range tab is used to quickly generate a packed model. The limits specified in this panel refer to unit cell coordinates. When you press the Pack button, X-Seed generates all atoms (or, if in Whole Fragments mode, fragments that have at least one atom) within the specified limits. It is up to you to make sure that the ASU fragment is also within these limits because the limits will not apply to the ASU itself. This is easily achieved using the Transformations control panel. The Limit button is used to remove any symmetry-generated atoms that lie outside the specified range. This function is performed automatically when you press the Pack button. The Pack feature takes the current Grow Mode setting into account, but the Limit function does not.

4.4.3 Symmetry Tab

When this tab is activated, you will once again be able to select atoms. You can select one of the valid symmetry operators (Jones-Faithful notation) of the current space group from the dropdown list. This will populate a 4x3 matrix according to the symmetry element selected: the leftmost 9 numbers are the 3x3 transformation matrix and the last column specifies the translations in the directions *x*, *y* and *z*. You can also edit this matrix if needed. When you press the Apply Matrix button, the
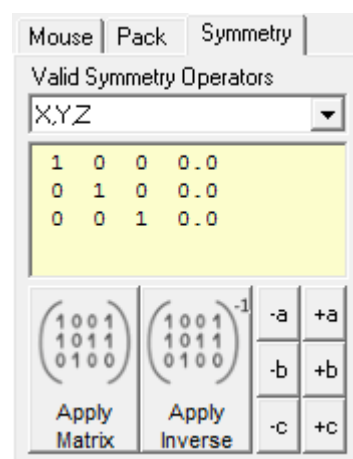
symmetry transformation in the yellow matrix box will be applied to the *selected atoms*. The originally selected atoms will be unselected and the newly generated atoms will be selected. There are 6 shortcut buttons for simple unit cell translations in positive and negative *a*, *b* and *c*. You can also apply the inverse of the shown matrix.

4.5 Add/Remove Bonds

Refer to **Section 2.3.1** for details of how **X-Seed** handles the connectivity settings of the model and how it transmits this information to **SHELXL**. When this control panel is activated, normal atom selection is turned off. In this mode, atoms are selected in pairs in order to manually create or remove bonds between them. Automatic bond generation is accessed via the buttons on the control panel. When two non-bonded atoms are selected, a normal or hydrogen bond between them will be added to the connectivity list (depending on which option is checked in the Manually panel). If the atoms are already bonded according to the connectivity list, the bond will be removed. Bond generation and removal between any two atoms also applies to all symmetry-equivalent instances of the atoms. Use the automatic mode to locate hydrogen bonds. Pressing the Locate all Unique H-Bonds button will initiate a search for *all* possible hydrogen bonds to all the donor/acceptor atoms in the current model according to the criteria specified in the Program Settings dialog. If necessary, symmetry-generated atoms will be added to the model. However, pressing the Locate All H-Bonds button will initiate a search for all hydrogen bonds *within* the current model. No symmetry-generated atoms will be created in this case. A checkbox is included to allow 1-4 hydrogen bonds, if desired. Normal bonds are bonds created by **X-Seed** strictly according to the bonding radii assigned to the different elements.

When 'covalent' bonds are added or removed, the appropriate **BIND** and **FREE** commands will be inserted into the next **SHELX INS** file. Bonds that have created or removed will reside in a list that **X-Seed** maintains internally. This control panel contains three buttons that allow you to clear these lists. These functions are also available *via* the Delete submenu of the Main Menu. It is also possible to delete bonds between specific atom types that fall outside a specified range. Use of this feature should be intuitive, but it should be noted that this operation applies only to *currently selected bonds*.
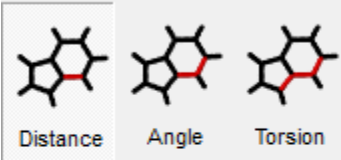
4.6 Generate H-Atoms (HFIX)
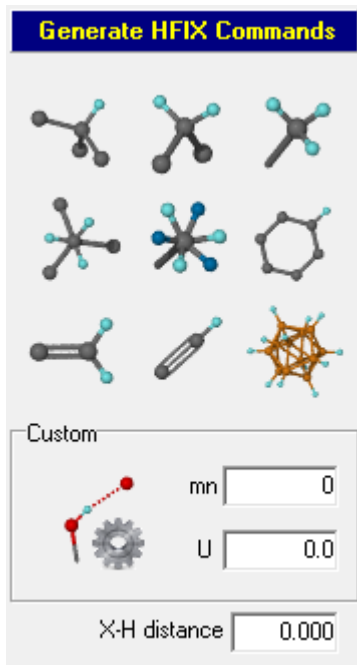
This control panel facilitates the addition of hydrogen atoms (in calculated positions) to the structural model. Please keep in mind that it is *SHELX* that will actually add the hydrogen atoms, and not *X-Seed*. Therefore, by applying the settings on this page you are not generating the hydrogen atoms yet – this will be done during the next set of refinements with *SHELXL*. Remember, the main purpose of *X-Seed* is to construct a *SHELX INS* file and not to do the calculations itself. So the purpose of this control panel is to insert the appropriate **HFIX** commands into the *INS* file. After refinement, the model will be updated to include the hydrogen atoms added by *SHELX*.

Select the atoms to which hydrogen atoms of a particular type (see **HFIX** and **AFIX** in the *SHELXL* instruction manual) have to be added and press the appropriate button. The most common *SHELX* models for adding hydrogen atoms are supported. The actual **HFIX** parameters for each type of model can be customized using the Hydrogen Atoms tabbed page on the Program Settings dialog. Use the Custom button to associate a customized **HFIX** instruction to atoms. If you wish to cancel any **HFIX** associations that you may have applied erroneously to any atoms, apply the Custom setting to these atoms with the mn and U values set to zero. It is the user's responsibility to ensure that the **HFIX** associations make sense, as *X-Seed* will not check the validity of these associations. The worst problem that can occur is that the next *SHELXL* run will terminate prematurely with an informative error message, after which you can correct any mistakes using the Custom button. Any **HFIX** associations applied to atoms will override previous such associations. Naturally, **HFIX** associations are no longer retained if the next *SHELXL* run is successful and the model is updated. The default settings for the different **HFIX** models, as set in the Program Settings dialog, can be altered according to your own preferences. When you exit *X-Seed*, your preferred settings will be written to the *Windows Registry* and thus retained for future use. The desired X-H distance can be specified by entering a non-zero value.

4.7 Measure

Measure bond lengths and bond and torsion angles. Select the desired option and click on atoms to make the measurements. You can log all of the measurements by selecting Display | Measurement Log from the Main Menu. This is a useful feature is you would like to tabulate various measurements, and this can also be semi-automated when using the Macro feature.
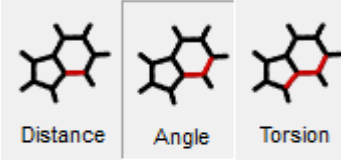
In Distance mode, select two atoms to measure the distance between their centers. If there is a closer symmetry equivalent instance of the second atom selected, then the distance will be shown together with the symmetry operator required to generate the closer instance. A button will also appear, allowing the user to generate this instance of the second atom.
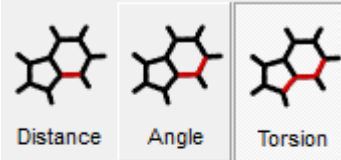
In Angle mode, select three atoms A1, A2 and A3. The distances A1···A2 and A3···A3 will be shown, together with the angle A1···A2···A3.

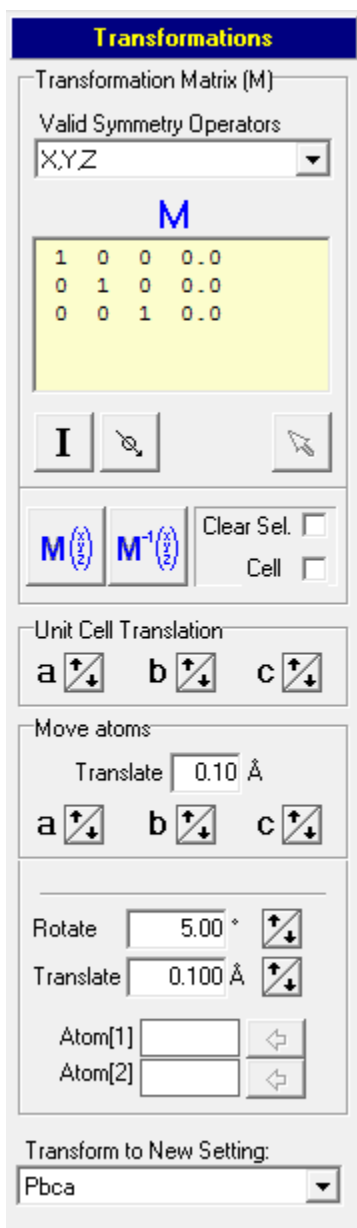In Torsion mode, select four atoms A1 to A4. The three distances A1···A2, A2···A3 and A3···A4 will be calculated, as well as the angles A1···A2···A3, A2···A3···A4, and the torsion angle A1···A2···A3···A4. The height of A4 above the plane defined by A1, A2 and A3 will also be given.

4.8 Transformations

Use this control panel to transform the positional coordinates of *selected* asymmetric unit atoms. When selected atoms are transformed, they are moved and not regenerated in the new positions as described in the **Section 4.4** on 'growing' the structure.

Transformation of an atom's coordinates entails multiplication of the coordinate vector by a 3x3 matrix, followed by translation. The transformation matrix **M** to be applied is represented by the 4x3 matrix shown on the control panel. The first three columns represent the 3x3 matrix mentioned above and the 4th column represents the translation vector. The matrix can be edited manually (much caution needed here) or generated from a valid symmetry operator of the current space group. If you choose a symmetry operator from the dropdown list, the matrix will be updated immediately to reflect the chosen symmetry transformation. Shortcut buttons are also available to reset the matrix to the identity matrix, or to generate a matrix that will invert the structure. Using the buttons with the blue labels, you can apply either the transformation **M**, or its inverse, to *the selected atoms*. The model will be updated immediately. You can also set whether you would like the selection to be cleared after a transformation has been performed (Clear Sel.). If Cell is checked then the transformation will apply to the unit cell. In addition to symmetry transformations, you can also perform simple unit cell translations on the *selected* atoms using the set of three up/down buttons. Inversion of the structure is possible even if the space group is non-centrosymmetric since the need for doing this may well arise from time to time. *X-Seed* will follow your instructions faithfully and assume that you know what you are doing. You can use this feature of *X-Seed* to positions any asymmetric unit atoms exactly where you want them - this may be useful when your asymmetric unit consists of fragments that are far apart, or if the atoms or fragments are far from the origin.

Using the controls in the Move Atoms panel, selected atoms can be nudged by a set distance in the direction of any of the unit cell axes. Alternatively, you can specify a vector by selecting two atoms and pressing the left-arrow button after each selection to define Atom(1) and Atom(2). Thereafter, and selected atoms can be either rotated around this vector by a set angle, or translated in the direction of the vector by a set distance.

Transformation using the mouse: when the Transformations control panel is active, it is possible to move selected atoms around using the mouse. This can only be done when *no symmetry generated atoms are present*. Select atoms that you would like to move, hold down the Shift and Ctrl keys and use the left mouse button down to drag the selected atoms in the plane of the screen. Ctrl-drag rotates the selected atoms. This feature is useful for 'cleaning' up the geometry of a disordered fragment before further refinement (e.g., if an atom is too far from its supposed position for **SHELXL** to place it at an electron density peak).

4.9 Animate
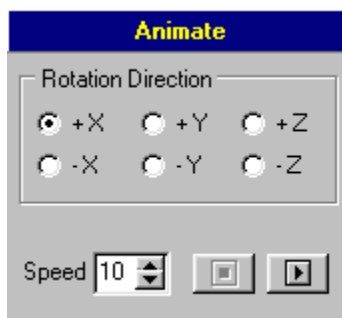
This is a relatively useless feature but it was easy to implement. In fact, there is no shortcut tool button for this control and you have to activate it from the Display menu. I suppose you could use this feature to set your model spinning whilst you sit back and contemplate your structure. The controls should be fairly self-evident

4.10 Restrain
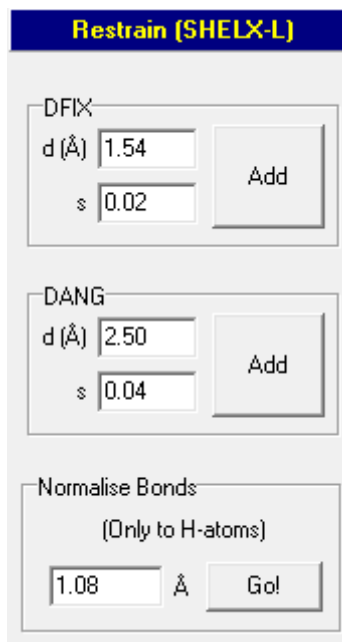
This control panel is activated via the Edit | Constrain menu and it allows you to add **DFIX** and **DANG** restraints to the model for the purposes of refinement with **SHELXL.** Select bonds and then press the Add button. **DFIX** or **DANG** instructions with the currently set d and s parameters will be added to the next **INS** file. Bond selection is automatically cleared when you press the Add button. Appropriate **EQIV** instructions will be generated automatically, if needed.

   Tip: bonds need to be selected in order to implement **DFIX** or **DANG** restraints. If you wish to apply these restraints to atoms that are not bonded, you can temporarily bind them using the Add/Remove Bonds control panel. Then you can select the appropriate bonds and apply the restraints. Thereafter, just delete the bonds again.

   You can also use this control panel to normalize bond distances to hydrogen atoms. The bonds to be normalized must be selected for this feature to work. Pressing Go! will move the hydrogen atoms of the selected bonds along the X-H vector such that the distance is adjusted to the desired value. This feature is useful for running programs such as *Opix*, which typically require hydrogen atoms to be in more realistic positions than those produced by refinement against X-ray data.

4.11 CSD Browser
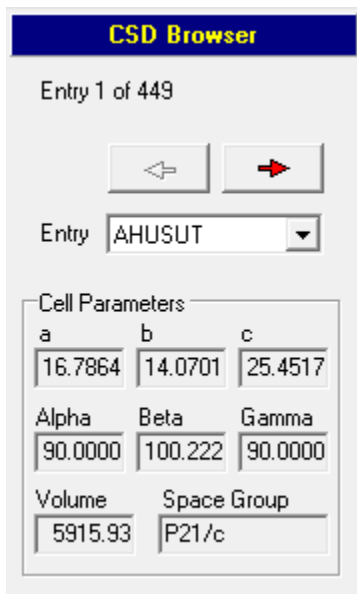
A **CSD** 'Legacy' **DAT** file usually contains several structures. When such a file has been read (see File under the Main Menu topic), this control panel is activated. You can browse through the structures using the buttons with the red arrows, or by selecting a particular entry from the dropdown list box which contains the **CSD** REFCODEs of all the structures. If a particular entry does not contain any atoms, a message to this effect will be displayed in the model display area. The unit cell parameters, cell volume and space group of the current entry are displayed on the panel. Each time a structure entry is loaded, **X-Seed** determines which atoms belong to the asymmetric unit and which are symmetry-generated, and then displays all the atoms contained in the current entry. **X-Seed** will also attempt to determine atom site occupancies and the molecular formula of the entry (taking Z = 1). The current entry can be exported in any of the supported formats using the File | Export Structure menu option.

Once loaded, the structure can be manipulated in much the same manner as a **SHELX** structure (i.e. you can change the connectivity, delete atoms, extend the structure, make **POV-Ray** images, compute XRD powder patterns using **Lazy Pulverix**, etc.). Note that any modifications made to the structure will **not** be retained when a new REFCODE entry is loaded.

4.12 POV-Ray

X-Seed's POV-Ray-related features are quite extensive. The present section only briefly explains how to set the controls and what they mean. It is more constructive to make you aware of the philosophy of operation of **X-Seed** (particularly that atoms or bonds need to be selected before settings are applied to them by pressing an Apply button) and for you to then explore the different options available by yourself. The POV-Ray control panel consists of four pages, each with many different, and sometimes non-intuitive controls.

You can start rendering by pressing the Render button, **or** the title bar of this control panel **or** Ctrl-R even if the **POV-Ray** control panel is not active. If the Quick render option is checked, the image will be rendered one quarter of the specified size, and with all the time-consuming high-quality settings (i.e. antialiasing, shadows, metallic) turned off. The Quick option is useful for rapid checking of placement of the model with respect to the image borders without having to adjust the high quality settings desired for the final image.

4.12.1 Image Tab
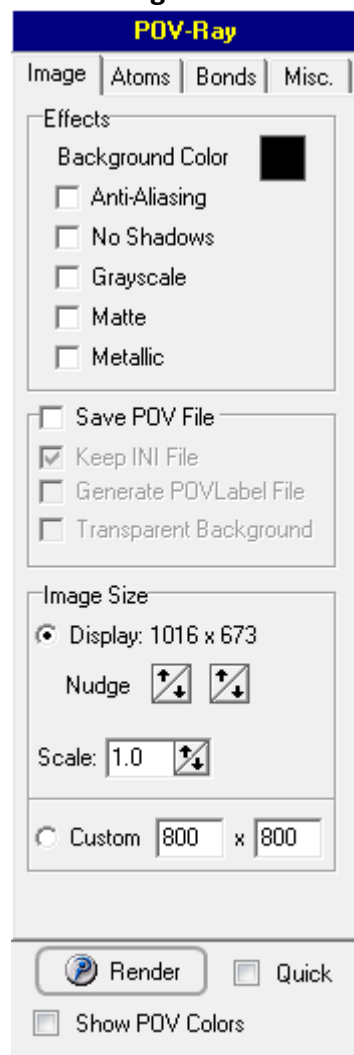
Here you can control several important aspects of the rendered image. Keep Antialiasing switched off until you are ready to produce the final high-quality version. Antialiasing will vastly improve the quality of the image, but the price to pay for this is time. By default, antialiasing is turned off. **POV-Ray** does not use the background color setting of the model display window. Click on the Background Color color indicator box to choose any background color you like for your image. Additional image effects include a Matte setting (to produce non-shiny objects) and No Shadows to suppress the generation of 'realistic' shadows in the image. A Metallic setting is included to give the image a metallic look, but this is generally not too useful for images destined for publication.

By default, the rendered image is *not* saved to disk. Most often, you will probably end up changing and re-rendering the image several times before you are satisfied with the view. When you are ready to produce the final image, check the box marked Save POV File. The next time you render the image, you will be prompted for a file name and the image will be saved in **PNG** format (for Portable Network Graphics). If Keep INI File is checked then both the **POV** and **INI POV-Ray** files will be retained. This is very useful if you'd like to render the image again in future without recreating it in **X-Seed** (e.g. to change the images size, etc.). If you are intending to produce an image with atomic labels embedded, check the box marked Generate POVLabel File. This will save the atom labels and their coordinates on the image – this file, together with the image file can be read into the separate program **POVLabel** to allow you to place the labels where you prefer them to be. If you check Transparent Background the image will have no background color. This is very useful for placing molecular graphic images in **PowerPoint** presentations where the background color might not be the same as that of your image, had you chosen a fixed-color background.

The ray traced image can have any dimensions (in pixels) that you like. By default, the image produced by **POV-Ray** will have the same dimensions of the model display window of **X-Seed**. However, you can override these dimensions by entering your preferred values (in pixels) for the horizontal and vertical dimensions. Alternatively, you can use the same aspect ratio of the **X-Seed** model display window, but scaled either up or down. If you would prefer the model display window to have very specific dimensions, you can use the up/down spin buttons to nudge the dimensions of the **X-Seed** window.

More on POVLabel: If you wish to create an image that can be labeled, save the image as a bitmap and make sure that the POVLabel File option is checked. This will instruct **X-Seed** to also write a file called **name.LBL** to accompany **name.BMP** written by **POV-Ray**. The utility program **POVLabel.exe** (part of the **X-Seed** program suite) can then be used to read **name.LBL** and

allows you customize the positions and text properties of the atomic labels. See the **Chapter 9** on using *POVLabel* for details.

Note: if your custom dimensions do not have the same aspect ratio of the model display area, then *X-Seed* uses only the horizontal size of the model display area to determine the scale of the *POV-Ray* image.

4.12.2 Atoms Tab

IMPORTANT: if you wish to change how an atom is portrayed by *POV-Ray*, you must (1) customize the atom settings on the control panel, (2) make sure the atom is selected and (3) click on the Apply button.

When you press the Apply button, **all** of the checked settings will apply to the atom. Check Clear Selection to clear the atom selection each time the Apply button is pressed. If you wish to leave a certain setting of the atom unchanged, make sure that none of the settings in this category are checked. Any atom can be assigned any color. The CPK color of the atom is taken to be the color used for that element by *X-Seed*. These colors can be changed using the Element Settings dialog. Alternatively, you can apply a custom color to selected atoms. Click on the custom color indicator box to select the color to be applied. It is also possible to use predefined textures for atoms. These textures can be selected from the Texture dropdown list. The textures in this list are extracted from the file *textures.inc* that is supplied with *X-Seed*. If you wish, you can add or remove textures from this file. By default, atoms are shown as spheres of radius 0.2 times that of their van der Waals radii (0.15 for hydrogen atoms). The van der Waals radii used for the different elements can also be changed in the Element Settings dialog. In addition, you can set a fixed radius. Three shortcut buttons are available to quickly set the radii for stick, ball-and-stick and spacefilled representations. These buttons only change the settings - you still have to *apply* these settings to the selected atoms. An example of each type of representation is given below.
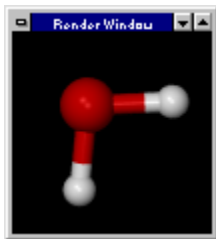
 When only one atom is selected, this button will appear. The button allows you to change the atom settings on the control panel to correspond to those of the selected atom.

 Stick representation. If an atom has been assigned a radius of zero, it will be shown with the same radius as the thickest bond to it. Thus terminal bonds are shown with rounded rather than flat ends.

 Ball-and-stick representation. Individual atom and bond radii can be set to any value.

 Space filled representation.

 Anisotropic atoms can also be shown as thermal ellipsoids. In order to do this, check the Ellipsoids setting in the Display as panel. The Radius panel will be replaced by controls that allow you to set various options relating to how ellipsoids are shown. The probability factor (default = 50%) can be selected from a dropdown list. There are several effects that can be applied to ellipsoids. These are best illustrated by the examples shown below, but you are encouraged to experiment with different settings yourself. By default, an ellipsoid's interior color is the same as its CPK color.

In all the following examples, a custom interior color (i.e. white) has been set.

 An ellipsoid with no special effects.

 Octant cutout only.

Slices only (default setting for all ellipsoids).

Slices + octant cutout.

Slices + octant cutout + octant border.

Octant cutout + axes.

Octant cutout + slices + axes.

Octant cutout + axes + hatching.

As above, with slices and octant border.

Octant cutout + tori.

As above with axes and hatching.

Special note: A sliced ellipsoid with no border cannot be hatched.

4.12.3 Bonds Tab
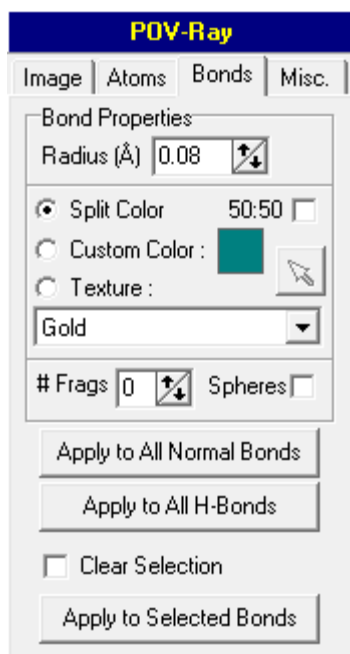
Important: in general, if you wish to change how a bond is portrayed by **POV-Ray**, you must (1) customize the bond settings on the control panel, (2) make sure the bond is selected and (3) click on the Apply button. However, for convenience, buttons are also available to apply the current settings to all normal or all hydrogen bonds, regardless of which are selected.

Check Clear Selection to clear the bond selection each time the Apply button is pressed. Any bond can be assigned any combination of radius, color and fragmentation settings. If a bond is assigned to have split colors, the color of the bond will depend on the colors of the two atoms involved. If the two atoms do not have the same color, by default, the bond will be split into two colors with the position of the color division being a proportional to the bonding radii of the atoms. However, you can use the 50:50 option to force the color split to occur midway between the two atom positions. Any bond can be shown as fragments, and up to 30 equally-spaced fragments are allowed. By default, the bond fragments are sliced cylinders. However, the fragments can also be represented as spheres.

When only one bond is selected, this button will appear. The button allows you to change the bond settings on the control panel to correspond to those of the selected bond.

4.12.4 Misc. Tab

This page contains miscellaneous **POV-Ray** settings. If you have created centroids in the structure, these will only be shown if Show Centroids as Spheres is checked. Thus, by default, bonds that have been added between atoms and centroids will be shown, but not the centroids themselves. However, if you would like to have the centroids displayed, you can use this option to turn them on.

The Atom Transparency setting applies to the transparency level of any atoms for which you have chosen to overlay semi-transparent van der Waals spheres. Of course, this setting is only effective if the primary settings for the atoms involve showing them as smaller than their van der Waals surfaces (e.g. atoms displayed in stick, ball-and-stick or thermal ellipsoid mode).

Several tabbed pages are available to control various settings. The Connolly 1 and Connolly 2 pages control the settings for the display of up to two surfaces generated using **MSRoll**. The surface file needs to be specified (either **name.CAV** for an internal surface or **name.VET** for an external surface). The texture, color and transparency can be set for each surface. While smooth surfaces are more visually appealing, there is an option to show the surface as triangles. The surface can also be color coded according to the closest atoms, but this feature is almost useless.

The way that the unit cell is rendered can also be customized. By default, the unit cell color will be the same as that used in the display (see Display Settings control panel). However, it is possible to use a predefined texture for the unit cell edges. The radii of the edges can also be set.
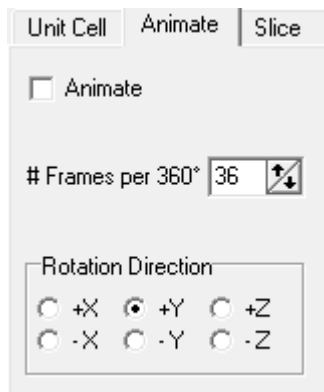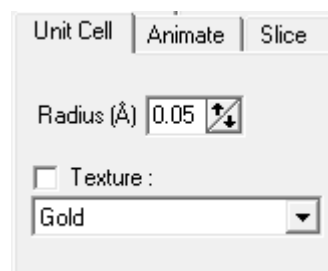
Frames for animations can be created using the Animate option. An animation consists of a full 360° rotation. Select the axis about which the animation will take place, and the number of images per rotation. When you press the Render button, **POV-Ray** will render all the images of the animation as sequentially numbered **PNG** files. Use **PNG2AVI** (from the Interface menu) to create an **AVI** file from the **PNG** images. The **AVI** file might be quite large – however, reading and saving it using a program such as **FreemakeVLC** reduces the file size dramatically.
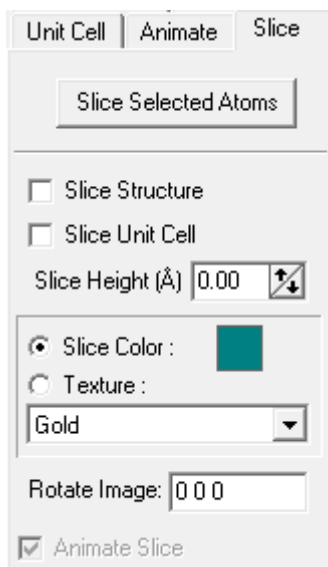
It is possible to show a section through selected atoms of the structure in the rendered image by checking the Slice Structure checkbox. First select the atoms to be sliced and press Slice Selected Atoms and then Render. The user can choose to also slice the unit cell edges, if they are displayed. By default, the slice is through the origin of the scene. However, the height of the slice in the projection of the **X-Seed** model display window can be changed. The slice can be colored or textured, and the image can be rotated *after* slicing. To specify the rotation after slicing, enter rotation angles in degrees about the *x*, *y* and *z* axes. The slice can also be incorporated into an animation.

A maximum of four light sources can be used to illuminate the scene. Each light source can be defined as one of two types, and each can also be assigned a number of settings. A relative light source is specified relative to the 'camera' (i.e. the point of view of the observer).

If Relative is selected for a particular light source, the three values in Position refer to rotation angles in degrees. Rotation angles of 0 0 0 refer to the position of the viewer. Values of -20 -20 0 will specify rotation of the light by these angles about the *x*, *y* and *z* axes, respectively. By default, the light source will be aimed at the origin of the image, but this can be changed.
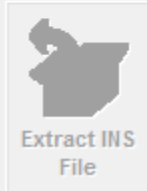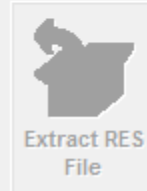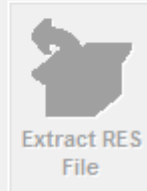
If Absolute is selected, the three Position values specify the location of the light source in the Cartesian coordinates of the scene. A certain amount of guess-work by the user will be involved. Generally, the default light settings provide adequate illumination of the scene. The user can also specify the intensity of each light source, as well as whether or not it casts a shadow.
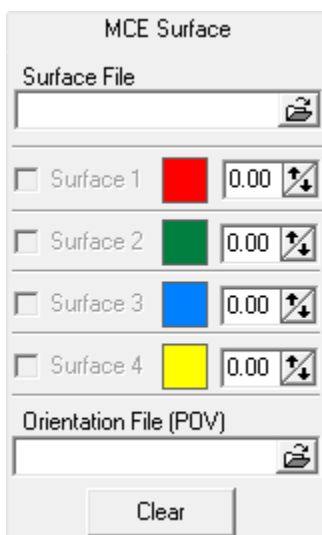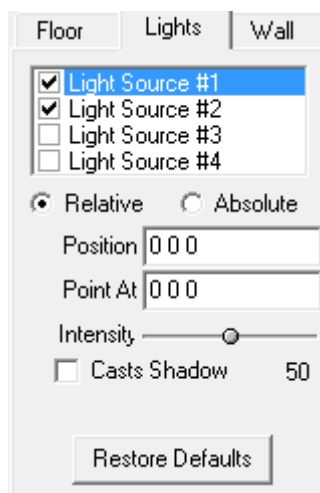
If you have produced difference electron density maps using Marching Cubes via Crystals (*.wrl file), then you can read and render them. This feature is still experimental.

The Floor and Wall options are of limited value for scientific illustration and will not be discussed further.

4.13 SHELX History

SHELX History

SHELXS Summary

Solution: Direct Methods

# Reflections	12673
# Rejected	986
R_{int}	0.0315
R_σ	0.0217
# Unique	2254
# Observed	1847
% Observed	82.0
Max h	10
Max k	12
Max l	30
Max 2θ	54.29

Run # 1 of 6

Autoload
- ○ None
- ○ INS   ⊙ RES

Extract INS File   Extract RES File

SHELX History

Final SHELXL Refinement

	R1	# Data
Fo > 4 (Fo)	0.0349	1734
All Data	0.0520	2253
After Merging	0.0515	2253

wR2	0.0947
GooF	1.022
Restr. GooF	1.022
Highest Peak	0.24
Deepest Hole	-0.15
# Parameters	118

Run # 6 of 6

Autoload
- ○ None
- ○ INS   ⊙ RES

Extract INS File   Extract RES File

As you proceed through structure solution and subsequent refinement, every **SHELX INS**, **RES** and **LST** file is logged within a file named **name.LOGX**, along with the screenout of the **SHELX** process. This dialog box allows you to browse through the **LOGX** file using the Run # control. The relevant solution or refinement parameters are summarized as you scroll through the history. By default, the None Autoload radio control is active, which means that only the parameters from the **LST** file will be listed. You can press the Extract INS File or Extract RES File buttons to extract and display the relevant files from the **LOGX** file, and you can then continue working on that particular version of the model. Using the example shown on the left, if you back-track to the RES file of Run # 3 of 6 and then continue refining, the next run will become Run # 7, and all previous runs will still be available for extraction. If either the INS or RES Autoload radio control is active, then scrolling through the history will automatically extract and load the relevant **SHELX** file for GUI editing. This SHELX History feature of **X-Seed** allows you to easily revert to an earlier version of your model and it is useful to either review the progress of the **SHELX** process, or to revert to an earlier version in case something went wrong.

5. Dialogs

Much of the functionality of X-Seed is accessed *via* numerous dialog boxes. The most important of these are described below.

5.1 Element settings
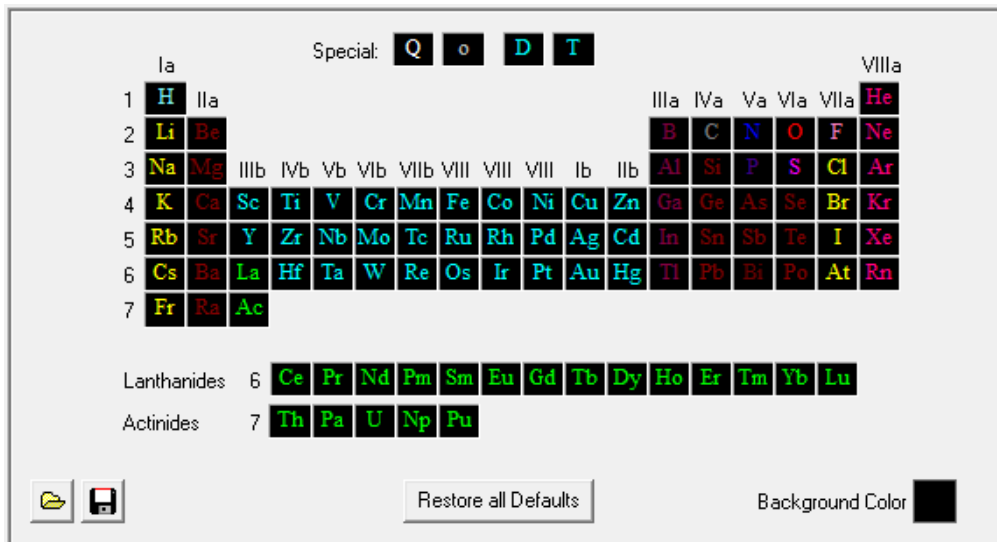
The Element Settings dialog is used to customize the display colors and van der Waals radii of the different elements. The first 95 elements, as well as deuterium, tritium, "Q" peaks and centroids are available for modification.

Click on an element in the periodic table to load its current settings into the lower panel:

Here the van der Waals radius and color can be set for each element type. Its bonding radius is shown, but cannot be altered since X-Seed uses the default bond radii used by SHELX to determine connectivity. A shortcut method of changing the color associated with a particular element is to double-click on its entry in the periodic table. This activates the color selection dialog directly.

Other controls on this dialog are described below.

Restore all Defaults — **X-Seed** can generate a set of default parameters for the elements. Use this button to generate these default values for *all* the elements.

Restore Atom Defaults — Re-generates the default values for the currently selected element *only*.

💾 Save the current element settings to disk.

📂 Retrieve a previously saved set of element settings.

You can save any number of sets of element preferences to disk for later use. When you exit **X-Seed**, the program automatically saves the current element settings to a file called "***last.elt***". The next time **X-Seed** is executed it loads and uses this set of element parameters. The Element Settings dialog is also the only facility that **X-Seed** has for changing the background color of the model display area

5.2 Crystal Data

Once a **SHELX** INS or RES file is loaded, this dialog is activated by selecting "Edit | Crystal Data" from the main menu, or by pressing the Crystal Data button on the ribbon. The relevant crystal

data parameters extracted from the **SHELX** file are displayed. The fields in this dialog should be largely self-evident, and only fields shown in black text are editable. Changing the current space group is as simple as typing in the space group symbol - see **Section 2.7** on Space Groups for details of space group notation. In accordance with the requirements of **SHELX**, the origin of a centrosymmetric space group *must* lie on a center of symmetry. As long as they meet this requirement, all settings of the 230 space groups are supported. The molecular formula represents the molecular entity that you regard as being the formula unit - not necessarily the entire contents of the unit cell (unless Z = 1). When you change the formula or Z, the "Calculated Density" field will be updated. If the calculated density is less than 0.8 or greater than 3.0, the density field will be shown in red to indicate a possible error in either Z or the molecular formula. If the NEUT checkbox is checked, then it is assumed that neutron diffraction data are available, and the NEUT command will appear in the **SHELX** instruction files.

5.3 Program Settings

The Program Settings dialog is activated *via* the **Preferences | Program Settings** menu selection. Currently, this dialog contains four pages, and all of these settings are saved to the registry when you exit **X-Seed**.
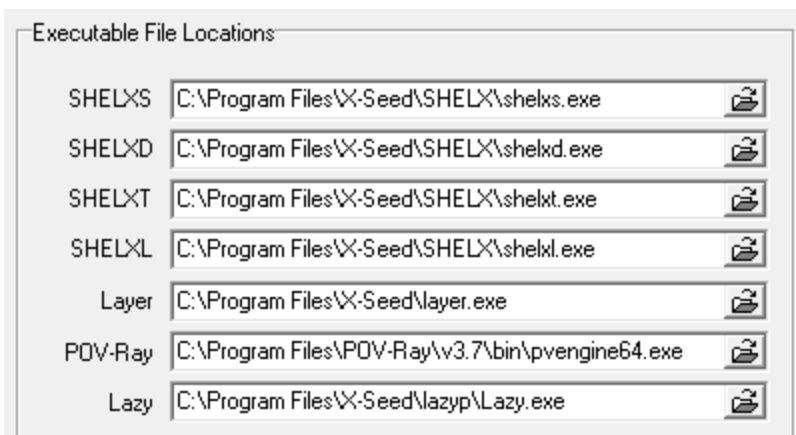
5.3.1 File Paths

X-Seed can execute several **DOS** and Windows programs automatically. Use this dialog to tell **X-Seed** where their program executable files are located. These settings will be saved when you exit **X-Seed**.

Note: the **POV-Ray** executable file is usually called "*pvengine.exe*" and is located in the "**bin**" subfolder of the **POV-Ray** installation folder.

5.3.2 Adding Hydrogens

	mn	U	Description
	13	-1.2	Tertiary C-H
	23	-1.2	Secondary C-H2
	33	-1.5	Staggered C-H3 Group
	43	-1.2	Aromatic C-H or N-H
	93	-1.2	Terminal X=CH2 or X+NH2+
	123	-1.5	Disordered Methyl Group (2 pos
	137	-1.5	Methyl Group (Max. e-Density)
	153	-1.2	BH Group (B coord. # = 4 or 5)
	163	-1.2	Acetylenic C-H (X-C-H Linear)

This is where you can customize the form of the **SHELXL HFIX** instruction cards for hydrogen atom generation (when you use the Generate H-Atoms (HFIX) control panel). The default values should be adequate for most purposes, but if you know what you're doing you can alter the settings. Consult the **SHELXL** instruction manual for the meaning of the **HFIX** parameters - see HFIX and **AFIX**. The description field allows you to specify the text in the hint box that pops up when the mouse cursor is over the relevant button in the Generate H-Atoms (**HFIX**) control panel.

5.3.3 Miscellaneous Settings

Residual Peaks

Residual peaks are defined such that [A--P] < [rA + rP - n]

where   rA = Bonding radius of Atom A,

rP = Bonding radius of Peak P, and

n = $\boxed{0.5}$ Å

This panel allows you to fine-tune **X-Seed's** definition of what constitutes a "residual peak". Residual peaks are peaks of electron density that are most likely not indicative of atoms that have to be added to the model. These peaks appear at relatively short distances from established atoms of the model,

especially "heavy" atoms. You can delete such peaks manually, or save time by selecting "All Residual Peaks" from the "Delete" menu. Only peaks that meet the criteria set out in this panel will be removed.

When **X-Seed** generates H-bonds automatically at your request, it uses the criteria laid out in this panel. Only elements that are listed in the "Valid Donor / Acceptor" field are considered during automatic hydrogen bond generation (although you can *manually* add H-bonds between any two atoms - see Add/Remove Bonds). You can add or remove any elements to/from this list. The element symbols in this field *must* be separated by spaces. You can also set the distance criteria for hydrogen bond generation as outlined in the panel. The "Use H-Bonds ... " checkbox can be checked if you wish hydrogen-bonded connectivity to be considered when **X-Seed** decided what constitutes a fragment.

Here you can also control a number of other miscellaneous settings. Some of these settings are self-explanatory. A default temperature can be specified if the SHELX file does not contain a `TEMP` instruction.

5.3.4 POV-Ray

Here you can specify default settings for the POV-Ray interface. The defaults should be sufficient for most purposes, but you can experiment with these settings if you prefer different settings.

5.4 Undelete Atoms

When asymmetric unit atoms are deleted, they can still be restored - until the next **SHELX** run has been accepted. Use this dialog (activated *via* the "Delete | Undelete Atoms" menu) to restore any asymmetric unit atoms that have been deleted. To undelete an atom, make sure that the checkbox next to its name is checked when you click on the OK button. Time-saving buttons are available to select, unselect and toggle selection of the atoms in the list. Symmetry-generated atoms, when deleted, are not retained for "undeletion" since it is possible to re-generate these atoms from the space-group and asymmetric unit atom list information

5.5 The SHELXS Dialog

This dialog provides an interface to structure solution by means of **SHELXS** and is activated *via* the "**Interface | SHELXS**" menu selection. The dialog has two tabbed pages – one for an editable INS file and the other for shortcuts to valid SHELX commands. The dialog also contains a number of buttons:
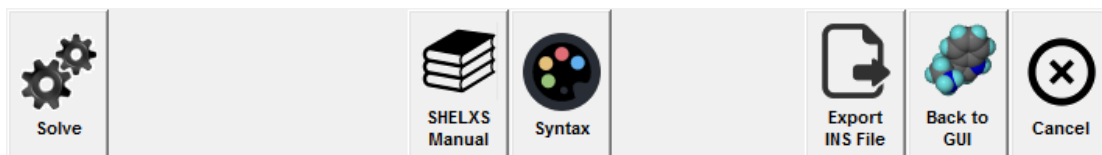
Solve: launch SHELXS using the current contents of the edit window as input;

SHELXS Manual: Launch your default web browser and load the online SHELXS manual;

Syntax: After editing the instructions, color the various commands by type;

Export INS File: write the contents of the current edit window to disk;

Back to GUI: return to the GUI, but retain any edits. Here it is important to understand that pressing this button causes **X-Seed** to clear the structure from its memory and to reload it from the edit window as if loading it from a file;

Cancel: return to the GUI, discard any changes made to the edit window. If any changes had been made, you will be warned that these will be discarded.

5.5.1 SHELXS Instructions

The edit window (yellow background) shows the commands that will be parsed to **SHELXS**. You can either edit these commands manually, or you can use the "XS Shortcuts" tabbed page. Changes are preserved as you switch back and forth between the two pages. When the instruction set is ready, you can launch **SHELXS** by pressing the Solve button.

5.5.2 XS Shortcuts

This tabbed page shows all of the **SHELXS** commands that are available. A command will be inserted if the relevant box is checked, and the command parameters will be used as shown. This page includes shortcut buttons for the three different types of structure solution protocols that **SHELXS** can implement (Direct methods, Patterson Function and Partial Structure Expansion). With the exception of "SHELXS Manual", all of the buttons at the bottom of the dialog as hidden when this tabbed page is visible – so that you can only launch **SHELXS** or return to the GUI when the instruction set is visible.

5.5.3 Solve Structure

When the Solve button is pressed, **X-Seed** writes the contents of the edit window to *name*.INS and launches **SHELXS**. The output of **SHELXS** is parsed to the edit window. When **SHELXS** completes its tasks, the user is presented with button to either accept or reject the results. Salient parameters are extracted from the LST file and displayed in the "Summary" tabbed sheet.

5.5.4 Summary
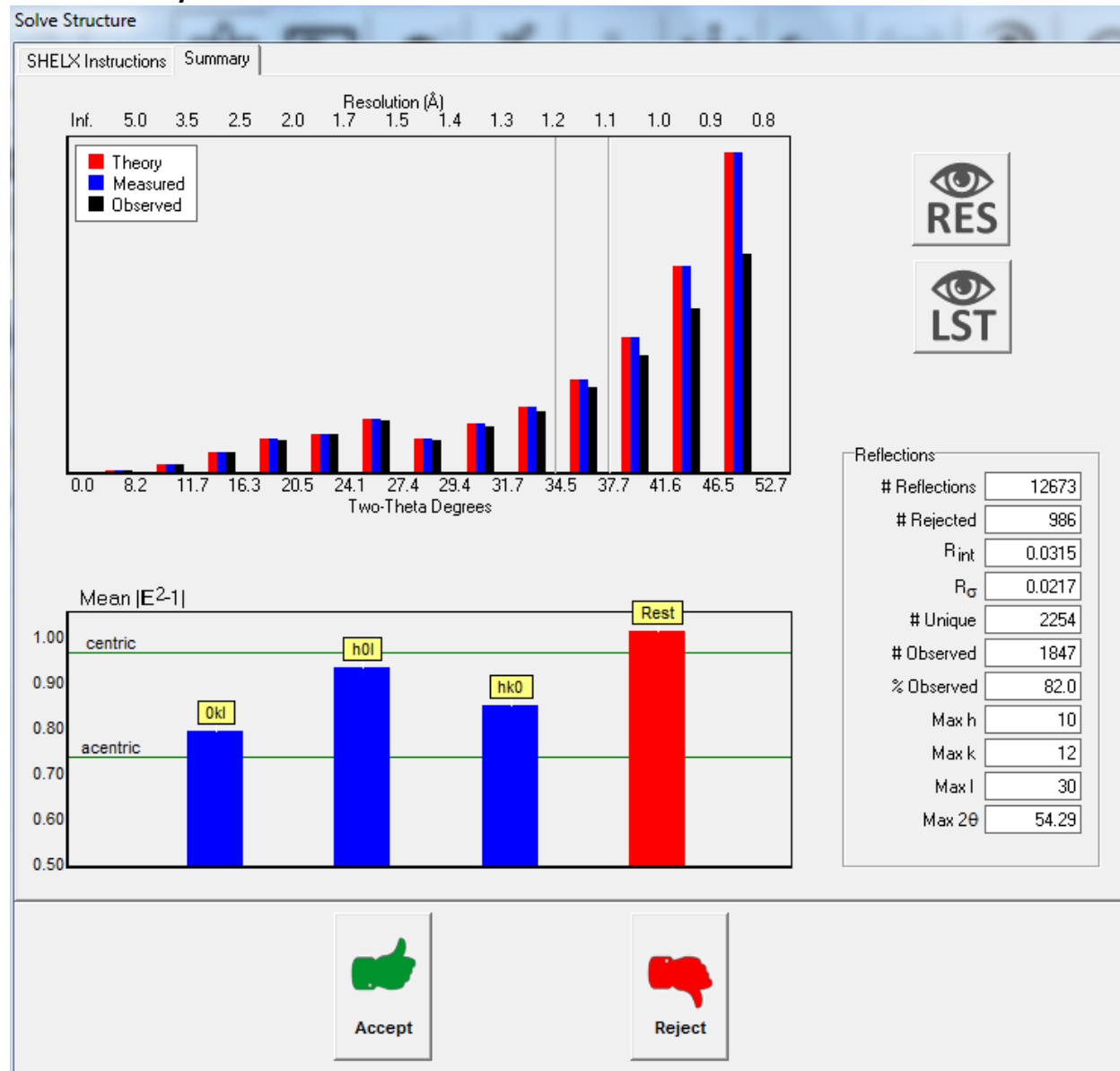
In addition to various important parameters (on the right), this tabbed sheet shows bar charts of number of reflections vs resolution, and "E statistics". The RES and LST buttons allow the user to load the **SHELXS** output files into Notepad for closer inspection.

If the user presses "Accept", **X-Seed** clears the current structure and loads the new RES file. If you press "Reject", **X-Seed** reverts to the previous model.

5.6 SHELXT
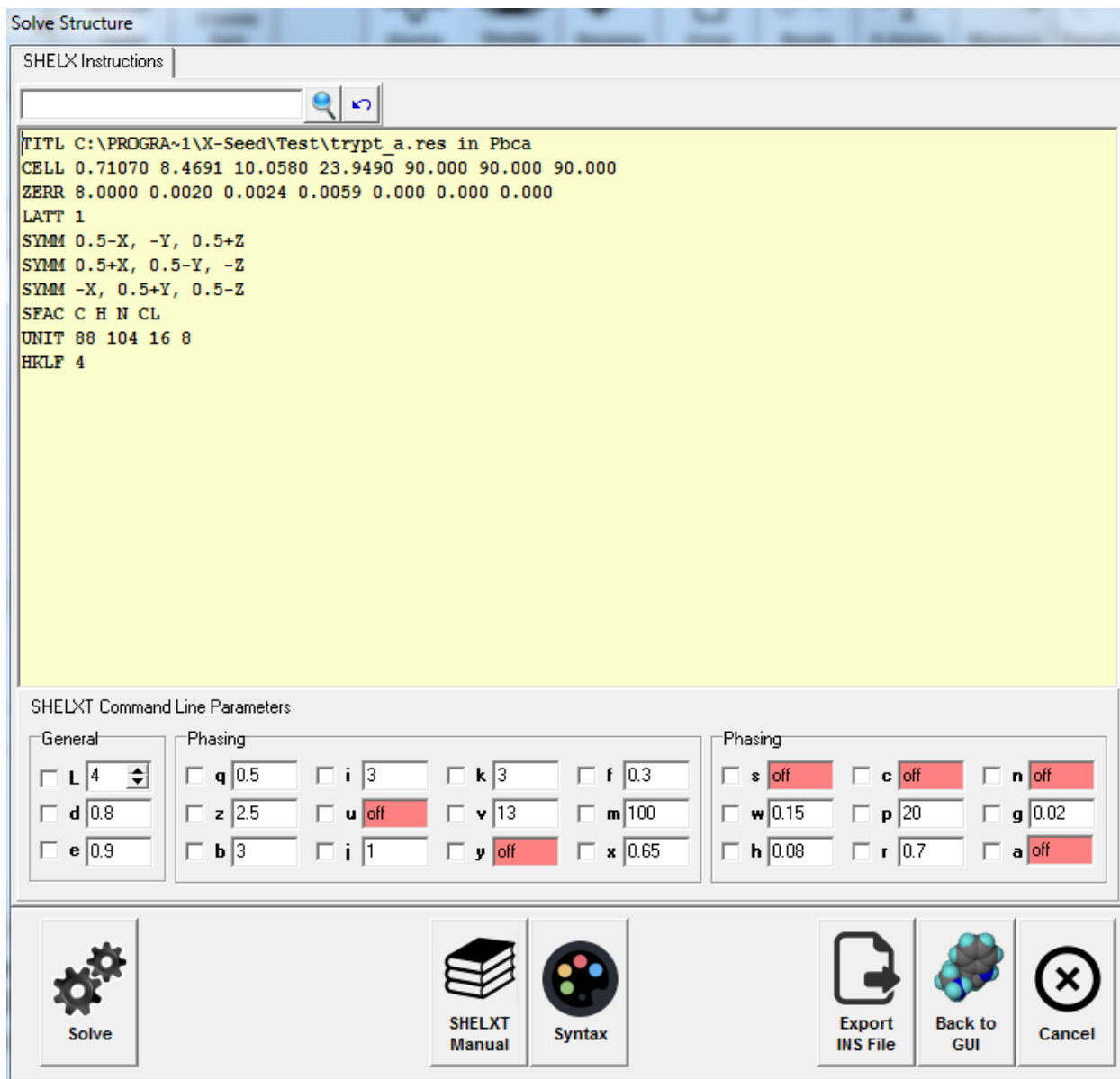
The **SHEXT** dialog box is similar to that for **SHELXS**, except that only one tabbed page is provided. All of the command-line parameters that are used by **SHELXT** are accessible.

Upon successful completion, **SHELXT** might have generated multiple solutions. These will be named *name_a*.RES, *name_b*.RES, etc, and corresponding *name_a*.HKL, *name_b*.HKL, etc. files will also have been generated. By default, **X-Seed** loads the file *name_a*.RES when the Accept button is pressed. The user should evaluate whether or not this is the most appropriate solution to select. Other solutions can be selected by simply opening the files using "File | Open".

5.7 SHELXD

```
Solve Structure

SHELX Instructions | XD Shortcuts

                              🔍 ↩

TITL pn1a in P2(1)
CELL 1.54178 15.0000 19.8000 16.5000 90.000 113.400 90.000
ZERR 2.00 0.0030 0.0040 0.0033 0.000 0.030 0.000
LATT -1
SYMM -X, 0.5+Y, -Z
SFAC C H N O S
UNIT 130 254 38 44 8
FIND 84
PLOP 112 141 157
MIND 1.0 3
NTRY 1000
HKLF 4
END
```

Solve | SHELXD Manual | Syntax | Export INS File | Back to GUI | Cancel

The **SHELXD** dialog is similar to that for **SHELXS**.

5.8 SHELXL

As above, the **SHELXL** dialog allows the user to manually edit the instruction set before launching **SHELXL**. One-off commands (e.g. `L.S.`, `PLAN`, `TEMP`, etc.) can either be typed or specified using the "XL Shortcuts" tabbed page. The "worst" reflections from the last **SHELXL** run (extracted from the latest LST file) are listed in a third tabbed page. Upon appropriate consideration, the user can check reflections to be omitted in the next run. Multiple-use commands such as `BIND`, `EXYZ`, etc. need to be typed into the edit window. Some of them may be generated automatically by **X-Seed** (e.g. if you force a bond between two atoms).

Note: X-Seed is compatible with almost all **SHELXL** instructions. Exceptions include **ANSC**, **ANSR**, **LAUE**, **RESI** and **SPEC**.

5.9 Lazy Pulverix
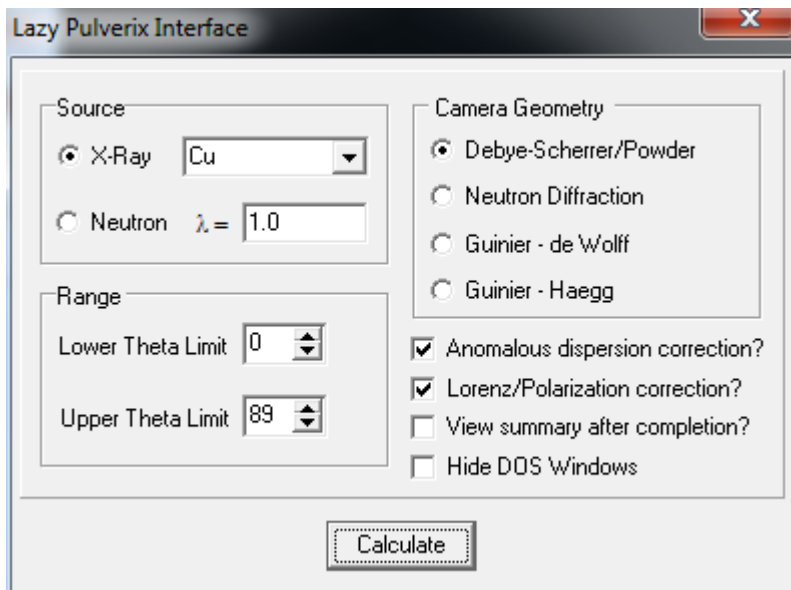
For a description of the **Lazy Pulverix** program suite, see the relevant topic in this help file. In order to function properly, the DOS programs **lazy.exe** and **pulv.exe** *must* be in the same folder (the install program puts them in a folder named "lazyp" in the **X-Seed** installation folder). If you wish to see the resulting powder pattern displayed after it has been calculated, the Windows program **xrdplot.exe** (part of the **X-Seed** package) must also be in the **Lazy Pulverix** folder. All the settings available to **Lazy Pulverix** can be set using this dialog. When you press the Calculate button, **X-Seed** will prepare and save a **LAZY** input file (in the structure's folder) and then execute the programs **LAZY** and **PULVER** in turn. When **PULVER** completes, **X-Seed** will *execute* the program **XRDPlot** and automatically load the calculated powder pattern trace. **XRDPlot** can be used to view and print the powder trace. If **XRDPlot** does not display a powder pattern, it means that the **Lazy Pulverix** run was unsuccessful.

5.10 About X-Seed
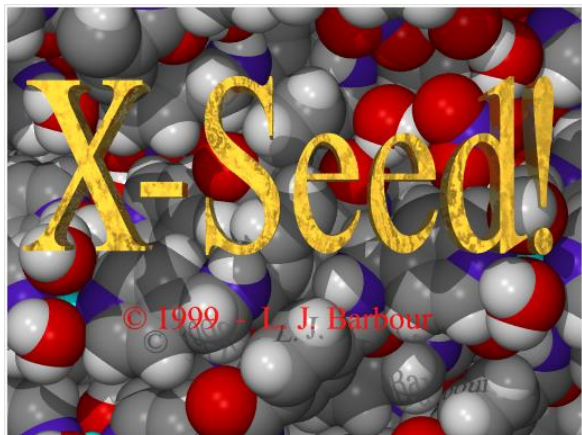
Show the *X-Seed* splash screen with credits and copyright information. When it is displayed, clicking anywhere on the splash form will close it.

5.11 CIF File

A **CIF** file can be opened using the File | Open menu, the Open button, or by dragging a **CIF** file onto the **X-Seed** window. Note that **X-Seed** does not load the crystallographic model from the **CIF** file. In fact, the current model in the **X-Seed** window remains there, and **X-Seed** simply loads some of the **CIF** file parameters into a dialog box, as shown below.

C:\Program Files\X-Seed\Test\trypt.cif

Crystal data for 1: $C_{11}H_{12}ClN_2$, $M = 207.68$, orthorhombic, space group $Pbca$ (No. 61), $a = 8.469(2)$, $b = 10.058(2)$, $c = 23.949(6)$ Å, $V = 2040.0(8)$ Å^3, $Z = 8$, $D_c = 1.352$ g cm^{-3}, $F_{000} = 872$, MoKα radiation, $\lambda = 0.7107$ Å, $T = 293(2)$ K, $2\theta_{max} = 54.3°$, 11682 reflections collected, 2253 unique ($R_{int} = 0.0328$). Final $GooF = 1.025$, $R1 = 0.0349$, $wR2 = 0.0862$, R indices based on 1734 reflections with $I > 2\sigma(I)$ (refinement on F^2), 118 parameters, 0 restraints. Lp and absorption corrections applied, $\mu = 0.334$ mm^{-1}.

RTF Table   RTF Report   Copy RTF   Extract RES and HKL   Back to GUI

Upon reading a *CIF* file, the dialog will automatically generate a *Rich Text* format summary of the structural and final refinement parameters. This summary can be copied to the Windows clipboard for pasting into a document. The formatted text is also written to a file *name_Report.RTF* for later retrieval. If you press the RTF Table button, a formatted table will be displayed and saved as *name_Table.RTF*. If the *CIF* file has been generated by *SHELXL*, it will also contain the *HKL* file and the final *RES* file. These files can be extracted using the Extract RES and HKL button. This feature is useful if you receive a *CIF* file from someone else and wish to check the refinement yourself.

5.12 MSRoll

If the program **MSRoll** is present in the **X-Seed** installation folder, the main menu will have the option Interface | MSRoll. **MSRoll** maps the internal and external surfaces available to either the surface (Contact Surface) or the center (Accessible Surface) of a spherical probe of a given radius. This is a simple dialog that serves as a convenient interface to **MSRoll**. You can specify the Probe Radius in Å, and whether you would like to evaluate either the Contact or the Accessible surface.
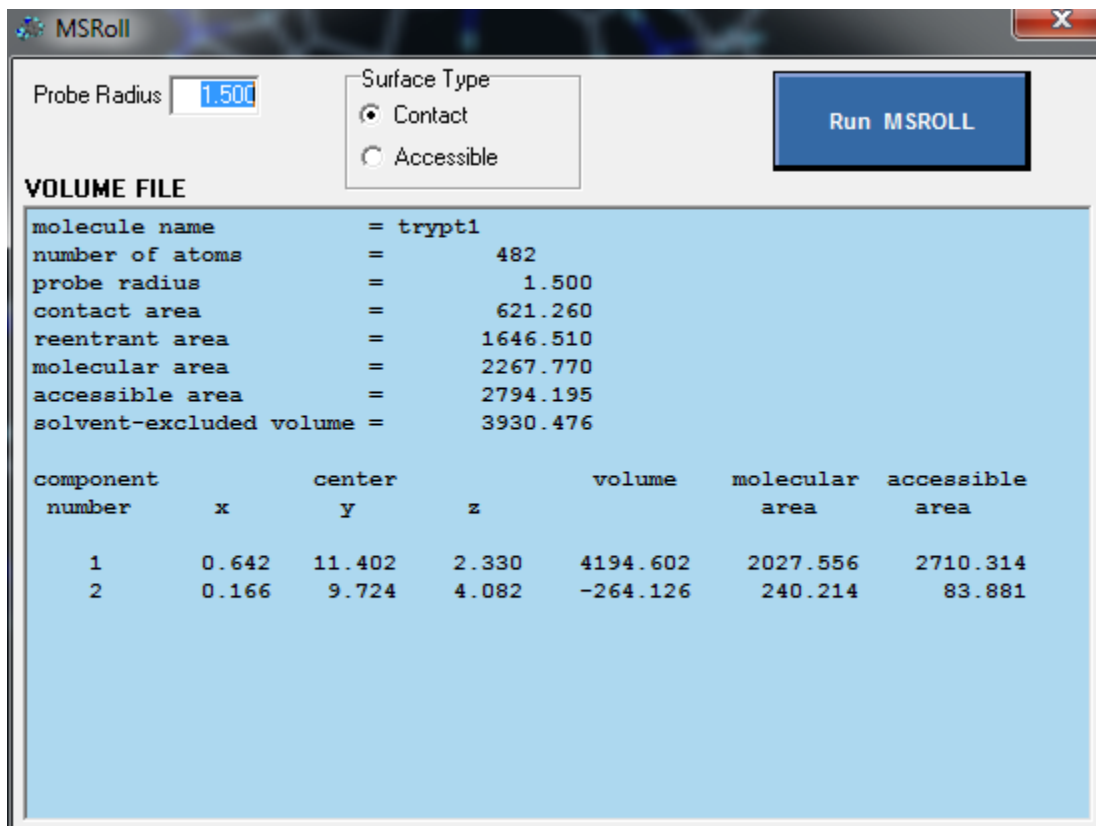
When you click the Run MSROLL button, **X-Seed** will export the *currently visible atoms* to a **PDB** file, as required by **MSRoll**. A **RAD** file will also be created – this file will supply **MSRoll** with van der Waals radii for all of the elements present, as specified in the Element Settings dialog

described in **Section 5.1**. *X-Seed* will then call *MSRoll* in a *Command* window by automatically parsing the *MSRoll* input files and desired Probe Radius.

Once *X-Seed* has detected that *MSRoll* has completed its task, the contents of the *MSRoll VOL* output file will be displayed in the dialog for easy inspection. All of the contiguous surfaces located by *MSRoll* will be listed as separate components, along with their centroids (in Cartesian space), volumes, areas and accessible areas. Positive values for volumes indicate exterior surfaces and negative values indicate interior surfaces (i.e. cavities). The exterior surfaces are written to *VET* files and the interior surfaces to *CAV* files. These files are generally very large and contain coordinates of triangles that define the surfaces. The surfaces generated by *MSRoll* can be incorporated into *POV-Ray* images (see **Section 4.12.4**).

Generating cavity surfaces for host-guest systems is relatively easy. For example, if you would like to show the space available to a solvent guest molecule, grow all molecules around the guest such that it is completely surrounded on all sides (i.e. the spherical probe will not 'fall out' of the cavity created by the host. Select the entire guest molecule (Ctrl-click), hide it (Ctrl-H), and then run *MSRoll*. Load the *CAV* file in the POV-Ray control panel and render. You can even unhide the guest molecule (Ctrl-U), specify a level of transparency for the *MSRoll* surface, and then render an image that shows the guest within the cavity.

Rendering channels requires more effort. Keep in mind that an unblocked channel will be experienced by the probe as part of the external surface since a probe placed in the channel will be able to 'drop out'. Here is a detailed example using a metallocycle published in *J. Am. Chem. Soc*. **2005**, *127*, 13134 (*CSD* code DAZFIU):

individual metallocycle   metallocycles packed along the *c* axis, forming a channel
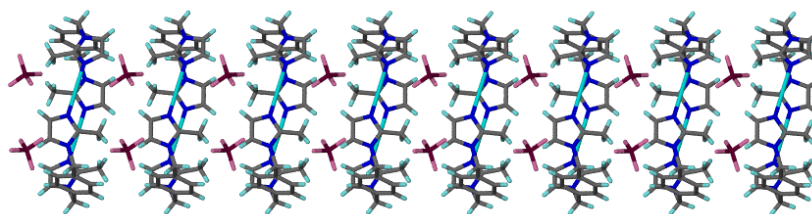
The metallocycles stack along the *c* axis, as shown above, to form an infinite channel. Using the stacked molecules as input to *MSRoll*, we obtain the following output:

We see that there is only one component, and that its volume is positive. This means that the probe 'fell out' of the channel and was thus able to also access the exterior surface of the stacked metallocycles. It therefore follows that, in order to visualize the channel, we need to block its ends. In this example, centroids were created for each of the two metallocycles at the extremities of

the stack: Ctrl-click on any atom belonging the metallocycle to select the entire metallocycle, and then press Ctrl-C to create the centroid. Add a large atom (e.g. iodine) to the molecular formula using the Crystal Data dialog (Section 5.2). Shift-click on one of the centroids to select both of them (since they have the same atom type), press F2 and select Iodine from the drop-down list. The centroids will 'become' iodine atoms that block the channel at both ends:

If you run **MSRoll** again, a negative volume will be reported for a component, indicating that an interior surface has been mapped.

Specify the **CAV** file in the **POV-Ray** control panel and render the figure again. It will look like this:
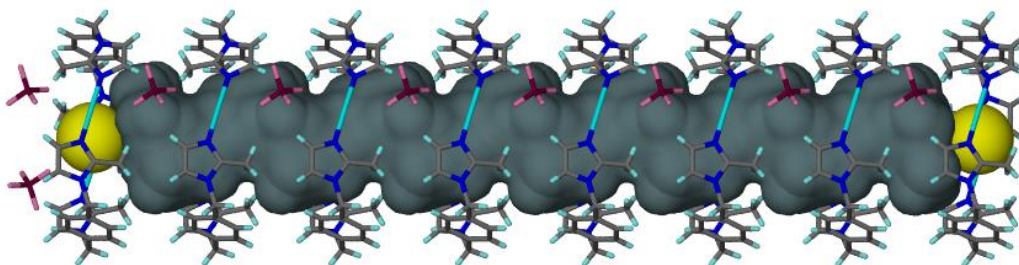
Clearly the discontinuities at the ends of the channels are not suitable for illustration, even if the iodine atoms are hidden. The solution is to crop the figure:
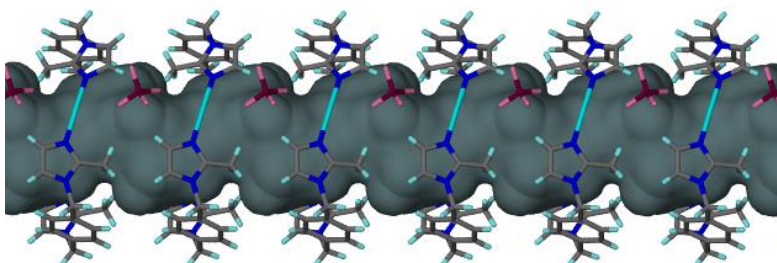
5.13 Cartesian -> Cell Coordinates
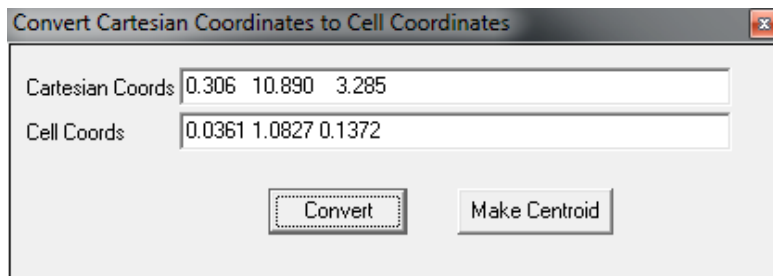
This is a simple dialog box that allows you to enter or paste a set of Cartesian coordinates (three numbers separated by spaces), and to convert these to unit cell coordinates for the current structure. The dialog box is accessed using the Display | Cartesian -> Cell Coordinates main menu option. You can use the coordinates to create a centroid in the structure, which can then be further manipulated (e.g. converted to an atom). This feature is useful, for example, if **MSRoll** has located several cavities. If you would like to see where these cavities are centered, copy the three Cartesian coordinates of the cavity from the MSRoll dialog (**Section 5.12**) and paste them here.

5.14 Convert PNG Files to AVI
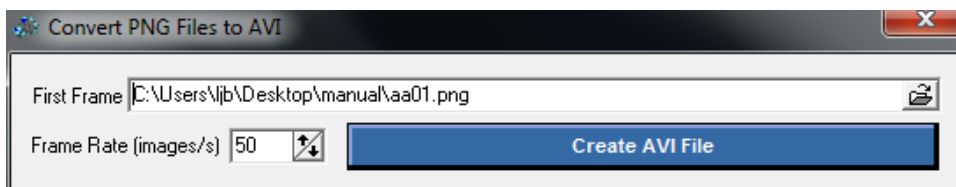
If you create a series of **POV-Ray** images representing an animation (**Section 4.12.4**), you can use this dialog (accessed via the Interface | PNG2AVI menu) to create an **AVI** file using the **PNG2AVI** program, which is part of the **X-Seed** installation. The animation files will have names that consist of a common root (specified by you before rendering), followed by number (e.g. **aa01.PNG**, **aa02.PNG**, **aa03.PNG**, etc.). Using this dialog, specify the first file in the series and then press the Create AVI File button. **PNG2AVI** will automatically create an **AVI** file. Note that the AVI file might be very large. You can most likely reduce the size of the file by simply converting and resaving it using a suitable video editing program (e.g. **FreemakeVLC**).

5.15 Add New Atom
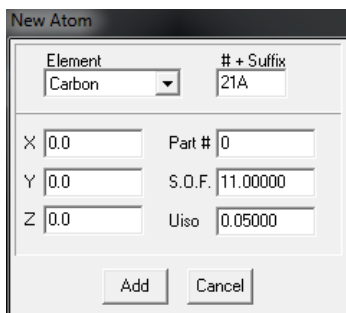
This dialog box is accessed by selecting Edit | Add New Atom from the main menu. Use this dialog box to add a new atom to the model. The controls should be self-explanatory.

It is also possible to add additional atoms by selecting Interface | SHELXL from the main menu. You can then edit atoms into the instruction set and press the Back to GUI button.

5.16 Duplicate Atom(s)
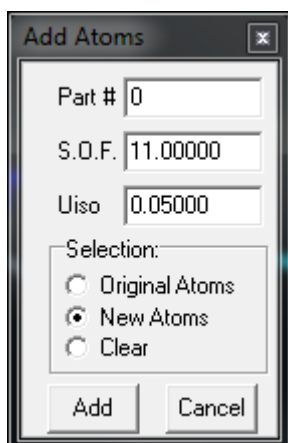
This dialog box is accessed by selecting Edit | Duplicate Atom(s) from the main menu. Use it to duplicate one or more selected atoms. This feature is useful for modelling disorder. The new atoms will have the same atomic coordinates of the selected atoms, but you have control over attributes such as **PART** numbers, site occupancies and U_{iso} values.

Here is an example of how this dialog could be useful. Consider the structure of *p-tert*-butyl-phenol with a disordered *tert*-butyl group, but where the second component of the disorder does not present itself well in the form of difference electron density peaks. You could follow this procedure (it is assumed that you are familiar with the **SHELXL** commands):

1. Assign the atoms C9, C10 and C11 a **PART** number of 1 and SOF of 21 using the View/Edit Atoms control panel (**Section 4.1**).

2. Activate the Transformations control panel.

3. Use atoms C5 and C8 to create a vector for rotation

4. Select the three atoms C9 to C11

5. Duplicate these atoms, but assign **PART** numbers of 2 and SOF of -21, and leave the default of selecting the New Atoms checked.
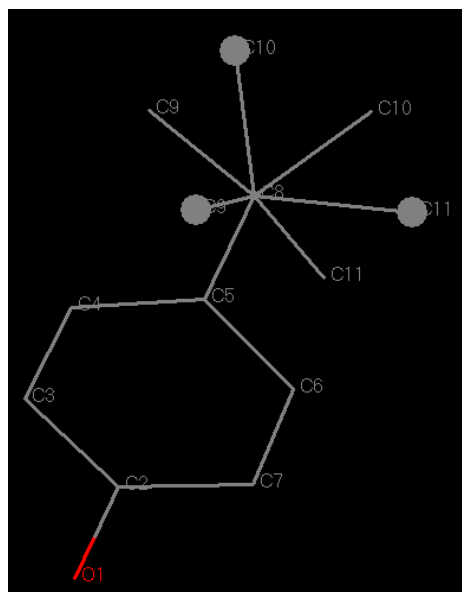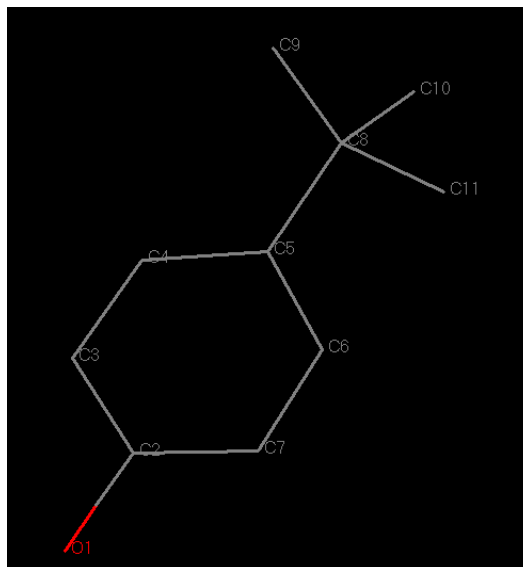
6. Press the Add button. X-Seed will immediately add three new atoms to the model. The new atoms will have the same names and atomic coordinates of the original atoms. The original atoms will be unselected, and the new atoms will now be selected.

7. In the Rotate field of the Transformations dialog, enter a value of 60 degrees and press the up-arrow. The new atoms will be rotated by 60 degrees around the C5-C8 bond. Now you can rename the new atoms and refine the disorder.

5.17 Transform Unit Cell
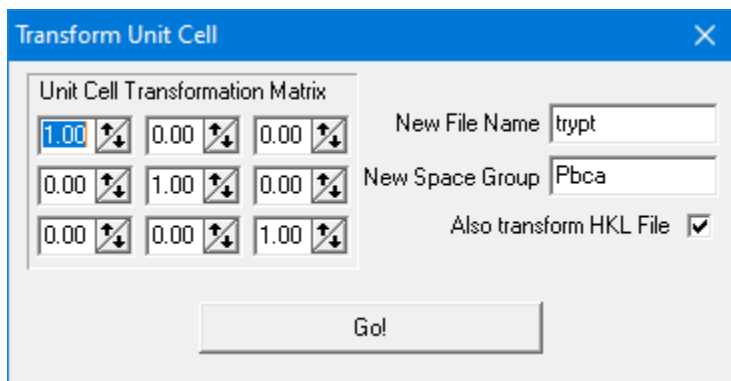
This dialog box is accessed by selecting Edit | Transform Unit Cell from the main menu. Use it to apply a transformation matrix to the current unit cell. If this also transformation involves a change of space group, simply enter the new space group. The atomic coordinates will be transformed by means of the inverse of the transpose of the transformation matrix. Check Also transform HKL File to apply the transformation matrix to the intensity data. It's a good idea to use a different file name for the transformed structure and intensity data file. You should know what you are doing when using this option.

5.18 Macro
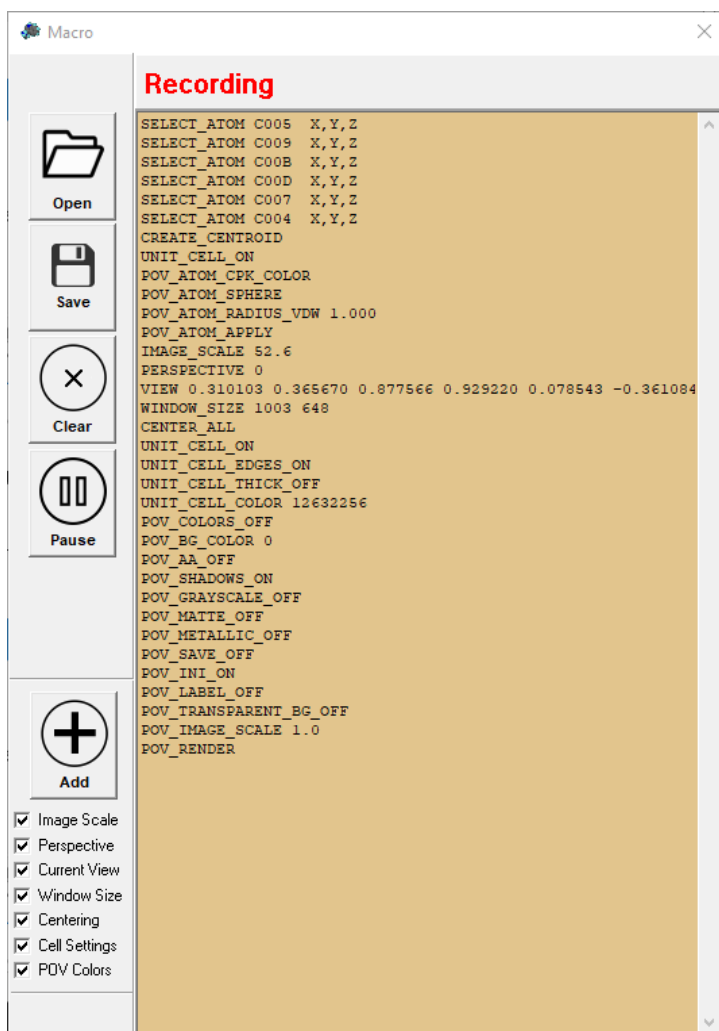
This dialog box is accessed by selecting Edit | Macro from the main menu. Use it to record a series of operations and replay them. When recording, the macro commands are automatically entered into the edit control as you perform various operations. You can save a macro and retrieve it later. You can add to the macro at any time by simply entering 'Record" mode. The main objective of the macro functionality is to allow you to produce images of a series of different but similar structures that require the same settings and operations. For example, if you have carried out crystal structure determinations on the same crystal at different temperatures and want to view them from the same direction and with the same packing and display settings. See Appendix A – Macro Commands for a complete list of macro commands that have been implemented to date.

5.19 Measurement Log
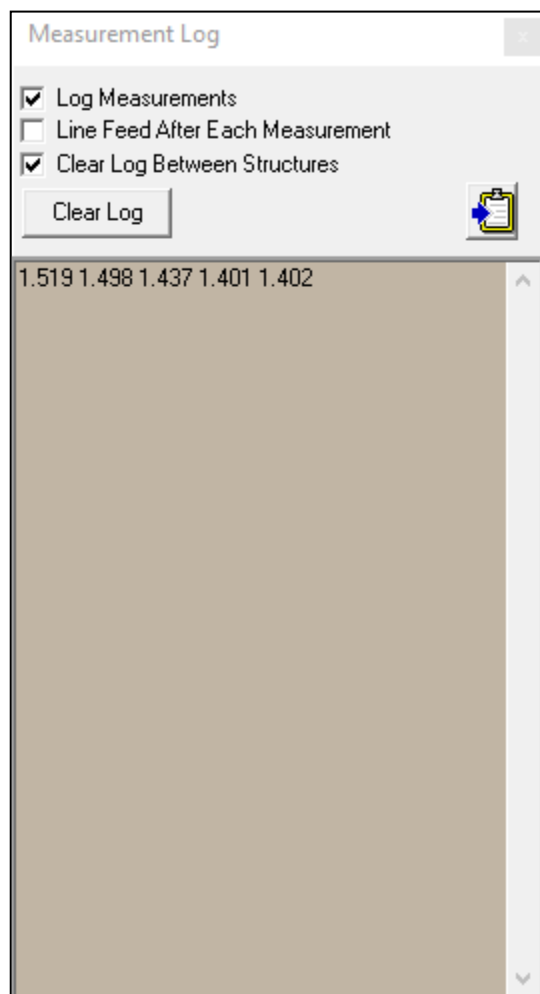
Access this dialog from the Display | Measurement Log menu. Check Log Measurements to keep track of each measurement of a distance, angle or torsion angle via the Measure control panel. This feature is especially useful if you wish to tabulate certain measurements across multiple crystal structures using the Macro feature.

6. Lazy Pulverix

A program to calculate theoretical X-Ray and Neutron diffraction powder patterns.

by   *Klaus Yvon, Wolfgang Jeitschko and Erwin Parthe*
              Laboratoire de Cristallographie aux Rayons-X
              Universite de Geneve
              24 Quai Ernest Ansermet
              CH 1211 Geneva 4
              Switzerland

              Published in *J. Appl. Cryst.* (1977), **10,** 73

LAZY PULVERIX consists of two programs:

LAZY decodes the input data and prepares the data file for *PULVERIX*
PULVERIX reads the input file and calculates the powder pattern

Running Lazy Pulverix from X-Seed

Select Interface | Lazy Pulverix from **X-Seed's** main menu. This will activate the Lazy Pulverix dialog which provides access to the various settings that can be used. Both the programs *Lazy* and *Pulv must* be in the same folder. If you wish to view the resultant powder pattern using the program *XRDPlot*, this program too must be in the *Lazy Pulverix* folder. The Program Settings dialog only requires you to specify the path to *Lazy.exe* and it is assumed that the other two programs share the same folder.

   If the executable path is set correctly (see Program Settings), *Lazy* and *Pulv* will be executed in turn when you press the Calculate button. Once the powder pattern has been calculated, the program *XRDPlot* will be executed and the trace loaded automatically. *XRDPlot,* also written by Len Barbour, is a simple program (for now) and merely provides a means of reading, displaying and printing a powder pattern which is contained as a series of lines in the file *name.LIN*. **XRDPlot** reads the powder lines and simulates a trace that consists of the summed Gaussian representations of the calculated peaks.

   The *Lazy Pulverix* program suite is particularly sensitive to correct formatting of its input file. Coding such a file manually is prone to errors and can be very frustrating to do correctly by trial and error. *X-Seed* reduces the task of calculating the powder pattern of the currently loaded structure (*SHELX* file, *CSD* structure, etc) to merely changing some settings on a dialog box and pressing a button. Currently, the only limitation is that *Lazy Pulverix* is dimensioned such that the input file cannot contain more than 500 atoms of the same type (i.e. in the asymmetric unit).

7. Layer

A computer program for the graphic display of intensity data as simulated precession photographs. *Layer* was conceived, developed and continues to be maintained by Len Barbour. A description of *Layer* has been published:

L. J. Barbour, LAYER – A computer program for the graphic display of intensity data as simulated precession photographs. *J. Appl. Cryst*. **1999**, *32*, 351.

Operation

Layer is a *Microsoft Windows*-based program that reads intensity data in *SHELX* Type 4 format and displays any level of the reciprocal lattice net. The program is primarily intended for instructive purposes, but may also prove useful as a visual complement to statistically based numeric analysis of diffraction data for space group determination.

Program outline

The executable file can be placed in any system folder while a shortcut to the program can be created on the system desktop. If an icon representing a *SHELX* type 4 *name.HKL* file is dragged onto the program's icon (or that of its shortcut) a new instance of the program is activated and the reflection data are loaded into memory. Alternatively, if *Layer* is already running, an *HKL* file can be loaded by clicking the Open File button. When loading a *name.HKL* file, *Layer* also searches the current folder for a *SHELX name.RES* or *name.INS* file in order to determine the corresponding unit cell parameters and radiation wavelength (only the `CELL` card is read). If the program fails to locate an *INS* or *RES* file, these parameters can be entered manually. The only restriction applied to the *HKL* input file is that the data must be consistent with the *FORTRAN* format convention `3I4,2F8.2`. The user can easily select the reciprocal cell axis down which to view the r.l. planes and navigation through successive levels is achieved by left- and right-clicking with the mouse cursor positioned in the data display area. Moving the mouse cursor about the data display area causes the parameters h, k, l, 2-theta, intensity and I/Imax pertaining to the closest reflection spot to be displayed. Display options that can be turned on or off include the reciprocal axis and horizontal and vertical indexing lines. Since intensity data sets rarely constitute a full sphere, it is also possible to substitute Friedels for missing reflection, thus reducing the occurrence of gaps in the display. The scale of the display can be altered by means of a slide bar control. Similarly, the user can control the relative spot intensities as well as the shade of the "film" background.

In grayscale mode, all reflection spots are shown as small circles 9 pixels in diameter while the darkness of each spot represents the reflection intensity. Since darkness values can range from 0 to 256 (from white to black), reflection intensities can range from the darkness of the film background to 256. Grayscale mode can only be used if the computer is capable of displaying more than 256 colors at a minimum screen resolution of 1024 x 768 pixels (this depends on the capability of the monitor as well as the memory capacity of the video adapter).

If the computer is not capable of displaying more than 256 colors, grayscale mode can be turned off and the reflections are then shown as black circles with their radii varying logarithmically according to relative intensity. Although the non-grayscale mode produces an unrealistic image, it is still possible to view symmetry relationships between the reflections, which, after all, is the main objective of the program.

Running Layer from X-Seed

Select Interface | LAYER from *X-Seed's* main menu. If the path to its executable file is set correctly (see Program Settings), *Layer* will execute and the current structure's *HKL* file will be loaded and displayed automatically.

8. POV-Ray

POV-Ray is a Freeware ray-tracing program and can be downloaded from this web site:
http://www.povray.org/

POV-Ray uses a sophisticated scene description language and is extremely well-suited to rendering "photorealistic" looking ray-traced images containing balls and cylinders - the basic building blocks of molecular graphics. **X-Seed** is able to create valid **POV-Ray** scene description files to produce high-quality molecular graphics images with a wide variety of settings that can be applied to the atoms and bonds. In addition to writing the scene files, **X-Seed** will invoke **POV-Ray** automatically in order to render the images. It is not necessary for you to know anything about **POV-Ray's** scene description language to make stunning images using **X-Seed's** POV-Ray interface.

8.1 Installation and Settings

POV-Ray must be obtained and installed independently of **X-Seed**. By default, **POV-Ray** might have its option "Keep Single Instance" setting unset. If you leave this setting unset, **X-Seed** will invoke a new instance of **POV-Ray** each time you render a scene. After several images, your desktop will be littered with multiple instances of **POV-Ray**. It is highly recommended that you turn this option on. You can do this by running **POV-Ray** and then under Options in the main menu, click on Keep Single Instance. When you exit **POV-Ray**, this setting will remain set; i.e. you only need to do this once. Also make sure that **POV-Ray** is not set to exit on completion. Do this by running **POV-Ray** and then selecting "Render" from the main menu. Under On Completion, make sure that Do Nothing. **POV-Ray** might also make an annoying sound when it completes a render. This can be turned off by selecting Render | Sound Settings … | and then disabling sound upon Render Complete.

In the Program Settings dialog of **X-Seed**, make sure that the **POV-Ray** executable path is set. The **POV-Ray** executable file is usually called **pvengine.exe** and should be located in the **"bin"** folder under the **POV-Ray** installation folder.

8.2 Running POV-Ray from X-Seed

The library file **textures.inc** that is supplied with **X-Seed** must be placed in the **X-Seed** installation folder (**X-Seed**'s installation procedure does this automatically). This folder contains a set of predefined textures that can be used for atoms and bonds. By default, all atoms and bonds have a plastic-like texture and finish. Use the POV-Ray control panel of **X-Seed** to customize all the **POV-Ray** settings of the atoms and bonds. **POV-Ray** will be invoked when you press the Render button, the title bar of the POV-Ray control panel, or Ctrl-R.

8.3 POV-Ray Interface Tips and Tricks

8.3.1 Colors that don't shine

You may notice that some atoms don't look as shiny as others when rendered with the default plastic texture and finish. Examples are oxygen and nitrogen atoms rendered with their default red and blue colors, as shown below:
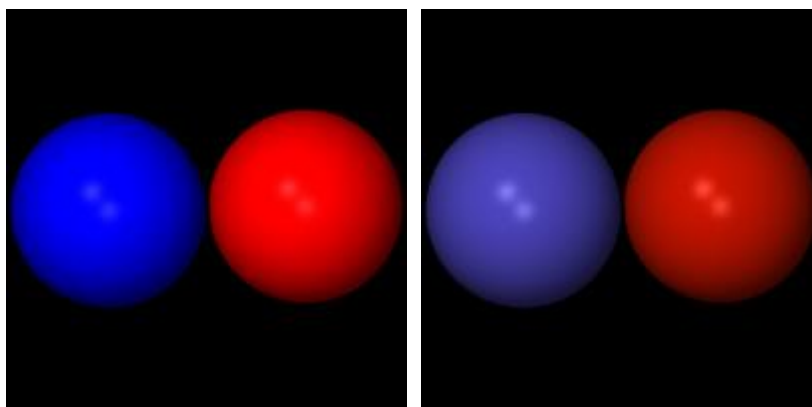
All colors passed to **POV-Ray** are given as combinations of amounts of red, green and blue (each ranging from 0 to 255). I have noticed that atoms assigned colors defined with very high values of one or more of the 3 primary colors don't show up as being very glossy. It is highly recommended that you use the Element Settings dialog to assign colors to elements such that the colors contain a mixture of all three primary colors. It is best that you experiment with the colors and choose the ones that suit your taste.

8.3.2 Antialiasing

By default, antialiasing is turned off. Antialiasing affects the quality of the image and the highest quality is achieved with antialiasing turned on with a factor of 0.0. The price to pay for the higher quality is the increased time taken to render the image. It is best to leave antialiasing turned off until you are ready to produce the final image. Usually the results are worth the time. Examples are shown below:
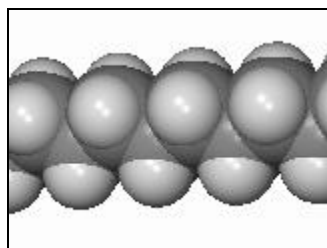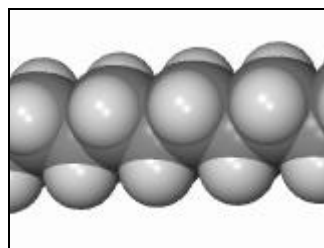
Antialiasing OFF   Antialiasing ON

8.3.3 Preliminary images

Before you create the final image, you will probably render several test images to check that all is well. The larger the image, the longer it takes to render. This also applies to the quality of the image. To save time, it is best to render test images using a small image size, and with antialiasing turned off. A "Quick Render" option has been built into the program for this purpose.

8.3.4 Animations

Remember, when you create an animation, **POV-Ray** has to render as many files as there are frames. Unless you have a lot of time on your hands, keep these frames small (e.g. 100x100 pixels). The more frames you have in the animation, the smoother the rotation - 36 frames per seem to produce a relatively smooth animation.

8.3.5 POVLabel

POVLabel still needs some work. However, it is perfectly functional in its present form. A more detailed description of its features is deferred until the program is more complete.

9. POVLabel

POVLabel reads a *name.LBL* file written by *X-Seed* and a corresponding *name.PNG* file written by **POV-Ray**. The bitmap is displayed on screen. Double-click on an atom to make its label visible. Double-click on a label to hide it. When you click on a label, it becomes the "current" label described below. When you click on the bitmap, and not on any label in particular, ALL labels become current. The font settings tool window can be used to change the settings of the current label(s). Use the left mouse button to drag the labels around until you are happy with their placement. Using the menu, you can save the label properties so that you can exit the program, but continue with the current settings at a later stage. When you select the save option, the *name.LBL* file is overwritten. You can export the labeled image as a bitmap in which case the labels are merged with the image. This feature performs a capture operation on the bitmap window of **POVLabel**, so make sure that no other windows overlap this window during the capture process. Do not overwrite the original *name.PNG* file unless you're sure you will not need it again.

POVLabel is still under development and will be extended in the future.

10 Tutorial

This tutorial consists of a worked example of the determination of the structure of tryptamine HCl. If you follow the procedure in this chapter exactly, you will complete the solution and refinement of a "well-behaved" structure and will be exposed to some of the most commonly used features of *X-Seed*. Schematically, tryptamine HCl has the following structure:
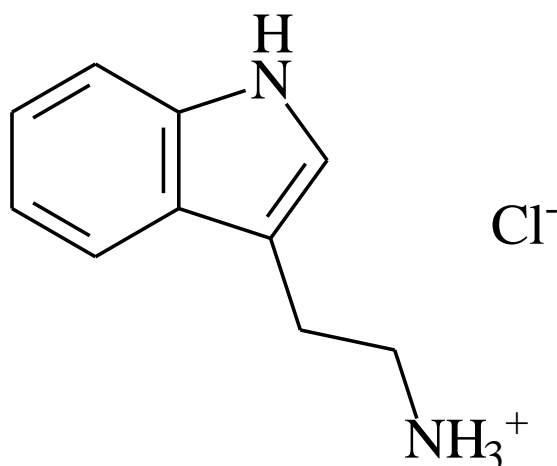
X-Seed's installation procedure places the data files required for the tutorial structure in the "test" folder under the *X-Seed* installation folder. This folder should contain the files *trypt.ins* and *trypt.hkl*. The *HKL* file was recorded on a Bruker SMART CCD system and the *INS* file was created by the program *XPREP* (part of the *SHELX-TL* program package). Of course the *HKL* file is essential, but if you know the cell parameters and space group, you don't need the *INS* file. However, having an *INS* file that contains at least the cell parameters makes life a little easier.

trypt.ins contains the following text:

```
TITL Tryptamine.HCl in Pbca
CELL 0.7107 8.4691 10.0580 23.9490 90.000 90.000 90.000
ZERR 8.0000 0.0020 0.0024 0.0059 0.000 0.000 0.000
LATT 1
SYMM 0.5-X, -Y, 0.5+Z
SYMM 0.5+X, 0.5-Y, -Z
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N CL
UNIT 88 96 16 8
TEMP -100
SIZE 0.40 0.35 0.10
TREF
HKLF 4
```

It is assumed here that you have already set the paths to *SHELXS* and *SHELXL* using the Program Settings Dialog.

10.1 Structure Solution

1)   Start **X-Seed.**

2)   Open the file **trypt.INS**. You can view the unit cell parameters and molecular formula by pressing the Crystal Data button. Note that the molecular formula is incorrect for tryptamine HCl – the numbers for carbon and hydrogen on the **SHELX UNIT** card are intentionally incorrect for the purposes of this tutorial.

3)   Close the Crystal Data dialog by pressing "OK" and then select Interface | SHELXS from the main menu. On the SHELXS dialog, press Solve to invoke **SHELXS** to carry out a routine, default solution using Direct Methods.

4)   A dialog will appear that summarizes the pertinent parameters extracted from the file **trypt.lst**. This dialog merely presents these parameters visually, but tells you nothing that you can't find in the **LST** file.

5)   Since this is a 'well-behaved' structure and has most likely been solved successfully, press the Accept button.

6)   The peaks and atoms contained in the file **trypt.res** will be displayed in the Model Display Window and you can use the mouse to rotate and resize the model.

7)   All 22 peaks extracted from the **RES** file will be displayed, along with the correctly-assigned Cl$^-$ ion. By default, a peak is assigned the bonding radius of a carbon atom. The connectivity will therefore be determined accordingly. Clearly some of the peaks represent atoms that should be modelled while others represent "noise". When rolling the mouse wheel, each detent will suppress display of the least intense peak. You can therefore roll the mouse wheel until it is possible to discern the backbone structure of tryptamine:
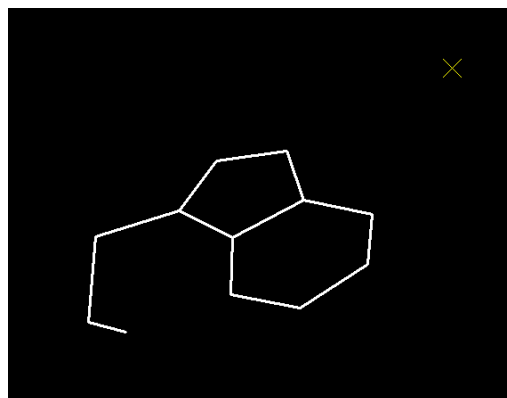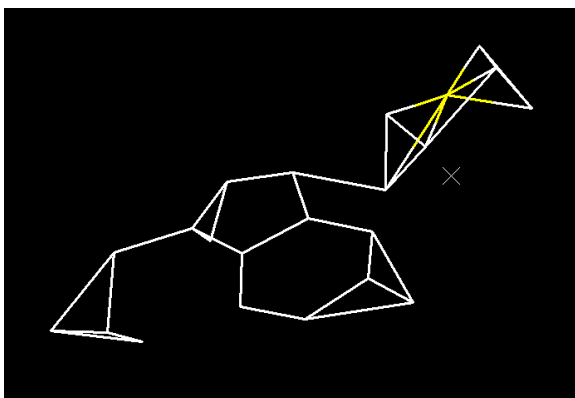
8)   Now you need to assign the correct element types to the peaks. It is possible to select atoms and then change the element assignments collectively by pressing F2 and selecting the element from the pop-up menu. However, in this particular example, most of the displayed peaks should become carbon atoms. To save some time, select Edit | Change Q's to C's. Now select the two nitrogen atoms by clicking on them – when selected, atoms are shown as small filled circles. Press the F2 key and then select Nitrogen from the pop-up menu to update the model. The structure is now solved and the atoms need to be labeled systematically.
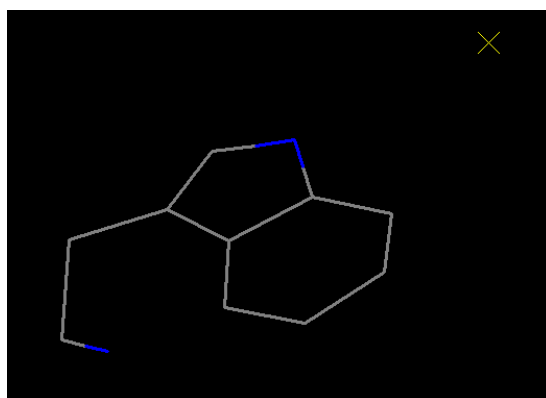
10.2 Labeling Atoms

(1)   Press L to show atom labels. The atoms are labeled according to the peak numbers from which they were derived:
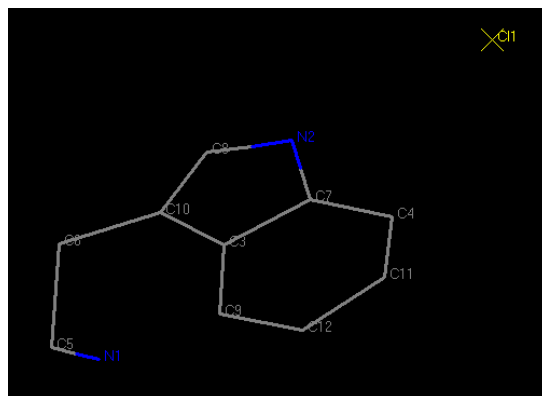
2)   Press the Rename button to activate the Atom Names control panel.

3)   Starting at **N1**, click on the atoms of the model in a logical sequence to label them systematically as shown below. You should see the labels change as you click on the atoms.
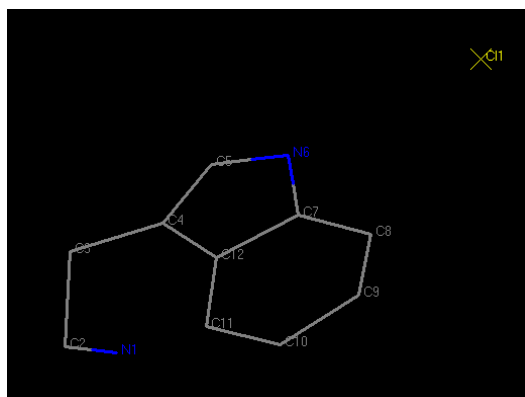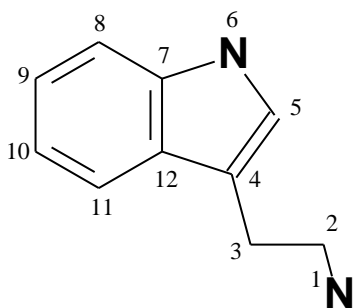
4)   Press Sort to sort the atom list

5)   Press L again to turn labels off.

10.3 Structure Refinement

1)   Select Interface | SHELXL from the main menu

2)   The instruction set should be as follows:

```
TITL C:\X-Seed\Test\trypt_a.res in Pbca
CELL 0.71070 8.4691 10.0580 23.9490 90.000 90.000 90.000
ZERR 8.0000 0.0020 0.0024 0.0059 0.000 0.000 0.000
LATT 1
```

```
SYMM 0.5-X, -Y, 0.5+Z
SYMM 0.5+X, 0.5-Y, -Z
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H N CL
UNIT 88 104 16 8
BOND
FMAP 2 1 53
PLAN 20 0 0
L.S. 4
FVAR 1.00000
Cl1    4    0.17280    0.64470    0.03970   11.00000    0.05000
N1     3    0.04760    1.35680    0.06680   11.00000    0.05000
C2     1   -0.05780    1.36280    0.11550   11.00000    0.05000
C3     1   -0.17210    1.24240    0.11420   11.00000    0.05000
C4     1   -0.08460    1.11080    0.11530   11.00000    0.05000
C5     1   -0.07240    1.02080    0.07590   11.00000    0.05000
N6     3    0.00960    0.91920    0.09050   11.00000    0.05000
C7     1    0.06580    0.93660    0.14510   11.00000    0.05000
C8     1    0.16550    0.85770    0.18020   11.00000    0.05000
C9     1    0.19540    0.90310    0.23170   11.00000    0.05000
C10    1    0.14350    1.02890    0.25000   11.00000    0.05000
C11    1    0.04340    1.10960    0.21660   11.00000    0.05000
C12    1    0.00090    1.05900    0.16290   11.00000    0.05000
HKLF 4
```

3) Press Refine to invoke **SHELXL**.

4) Press Accept to accept the refinement.
(Note: if you reject the run, the model will *not* be updated, the **res** and **lst** files pertaining to the latest **SHELXL** run will be deleted, and the previous **res** and **lst** files will be restored).

5) In the Model Display Window, you should see that the difference electron density peaks correspond to the positions of the missing hydrogen atoms. Peaks have the same bonding radius as carbon atoms, and will thus appear to be bonded to too many atoms in this case. Since we are going to place hydrogen atoms in calculated positions, we can ignore the peaks.

6) Since we don't need them, press Q to suppress display of the peaks, or Delete | Peaks.

7) If you would like to view the parameters associated with any atom, press the Atoms button to activate the View/Edit control panel. Just hover close to any atom to view its parameters on the control panel. Note that when you point at a bond, its length is given on the status bar at the bottom of the window.

10.4 Adding Hydrogen Atoms

1) IMPORTANT: **X-Seed** does not actually generate hydrogen atoms. It instructs **SHELXL** to do this in the next refinement using **HFIX** cards.
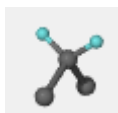
2)   Press the H-Atoms button to activate the Generate H-Atoms (HFIX) control panel.

3)   Select **C5**, **N6**, **C8**, **C9**, **C10** and **C11** and press

 to mark these atoms for `HFIX 43` hydrogen atom generation during the next call to **SHELXL**.

4)   Select **C2** and **C3** and press

 to mark these atoms for `HFIX 23` hydrogen atom generation.

5)   You may wish to verify that **N1** is protonated (of course it is, but it is instructive not to assume that at this stage) before using a geometric model to place its hydrogen atoms, so run another routine **SHELXL** refinement at this stage.

6)   Roll the mouse wheel until only the strongest difference electron density peaks are displayed. Two of these are satellites of the chloride anion (as may be expected for a relatively heavy atom), so you might want to increase the number of peaks. When you display the strongest 6 peaks, you will see that three of these are close to **N1** at positions consistent with the hydrogen atoms of a protonated amine, and in a staggered arrangement with respect to the substituents on **C2**.

7)   Turn off the display of the peaks (Q) and go back to the Generate H-Atoms (HFIX) control panel. Select **N1** and then press
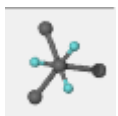
 to instruct **SHELXL** to generate three hydrogen atoms using the staggered model.

10.5 Continue Refinement

1)   Refine the model again using **SHELXL**. When the refinement completes you will see that the residual electron density peaks do not indicate that the model may be incomplete (e.g. missing solvent molecules, disorder), so you can delete all the peaks - select Delete | All Peaks from the main menu.

2)   Now it is time to refine non-hydrogen atoms anisotropically.

3) Activate the View/Edit Atoms control panel. Press the space bar to select all atoms. Additional controls will appear on the control panel (in response to atom selection).

4) Press the ANIS button to instruct **SHELXL** to treat the selected atoms anisotropically in the next refinement (hydrogen atoms, although selected, will *not* be treated anisotropically unless you are refining against neutron diffraction data).

ANIS

5) Run another routine refinement. R1 should have dropped to about 0.035 by now. Delete the peaks again.

6) Press E to toggle display of 50% probability thermal axes. Turn off display of hydrogen atoms (Press H) to see the thermal ellipsoid axes more clearly. Note that the atoms appear to be relatively well-behaved, thermally. When you are satisfied that the thermal motion is reasonable, you can restore the hydrogen atoms by once again pressing H and turn off the thermal ellipsoid axes (E).

7) You may have noticed that the model is complete, but **SHELXL** still reports that **"** `** Cell contents from UNIT instruction and atom list do not agree **`**"**. This means that the model does not agree with the `SFAC/UNIT` parameters. Press the Crystal Data button. The assigned unit cell contents are $C_{88}H_{96}N_{16}Cl_8$. However, the model, together with Z, indicates that the correct formula is $C_{80}H_{104}N_{16}Cl_8$. If you press the button Adjust molecular formula to fit current model, the assigned formula will be updated according to the model and Z. **Note** : only use this feature if the model is complete. While this correction of the formula does not affect refinement, the mistake would most likely carry through to the CIF file if not corrected.

8) Now it's time to run the final refinement. Select Interface | SHELXL. On the SHELXL dialog add the ACTA instruction (there is no need to impose a 2-theta limit for this structure, so you can leave this field blank). Go to the Reflections page. The 50 worst reflections (extracted from the latest **lst** file) are listed in descending order of **Error/e.s.d**. If you wish to suppress any of these reflections (e.g. if it appears that they may have been obscured by the beam-stop – i.e. $F_c \gg F_o$), check them and they will be inserted as **OMIT h k l** cards in the next refinement run. In this case, **Error/e.s.d**. does not appear to be unacceptable for any of the reflections, so **OMIT h k l** cards are not necessary.

9) Run **SHELXL** one last time to complete the refinement and to generate a **CIF** file.

10.6 Running Layer

1) Select Interface | Layer from the main menu to invoke layer.

2) Layer automatically loads the reflections from the file **trypt.hkl** and the cell parameters from **trypt.ins**.

10.7 Running LAZY PULVERIX

1) Select Interface | LAZY PULVERIX from the Main Menu to calculate the powder pattern for the structure.

2) Adjust the settings on the LAZY PULVERIX dialog as required, and press the Calculate button to run first ***LAZY***, then ***PULVERIX*** and then ***XRDPLOT***. You may have to follow the procedure outlined above and in Chapter 2 to set the DOS windows to close automatically when the DOS processes complete.

3) Barring any glitches, ***XRDPLOT*** should appear showing you the powder pattern for tryptamine HCl.

10.8 Final note

X-Seed has many features that were not used in this example. You will need to experiment with the program and read this instruction manual to familiarize yourself with these features. Although I strongly encourage questions from users, I have been asked many questions that have been addressed in the instruction manual. Please consult the manual before asking questions about ***X-Seed***.

Appendix A – Macro Commands

X-Seed currently implements the following macro commands:

SELECTION

```
1       SELECT_ALL
2       DESELECT_ALL
3       SELECT_FRAG #
4       DESELECT_FRAG #
5       TOGGLE_ALL
6       SELECT_PART #
7       DESELECT_PART
8       SELECT_PEAKS
9       DESELECT_PEAKS
10      TOGGLE_PEAKS
11      SELECT_ H_ATOMS
12      DESELECT_ H_ATOMS
13      TOGGLE_H_ATOMS
14      SELECT_ELEMENT #
15      DESELECT_ELEMENT #
16      SELECT_ISOTROPIC
17      DESELECT_ISOTROPIC
18      TOGGLE_ISOTROPIC
19      SELECT_ANISOTROPIC
20      DESELECT_ANISOTROPIC
21      TOGGLE_ANISOTROPIC
22      SELECT_ASU
23      DESELECT_ASU
24      TOGGLE_ASU
25      SELECT_SYM
26      DESELECT_SYM
27      TOGGLE_SYM
28      SELECT_NPD
29      SELECT_ATOM name matrix
30      UNSELECT_ATOM name matrix
```

VIEW

```
31      STEREO_ON
32      STEREO_OFF
33      UNIT_CELL_ON
34      UNIT_CELL_OFF
35      PEAKS_ON
36      PEAKS_OFF
37      ASU_ONLY_ON
38      ASU_ONLY_OFF
39      H_BONDS_ON
40      H_BONDS_OFF
41      POV_COLORS_ON
42      POV_COLORS_OFF
43      CENTER_MOLECULE
```

```
44      RESET_ROTMX
45      VIEW_PLANE h k l
46      VIEW_VECTOR h k l
47      HIDE_SELECTION
48      UNHIDE_ATOMS
49      IMAGE_SCALE #
50      PERSPECTIVE #
51      VIEW # # # # # # # # #
52      WINDOW_SIZE
53      CENTER_ALL
54      CENTER_VISIBLE
55      CENTER_SELECTED
56      UNIT_CELL_ON
57      UNIT_CELL_OFF
58      UNIT_CELL_EDGES_ON
59      UNIT_CELL_EDGES_OFF
60      UNIT_CELL_THICK_ON
61      UNIT_CELL_THICK_OFF
62      UNIT_CELL_COLOR #
```

DELETE
```
63      DELETE_RESIDUAL_PEAKS
64      DELETE_SELECTED_ATOMS
65      DELETE_PEAKS
66      DELETE_H_ATOMS
67      DELETE_SYM_ATOMS
68      DELETE_SELECTED_BONDS
69      DELETE_BIND_LIST
70      DELETE_FREE_LIST
71      DELETE_H_BONDS
```

GROW/ADD
```
72      PRESERVE_POV_SETTINGS_ON
73      PRESERVE_POV_SETTINGS_OFF
74      CREATE_CENTROID
75      GROW_ATOM name matrix
76      GROW_FRAG name matrix
77      GROW_SPHERE_ATOM # name matrix
78      GROW_SPHERE_FRAG # name matrix
79      GROW_H_BOND_ATOM name matrix
80      GROW_H_BOND_FRAG name matrix
81      GROW_SYM matrix
82      EXTEND_FRAGS
83      PACK_FRAG matrix
84      PACK_ATOM matrix
85      PACK_LIMIT # # # # # #
86      ADD_NEW_ATOM Name At# X Y Z SOF Uiso PART#
87      SELECTED_TO_ASU
```

POV-RAY
```
88      POV_BG_COLOR #
```

```
89      POV_AA_ON
90      POV_AA_OFF
91      POV_SHADOWS_OFF
92      POV_SHADOWS_ON
93      POV_GRAYSCALE_ON
94      POV_GRAYSCALE_OFF
95      POV_MATTE_ON
96      POV_MATTE_OFF
97      POV_METALLIC_ON
98      POV_METALLIC_OFF
99      POV_SAVE_ON
100     POV_SAVE_OFF
101     POV_INI_ON
102     POV_INI_OFF
103     POV_LABEL_ON
104     POV_LABEL_OFF
105     POV_TRANSPARENT_BG_ON
106     POV_TRANSPARENT_BG_OFF
107     POV_IMAGE_SCALE #
108     POV_RENDER
109     POV_ATOM_CPK_COLOR
110     POV_ATOM_CUSTOM_COLOR #
111     POV_ATOM_SPHERE
112     POV_ATOM_RADIUS_FIXED #
113     POV_ATOM_RADIUS_VDW #
114     POV_ATOM_TRANSPARENT_SPHERE
115     POV_ATOM_ELLIPSOID
116     POV_ATOM_CLEAR_SELECTION
117     POV_ATOM_APPLY
118     POV_BOND_RADIUS #
119     POV_BOND_CPK_COLOR
120     POV_BOND_CUSTOM_COLOR #
121     POV_BOND_5050_ON
122     POV_BOND_5050_OFF
123     POV_BOND_FRAGS #
124     POV_BOND_SPHERES_ON
125     POV_BOND_SPHERES_OFF
126     POV_BOND_CLEAR_SELECTION
127     POV_BOND_APPLY_NORMAL
```

MEASURE

```
128     MEASURE_DIST
129     MEASURE_ANG
130     MEASURE_TANG
```