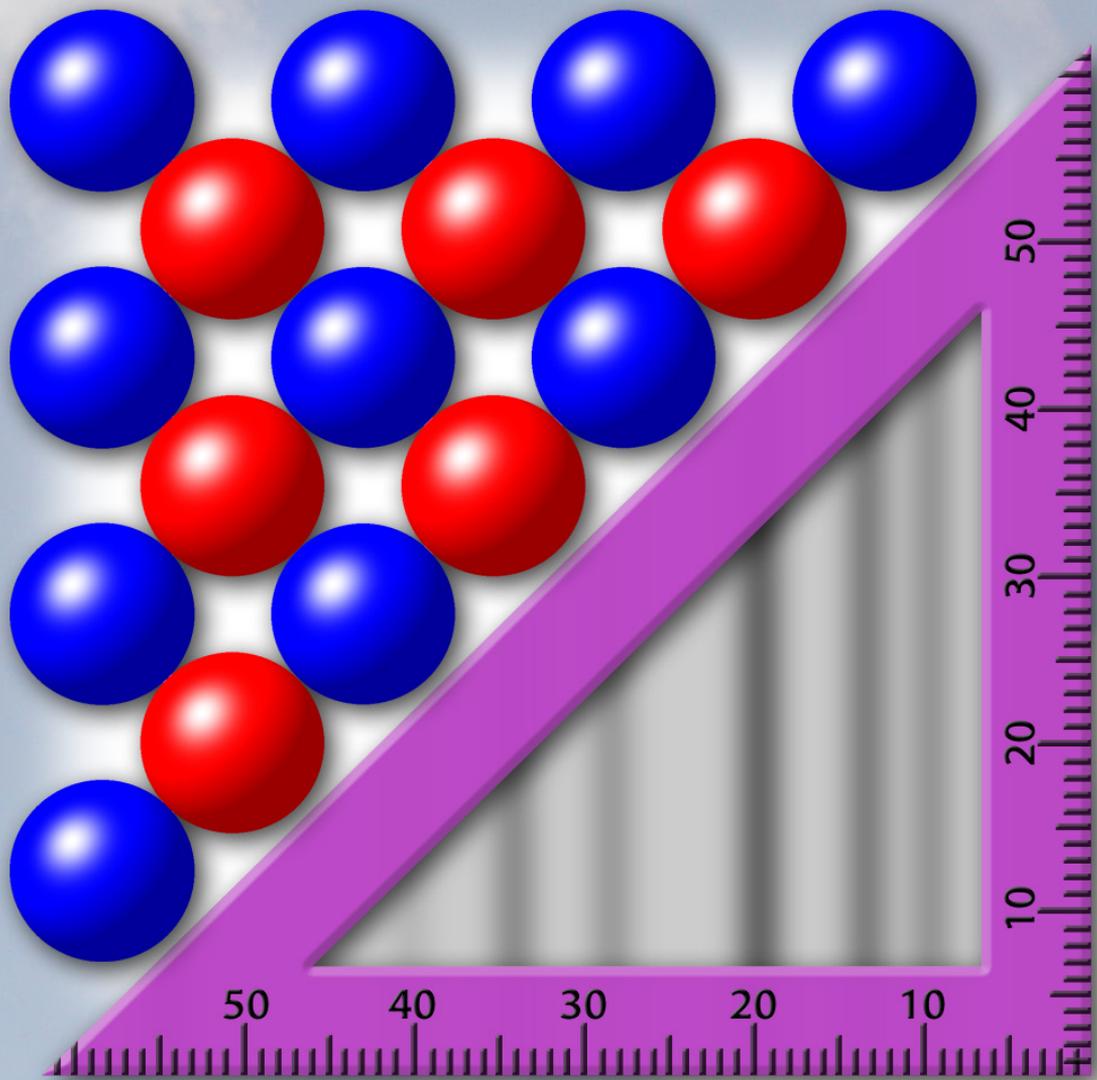


CrystalDiffract[®]

Interactive Powder Diffraction Software

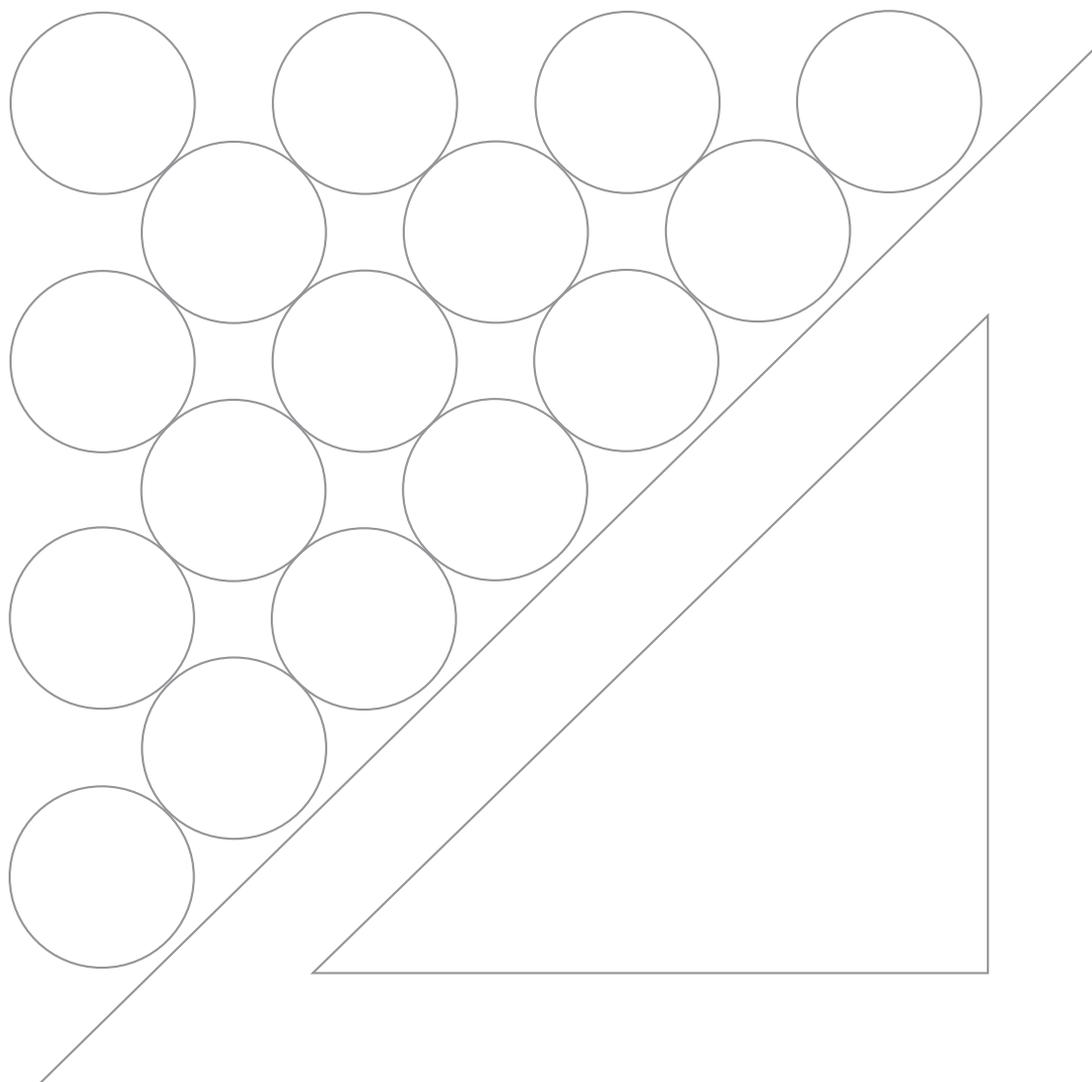


User's Guide

Version 6 for Mac & Windows

CrystalDiffract[®]

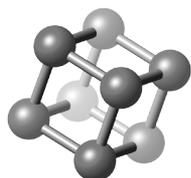
Interactive Powder Diffraction Software



User's Guide

CrystalDiffract[®]

USER'S GUIDE



CrystalMaker Software Limited
Oxford • England

While the authors have tried to ensure that all information in this guide is accurate, CrystalMaker Software Ltd cannot be held responsible for any errors. In particular, because CrystalMaker Software Ltd frequently releases new versions and software updates, images shown in this guide may be slightly different to what you might see on your screen.

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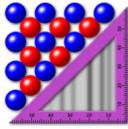
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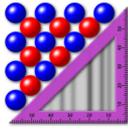


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Chapter 1

Introducing CrystalDiffract

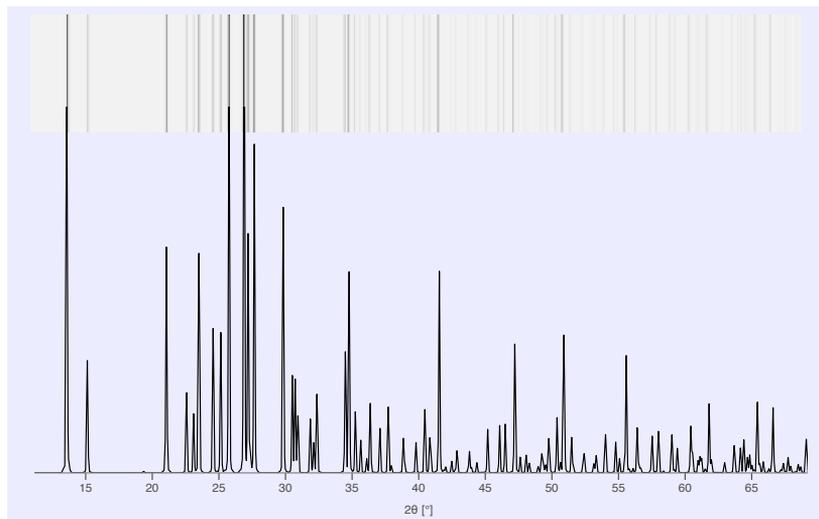
Welcome to CrystalDiffract®: a stand-alone program designed to make the world of powder diffraction accessible and fun. CrystalDiffract lets you simulate diffraction properties on your desktop, and includes tools for comparing simulated data with real, observed data.

This chapter explains what CrystalDiffract is designed for, its key features, and the major changes from earlier versions of the software.

Powder Diffraction

CrystalDiffract lets you simulate what happens when a powdered crystal sample (comprising millions of tiny *crystallites*) is exposed to a radiation beam, resulting in patterns of scattered intensity, which can be recorded as lines on a film, or as intensity peaks by a detector.

Powder diffraction patterns can be visualized as “films” (top) or as graphs (bottom).



CrystalDiffract differs from its sister program, SingleCrystal™, which is designed to simulate diffraction patterns from one, *single* crystal, when exposed to X-rays, neutrons or electrons.

CrystalDiffract Key Features

CrystalDiffract 6 is a self-contained powder diffraction program, combining the best graphing tools with advanced simulation, real-time interactive parameter control and intuitive measurements.

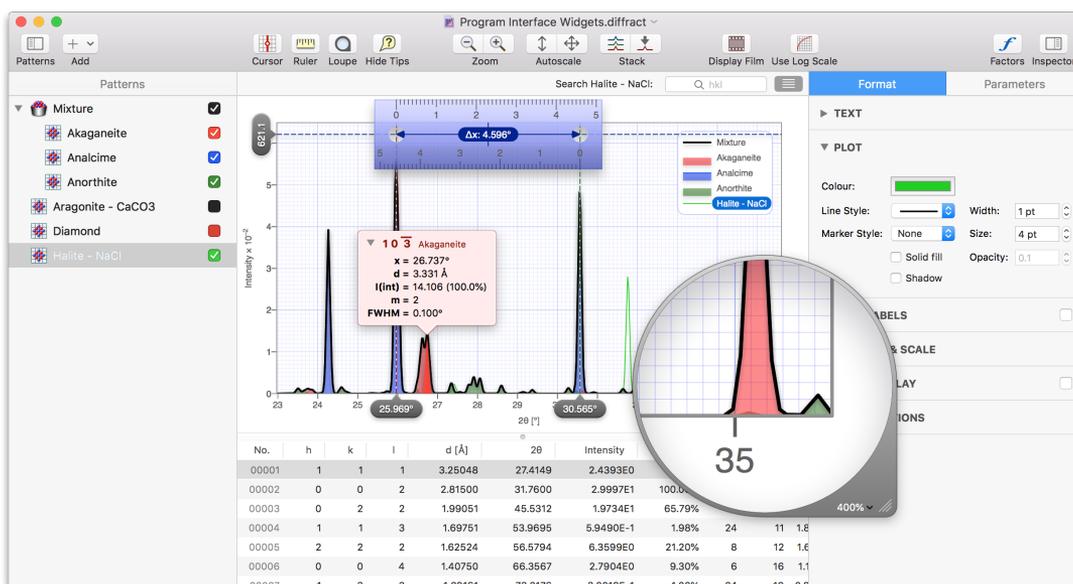
Crystal Building and Editing

CrystalDiffract includes a stand-alone crystal editor, so you can define new crystal structures directly in the program, and observe their diffraction properties. You can also load *CrystalMaker* binary “crystal” or text files, or import data from *CIF* text files.

Advanced Diffraction Simulations

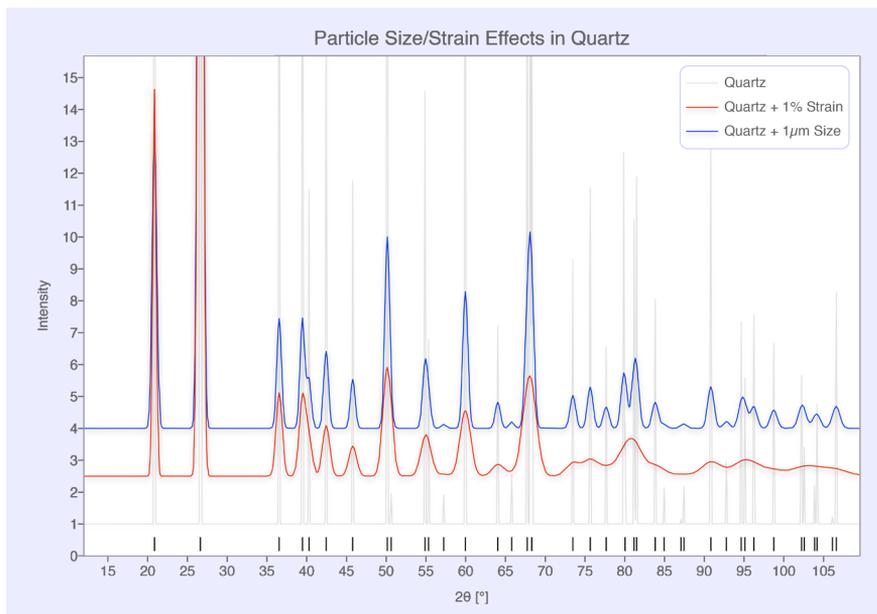
CrystalDiffract can simulate the key powder diffraction techniques used today, including:

- Angle-dispersive X-ray diffraction (laboratory source).
- Angle-dispersive neutron diffraction (reactor source).
- Angle-dispersive electron diffraction (polycrystalline sample in a transmission electron microscope).
- Energy-dispersive X-ray diffraction (synchrotron source).
- Time-of-flight neutron diffraction (spallation source).



CrystalDiffract features an engaging and intuitive user interface with high-quality graphics and easy measurement.

An interactive diffraction experiment in CrystalDiffract: simulating the effects of particle size and strain in the mineral quartz.



Powerful Graphics with Intuitive Measurements

CrystalDiffract features gorgeous anti-aliased “Retina” (high-DPI) graphics (Mac) with extensive formatting control and high-quality vector and pixel graphics output. The user interface makes it a breeze to browse diffraction data, search for peaks, measure and label data on screen.

Real-Time Parameter Control

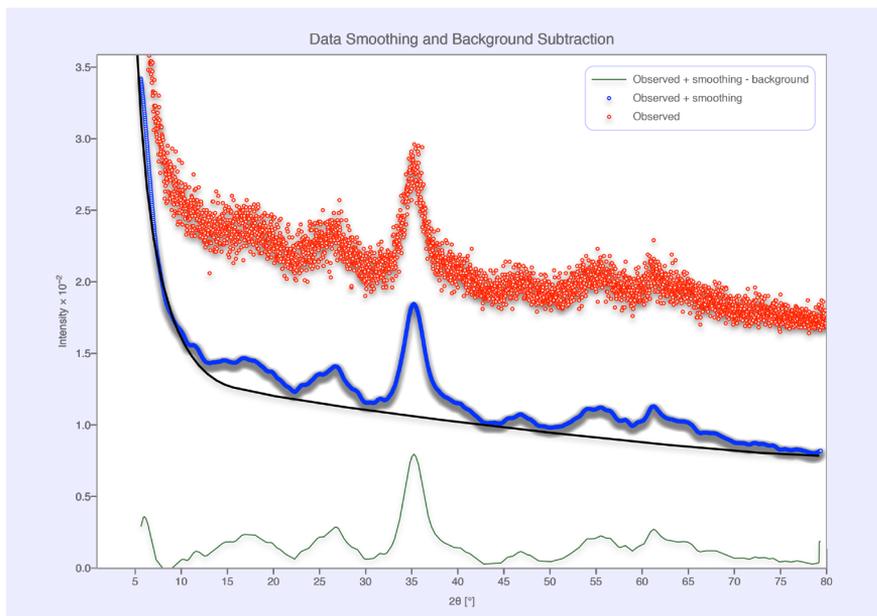
CrystalDiffract lets you adjust simulation parameters in real time, via a dedicated Parameters Inspector. For example, you can change peak shapes, line broadening, particle size and strain phenomena, preferred orientation, site occupancies and lattice parameters. This can be highly instructive in learning the fundamental, structural controls on diffraction: an ideal teaching tool.

Characterize Observed Data

CrystalDiffract is an ideal way to characterize real, observed diffraction data. CrystalDiffract can read from observed diffraction profiles in *xy* or *Rigaku ASC* formats. These profiles can be displayed in the same window with simulated patterns—which lets you identify diffraction peaks and perhaps detect the presence of impurities.

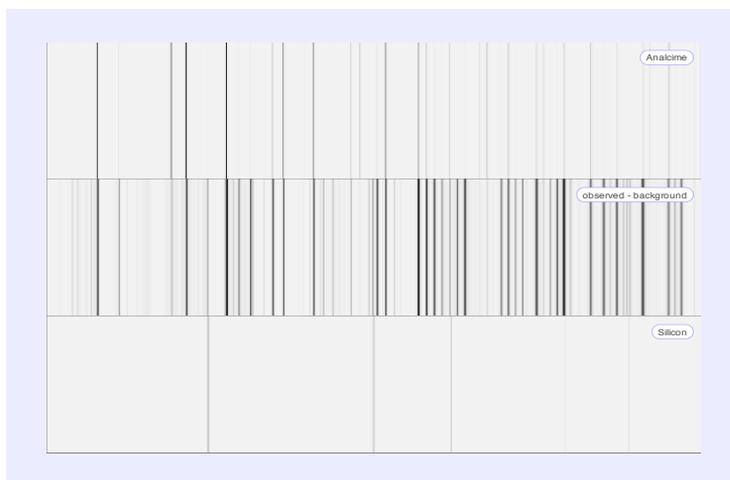
New tools make it possible to subtract the background from an observed pattern, smooth noisy data, rescale the x-axis, and apply *x*- and *y*-axis offset corrections.

Data smoothing and background subtraction for an observed pattern.



CrystalDiffract makes it easy to simulate multi-phase *mixtures*, which can include both simulated *and* observed components. Mixture composition can be adjusted in real time, using slider controls: a great way to characterize the composition of an observed sample.

Comparing observed and simulated diffraction patterns in "Film" mode.



What was New in CrystalDiffract 6?

CrystalDiffract 6.0, released in October 2013, was an all-new application, entirely rewritten using the latest object-oriented design, taking advantage of multi-processing and multi-touch technologies for Mac and Windows.



State-of-the-art design

CrystalDiffract 6 is a modern, 64-bit application.

- Object-oriented design, rewritten in Cocoa (Mac).
- 64-bit with multi-processor support for faster simulations.
- Multi-touch offers zooming and scrolling with a trackpad.
- Drag-and-drop patterns between windows.
- Full-screen mode including “Spaces” support (Mac).
- Supports document tabs in macOS 10.12 “Sierra”.



Beautiful new interface

The program was redesigned to maximize your productivity.

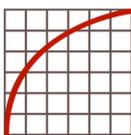
- Search and find reflexions using the integrated search bar.
- Cursor tool with info display in the Legend.
- Peak tips highlight reflexions and show data.
- Interactive Ruler for easier measurements.
- High-resolution Loupe, saves on scrolling and zooming.
- Scattering factors window with live visualization and editing.
- New preferences window.



Stunning graphics to go

Detailed control over all aspects of your diffraction plot.

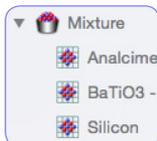
- Retina display with accelerated graphics (Mac and Microsoft Surface Pro)
- Copy/export/drag graphics from any view.
- Wide choice of line and marker styles for graphs.
- Control font sizes and styles.
- New labelling options, including rotate, arrows and positions.
- New Film display modes plus Gamma control.



Sophisticated simulations

New simulation modes, combined with flexible presentation.

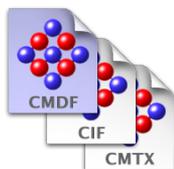
- Q-space ($2\pi/d$) axis option.
- Logarithmic y-axis option.
- Separate simulation and axis display.
- Improved time-of-flight simulation, with user-specified instrumental resolution ($\Delta d/d$).
- Reflexions grouped by symmetry, rather than just d-spacing.
- Export diffraction profiles for simulated mixtures.
- Toggle atomic displacement parameters (“thermal ellipsoids”).



Dynamic mixtures

Simulating multi-component diffraction has never been easier.

- Easy drag-and-drop mixture editing.
- Combine observed and simulated patterns in the same mixture.
- Visualize mixtures and separate patterns in the same window.



Flexible data input/output

A self-contained program to streamline your workflow.

- Imports Rigaku “ASC” diffractometer files.
- Imports CIF, STRUPLO and CMTX text files.
- Exports crystal structures as CIF or CMTX files.
- Visualize selected patterns as crystals in CrystalMaker.
- Integrated data editor lets you create new patterns directly.
- Self-contained: build new crystals from scratch.



Powerful data handling

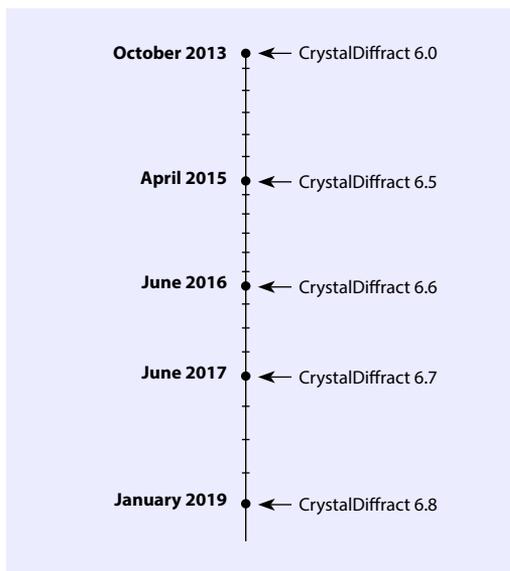
Rescale observed patterns, strip backgrounds & smooth noisy data.

- X-value scaling for observed data.
- Background subtraction with intuitive *Bézier*-curve control.
- Data smoothing for observed data, using a fast bilinear model.

CrystalDiffract 6 Updates

CrystalDiffract 6 was released in October 2013. Since that date there have been numerous incremental updates, plus a number of major releases, with significant functionality, as summarized in a timeline below:-

CrystalDiffract version timeline. Minor updates are indicated by the tickmarks.



These major “.x” updates as well as the minor “.x.y” updates were provided as free updates to licensed CrystalDiffract 6 customers.

What was New in CrystalDiffract 6.5?

CrystalDiffract 6.5, introduced in April 2015, introduced a bright new interface with powerful new tools, simulation modes, plus many “under-the-hood” changes.

The new interface included a revised window design with colourful toolbar icons and a tabbed Inspector, divided into separate “Format” and “Parameters” panes. New text controls and a Graph Options group made their appearance.

New tools included a Scroller, with full-attenuation preview, for easy navigation; a faster, smoother Loupe—and a “Square Loupe” display option.

Electron powder diffraction patterns could now be simulated, and a Preferred Orientation correction was introduced.

This version also included accelerated graphics for most screen operations, coloured graph backgrounds with output transparency control, plus a range of graphics export formats with transparency and scaling.

What Was New in CrystalDiffract 6.6?

CrystalDiffract 6.6, released in June 2016, featured numerous interface enhancements including a redesigned toolbar, updated window design and support for macOS 10.12 “Sierra”. The Windows version was completely rewritten, using the latest Microsoft Windows .NET framework, which provides a more stable basis for future software development.

What was New in CrystalDiffract 6.7?

CrystalDiffract 6.7, released in June 2017, was the first version to support CrystalMaker X, an all-new 64-bit program designed for the latest Mac and Windows operating systems. CrystalDiffract 6.7 can read from the new CMDX document format and works with CrystalMaker X’s powder diffraction commands. CrystalDiffract 6.7 for Mac was also designed to support macOS 10.13 “High Sierra”.

What’s New in CrystalDiffract 6.8?

CrystalDiffract 6.8, released in January 2019, supports the new “Live Powder Diffraction” mode introduced in CrystalMaker 10.4. This includes changes to the way in which structural data are sent from CrystalMaker to CrystalDiffract, which allows both the new “**Live Powder Diffraction**” mode and a new “**Replace Pattern**” command.

CrystalDiffract 6.8 for Mac includes major changes designed to ensure optimum performance under macOS 10.14 “Mojave”. This includes new drawing code and support for “Dark Mode” (which required extensive modernisation of the user interface and other graphics updates). The application now uses Apple’s recommended “hardened runtime” for greater security.

Other changes include “spring-loaded sidebars”, haptic feedback (feel your peaks as you move the cursor over them), significantly-faster CMTX and CIF export, as well as an updated Help system.



Chapter 2

Getting Started

CrystalDiffract is a professional application available for Mac and Windows computers, with extensive support options and examples files. This chapter provides a brief introduction to the software, including system requirements, installation, and licensing.

A Note about the Demo Version

This User's Guide is designed for the *Full-Feature* version of CrystalDiffract.

If you are using the free, *Demonstration Version*, some features may not be available: this version is designed to give you a *flavour* of the full program, but you cannot save files, record program settings, or specify preferences. Neither can you import observed data files (although you can read such data if they have been saved in a diffraction experiment). However, you can explore the example files provided—which include saved diffraction experiments demonstrating many aspects of the full-feature version.

Using this Guide

This User's Guide should provide a very comprehensive outline of the major program features. We don't expect you to read it from cover-to-cover, but would recommend the following key sections:

- **All users** should browse *Chapter 2: CrystalDiffract Interface*. This is designed to provide a quick orientation to the program; the interface changes from version to version, and will certainly be different to other programs you may have used, so it's important to get your bearings earlier, rather than later!
- **If you are a new user**, we strongly recommend that you complete the Guided Tours section. The series of short, structural exercises is designed to illustrate some of the most important program features and should address most of the queries that you might have when using the software.

The User's Guide describes the program interface, followed by sections on simulating diffraction, how to put data *into* the program—then describing display

and manipulation, before finishing with how to get data *out* of the program: exporting data and graphics.

Searching for Topics

We have tried to provide a comprehensive yet logically-structured guide. If you need to find specific information, here are some suggestions:

- This guide includes a *Table of Contents* (at the beginning) and an *Index* (at the end).
- If you are viewing the guide electronically, you can click on the *Contents* or *Index* page entries to go directly to the corresponding pages.
- If you need to search for a keyword or phrase, you should be able to use the **Search** command in a PDF viewer such as Adobe (Acrobat) Reader or Apple Preview.

Interface Reference Convention

In the following chapters we refer to elements of the program’s interface (such as button names, menu commands and keys on your keyboard) using **this font**.

You will also encounter many references to menu commands written in an abbreviated manner, such as “**Edit > Copy**”, which means “from the **Edit** menu choose the **Copy** command”.

Mac & Windows Shortcut Keys

Mac and Windows operating systems use different key combinations for menu shortcuts (“accelerator keys”). In this guide we make repeated reference to **command** and **option** keys, which are included on the standard Mac keyboard. Windows users should use the following translation:

Mac	Windows
command	control
option	alt

System Requirements

CrystalDiffract is a modern 64-bit application, designed to take full advantage of modern operating systems. The Mac and Windows versions are separate applications, written from scratch, for their respective operating systems, using only the official development frameworks from Apple and Microsoft. This guarantees optimum performance and the best-possible user experience.



Mac

CrystalDiffract for Mac is a state-of-the-art 64-bit multi-processor, multi-touch *native* application. It is designed to take full advantage of recent Mac hardware and system software. The program requires:-

- 64-bit Intel-based Mac
- macOS 10.10 “Yosemite”, 10.11 “El Capitan”, 10.12 “Sierra”, 10.13 “High Sierra” or **10.14 “Mojave”** (recommended).

We regret that, owing to Apple’s relentless refinement of their operating systems, with regular and numerous changes to their application programming interfaces (API) we are unable to maintain backwards compatibility to older versions of macOS.



Windows

CrystalDiffract for Windows is currently available in two versions: the latest version (6.7), and a legacy version designed for users of Windows XP systems.

CrystalDiffract 6.7 requires:-

- 64-bit Intel-based PC
- Windows 7 (Service Pack 1), Windows 8 or **Windows 10** (recommended).
- **.NET** Framework 4.6 for later

A legacy version, **CrystalDiffract 6.5**, provides support for:-

- Windows XP (Service Pack 3).

Windows users are reminded that Windows XP support is limited: this is a legacy operating system that is no longer supported by Microsoft. We cannot guarantee the reliability of this software running on this operating system in the future.

Please note that no versions of CrystalDiffract will run on earlier versions of Windows, such as NT or 2000.

Installation

Mac and Windows versions have different installation procedures: Mac users can simply drag-and-drop files, whereas Windows users will need to run an installer program.



Mac Installation

This is a simple matter of dragging-and-dropping the CrystalDiffract application from a downloaded zip file, to your hard disc (e.g., to your *Applications* folder). As a modern Mac application, CrystalDiffract includes all its essential resources (including online help and this User's Guide), neatly packaged within the application "bundle." We would also recommend that you copy the *Examples Files* to your hard disc—possibly to your own *Documents* folder.



Windows Installation

Windows users will need to run the installer program. This gives the option of installing the essential program files (application, online help, user's guide), plus supporting resources (examples files).

Licensing your Installation

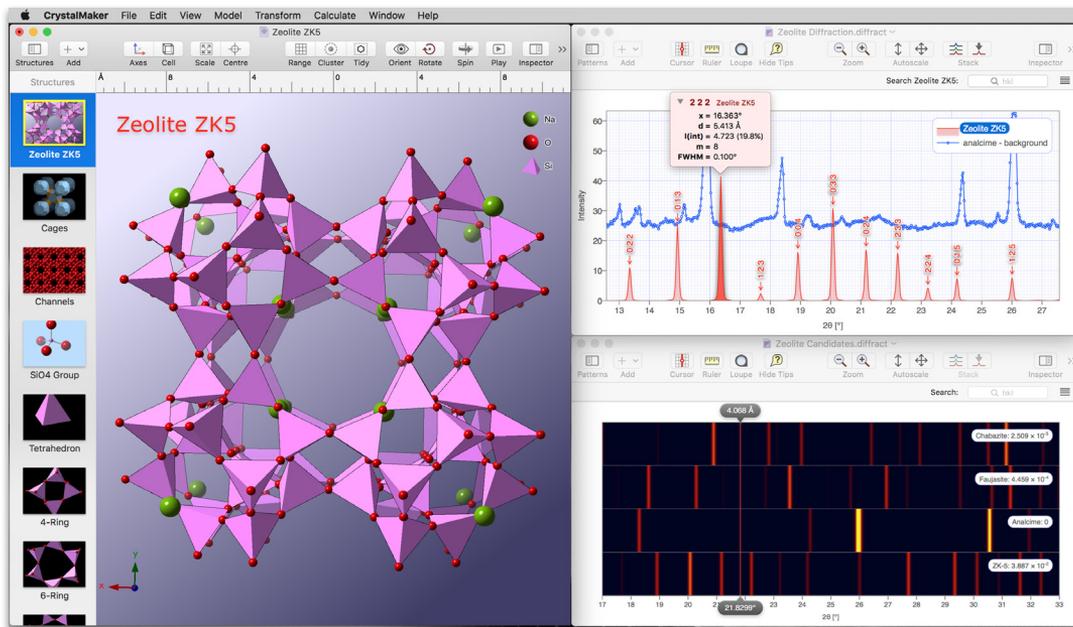
The first time you launch CrystalDiffract you are prompted to personalize your copy of the program. This process also creates a preferences file.

Registering Your Licence

It is very important that your licence is registered as we can only provide technical support (and upgrades) to registered users. You can register when you install the software, by clicking the **Register** button in the reminder dialog that appears following your installation. Alternatively, you can register later, by choosing the **Help > Register CrystalDiffract** command.

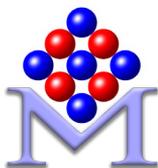
Multi-User Licence Registration

We only require one registration per licence. So, if you have a multi-user licence, such as a Group or Site Licence, only the official "keeper" of the licence needs to register with us. Once we have received that registration, the other users are entitled to receive technical support, within the terms of the specific licence.



Zeolite crystal structures displayed in CrystalMaker X (left) with corresponding diffraction patterns in CrystalDiffract (right).

CrystalMaker X Integration



CrystalDiffract versions 6.7 and later are designed to work seamlessly with CrystalMaker® X: an award-winning program for crystal and molecular structures modelling.

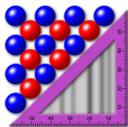
CrystalMaker provides seamless display of data files from major databases and supports a wide variety of file formats. Just drag-and-drop a text file into CrystalMaker for automatic format detection and structure display. CrystalMaker lets you display a structure then, with a single menu command, see its diffraction pattern appear in CrystalDiffract.

Further information about CrystalMaker X is given at the end of this guide, or you can visit crystalmaker.com/crystalmaker and download a free demo version.

Garbage In, Garbage Out?

Although CrystalDiffract allows you to build your own crystals, or import data from CIF files, it is important to verify that the structures are reasonable (and correct!).

CrystalMaker makes it easy to verify a structure, with instant display and automatic bond searches. The combination of CrystalMaker and CrystalDiffract gives you a great crystallographic toolbox.



Chapter 3

The CrystalDiffract Interface

CrystalDiffract is designed to provide a seamless drag-and-drop workflow, with an easy-to-use interface and real-time parameter control. This chapter provides a detailed overview of CrystalDiffract's interface, so you can work with multiple patterns in the same window, access tools and adjust parameters.

Window Design

CrystalDiffract has a single-window program interface with a toolbar, and a Graphics pane for plotting your diffraction patterns. Three (optional) sidebars let you view a list of patterns, a list of reflexions (for the selected pattern), and an Inspector with formatting and parameter controls.

Toolbar

At the top of each window is a toolbar with buttons for measuring and manipulating diffraction patterns. The toolbar can be customized to include additional buttons for operations such as scrolling or resetting offsets.

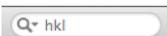
To show or hide the toolbar:

Do one of the following:

- Choose: **View > Layout > Show/Hide Toolbar**.
- Right-click in the toolbar and choose: **Show/Hide Toolbar** from the contextual menu.

To customize the toolbar:

- Right-click in the toolbar and choose: **Customize Toolbar** from the contextual menu.

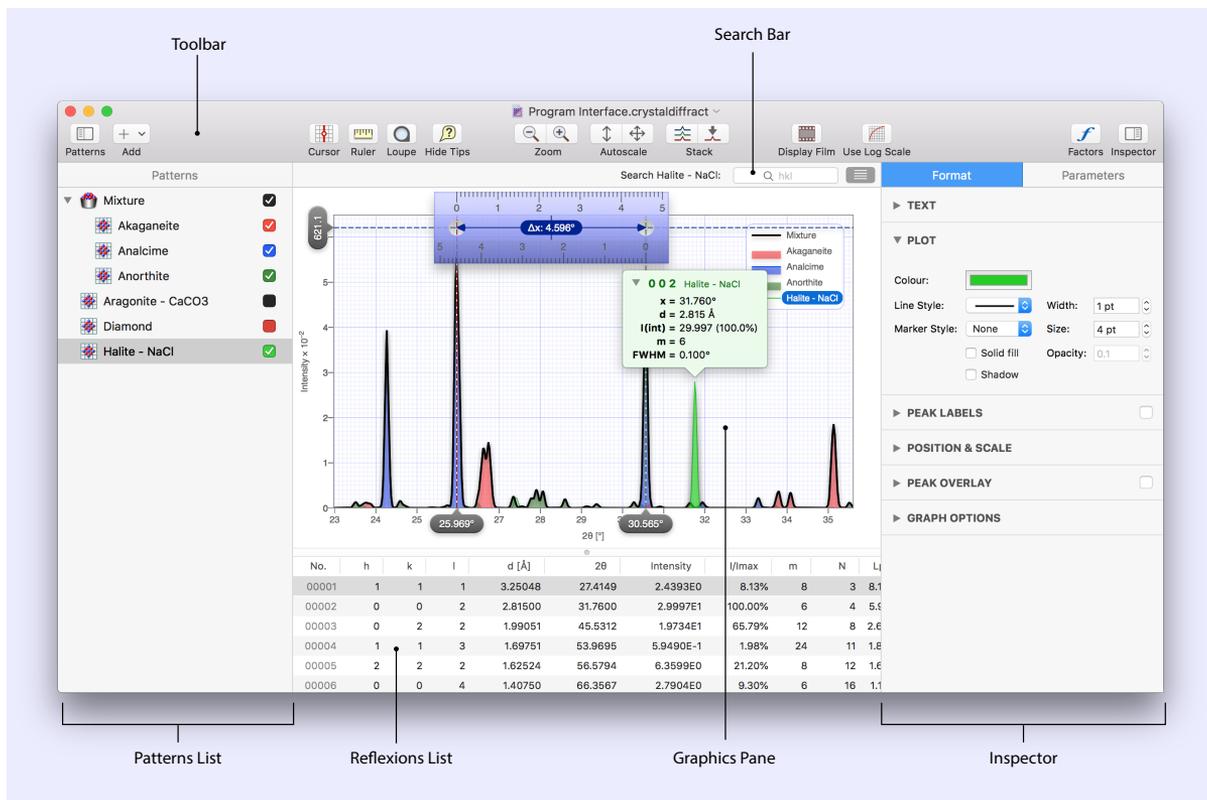


Search Bar

Immediately below the toolbar is a search bar for the *currently selected* diffraction pattern (which must be a simulated—crystal— pattern). You can enter the Miller

indices for the reflexion and press the return or enter keys on your keyboard to locate that reflexion in the Graphics Pane below.

Selecting a Pattern. You can select a pattern by clicking its name in the Structures List—or by clicking on the “Search” title (the text to the left of the Search field) and choosing a pattern to search from the popup menu that appears.



The CrystalDiffract interface (here shown running on OS X 10.10 “Yosemite”).

Graphics Pane

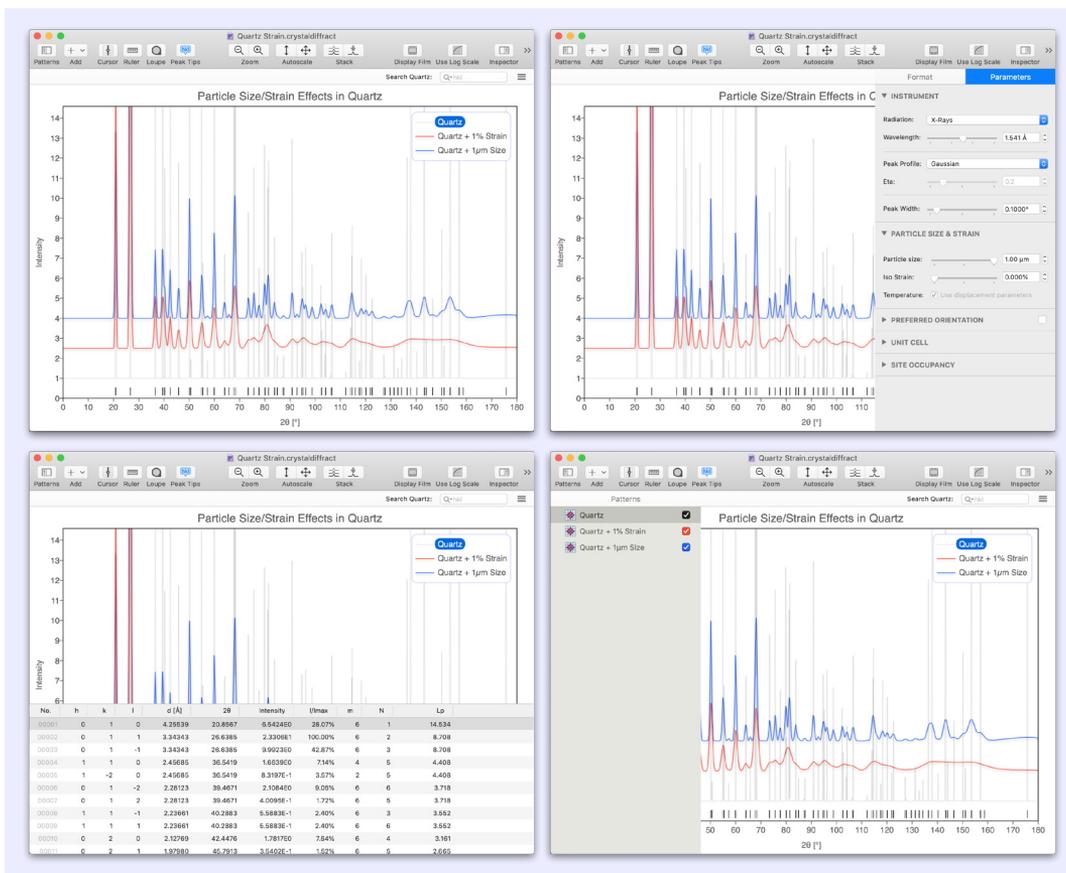
Diffraction patterns are plotted in the Graphics Pane. This does not initially have visible scrollbars, but you can use your mouse to click-and-drag the pattern horizontally. When you do this, a scroller view becomes visible, with a preview of the full pattern and a “thumb” control which you can resize or drag with the mouse.

If your computer has a trackpad, you can use standard multi-touch gestures for scrolling and scaling.

Spring-Loaded Sidebars

As an alternative to keeping the various sidebars open, you can instead have them slide into view only when required: simply move the mouse towards the appropriate edge of the window and pause. For example, to show the Patterns List you would move the mouse towards the left-hand side of the Graphics Pane and hover; beyond a certain “trigger point” (about 15 pixels from the edge) the Patterns List will then slide into view, over the Graphics Pane. Moving the mouse back over the Graphics Pane (and outside the sidebar) will cause the sidebar to slide back, out of view.

Haptic Feedback. Mac users with force-touch trackpads (and with “Haptic Feedback” enabled in the Trackpad pane of their System Preferences panel) will feel a slight vibration or “buzz” as they pass over the “trigger point”.



Spring-Loaded Sidebars in Action: Users with small screens can opt to work without any sidebars visible (top left). Moving the mouse towards the appropriate window edge causes a sidebar to slide into view, over the structure, as seen for the Inspector (top right), Reflexions List (bottom left) and Patterns List (bottom right). Moving the mouse outside the sidebar and back over the structure causes the sidebar to slide back out of view.

Reflexions List

You have the option of displaying a list of *simulated* reflexions below the Graphics Pane. This Reflexions List shows data for the *selected* pattern. You can sort the list by clicking on one of the column headings; click a second time to reverse the direction of the sort. To locate a particular reflexion on the graph (or film), double-click its corresponding entry in the list.

To show or hide the Reflexions List:

Do one of the following:

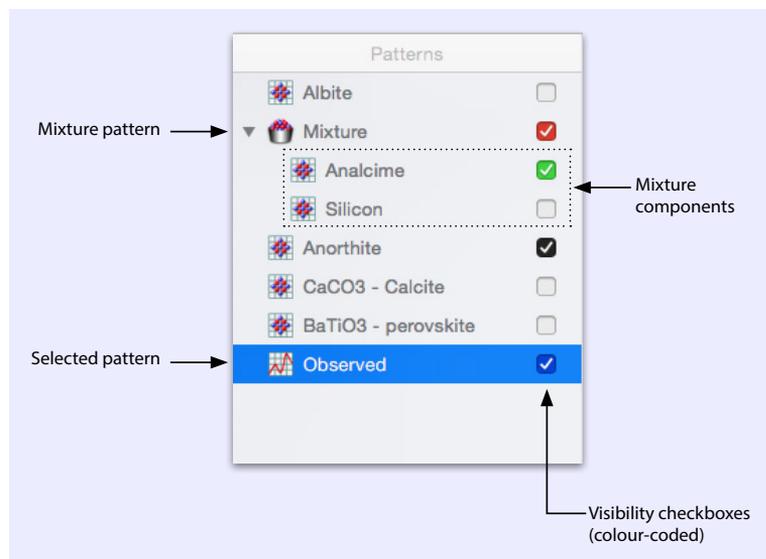
- Choose: **View > Layout > Show/Hide Reflexions List.**
- Click the Reflexions List button (☰) in the search bar.

Save Your Reflexions: To save the contents of the reflexions list as a text file, choose: **File > Export > Reflexions List.**

Patterns List

You can work with multiple diffraction patterns in the same window; these are listed in the Patterns List. You can drag text files, CrystalMaker crystal files and folders into this list. The corresponding diffraction patterns can be plotted by clicking their checkboxes.

The Patterns List.



Displayed patterns can be *selected* by clicking on their Patterns List entries. You can drag selected patterns to rearrange the list of patterns (this affects the order in which patterns are plotted: the topmost pattern is plotted last, and so will appear

“above” other patterns). Selection also allows you to edit pattern parameters, using the Parameters Inspector (described overleaf).

The Actions *contextual menu* lets you reset pattern colours, sort the list, and create mixtures from selected patterns. To display the contextual menu, right-click (control-click on Mac) anywhere in the Patterns List. You can resize the list by clicking-and-dragging the thin divider line between it and the Graphics Pane.

To show or hide the Patterns List:

Do one of the following:

- Click the **Patterns** button in the toolbar.
- Choose: **View > Layout > Show Patterns List**.

To add patterns to the list:

Do one of the following:

- Click-and-drag one or more diffraction files from your operating system, into the Patterns List.
- Click the toolbar’s **Add** button and choose: **Add Pattern from File** from its popup menu.
- Choose: **File > Open in Same Window**, then use the navigation dialog to specify the file you want to add.

To show or hide a pattern:

- Use the pattern’s visibility checkbox to toggle its plot state.

To delete a diffraction pattern:

- 1 Select the patterns you wish to delete by shift-clicking on them.
- 2 Right-click in the Patterns List to display its contextual menu and choose: **Remove**.

To duplicate a diffraction pattern:

Do one of the following:

- Hold down the **option/alt** key, then click-and-drag the pattern to duplicate it.
- Select a pattern, then choose: **Duplicate** from the Patterns List contextual menu.

Inspector

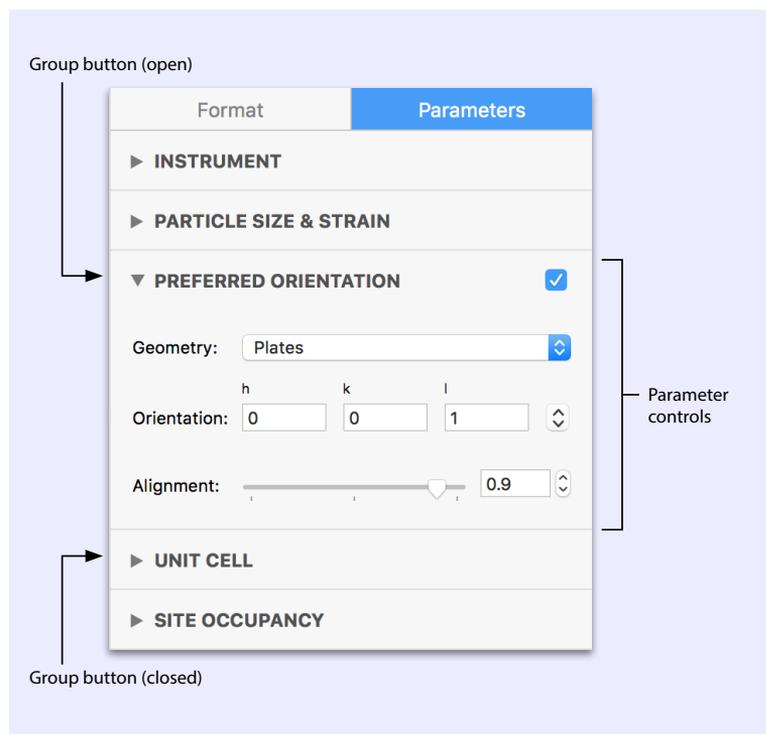
The Inspector provides contextual editing controls for formatting and diffraction parameters.

The **Format** Inspector lets you change the way in which your diffraction patterns are plotted, with controls for colours, line and marker styles, scaling offsets and labelling.

The **Parameters** Inspector provides interactive control over all aspects of your diffraction experiment, including wavelength, peak broadening, particle size and strain, preferred orientation—and even lattice parameters and site occupancies. You will also find controls for processing observed data, including smoothing and background subtraction.

Controls are arranged in “disclosure groups”. Click a group button to reveal the controls available. You can edit parameters interactively, using slider controls, and observe how the diffraction pattern changes.

The Inspector, showing the Parameters tab selected.



Displaying a Diffraction Pattern

CrystalDiffract can read from observed data files, crystal structure files, legacy diffraction session files (generated by the previous version of the program) as well as current CrystalDiffract documents.

To load a file in a new window:

Do one of the following:

- Drag-and-drop a file onto the CrystalDiffract application icon;
- Drag-and-drop a file into an empty CrystalDiffract window.
- In CrystalDiffract, choose **File > Open** then use the file dialog.
- Drag-and-drop a file into the Patterns List, then click the new entry's checkbox.

To open a file in an existing window:

Do one of the following:

- Choose: **File > Open in Same Window**.
- Drag-and-drop your file(s) into the window: either the Graphics pane, if you want its diffraction pattern displayed immediately; or into the Patterns List if you wish to defer plotting until you have clicked the pattern's checkbox.
- Click the toolbar's **Add** button and choose: **Add Pattern from File** from its popup menu.

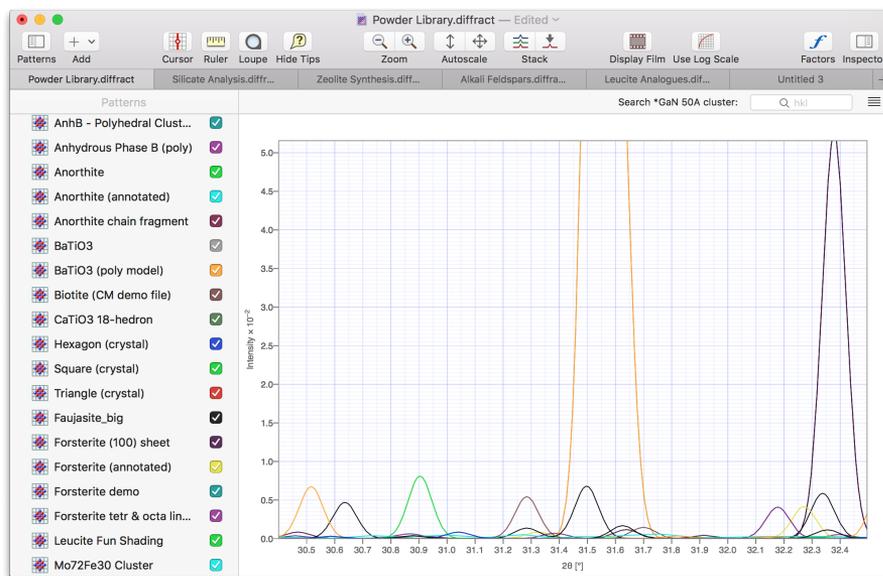
Working with Multiple Documents or Tabs

CrystalDiffract for Mac lets you tile or stack multiple windows to help tidy up your display—these commands are available on the Window menu.

In addition, if you are using macOS 10.12 “Sierra” or 10.13 “High Sierra”, you can collapse multiple windows into a single, tabbed window, using the **Window > Merge All Windows** command.

New tabs can be created using the “+” button on the right-hand side of the resulting tab bar. Existing tabs can be torn off to make new windows, or moved into other diffraction windows.

Document tabs for macOS Sierra or High Sierra.



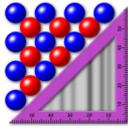
Help and Updates

Many controls have tool tips associated with them. For detailed help, choose the **Help > CrystalDiffract Help** command. Help files are displayed in a Help Viewer application (Mac) or browser window (Windows). We also include a number of support topics on our website, and links to these are included on the program's **Help** menu.

Checking for Updates

We provide regular, free, incremental program updates during the lifetime of the product. These include new features, interface enhancements and occasional bug fixes.

You can check whether an update is available using the **CrystalDiffract > Check for Update** command (on the Windows version, this command is located on the **Help** menu). The program will alert you if a newer version of the software is available. You can then download this from our website.



Chapter 4

Principles of Diffraction

CrystalDiffract can simulate an x-ray or neutron powder diffraction pattern for any crystal structure, using its tabulated atomic scattering factors and neutron scattering lengths. This chapter introduces the principles of diffraction and outlines the various different types of powder diffraction experiments. This is followed by a summary of how the powder profile is calculated.

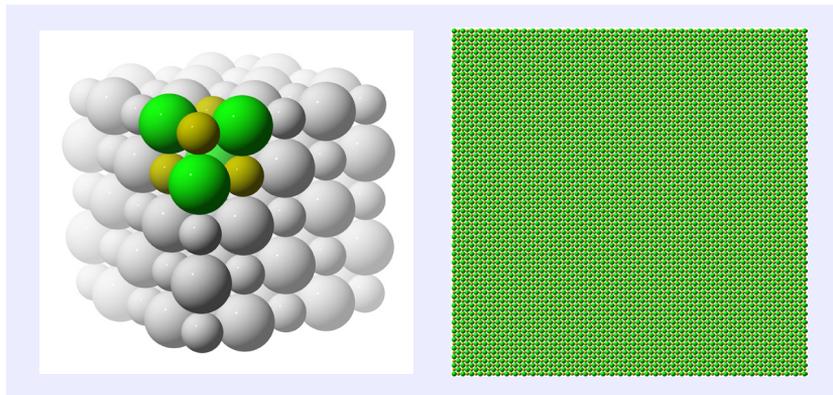
Crystals, Powders, Diffraction

CrystalDiffract is a specialised tool for understanding the process of diffraction from powdered crystalline samples. We need to understand what we mean by “crystalline”, and then explore why it is necessary to grind crystals into powders.

Crystalline Materials

The starting point for simulating a diffraction pattern is a crystal structure: the regular arrangement of atoms inside a basic building brick: the “unit cell”. Crystals typically contain billions of unit cells forming a three-dimensional lattice.

A crystal structure is derived from a basic unit (left) that is tiled in 3D to form an extended lattice (right)—as illustrated here for the mineral halite (NaCl).



It is the very regularity of such structures that allows diffraction in the first place. The precisely-oriented planes of atoms, repeated almost *ad infinitum*, provide miniature diffraction gratings for X-ray or neutron radiation.

Why Powder Diffraction?

Powder diffraction has a number of advantages over single-crystal techniques. Sometimes it is difficult to find (or grow) good quality single crystals, whereas powders are much easier to manage. Single-crystal diffraction (using X-rays or neutrons) is quite an arduous process, requiring precise orientation of the sample (or, in the case of electron microscopy, specially-prepared, thin crystal flakes). Data collection tends to be very slow, as individual scattered beams are measured (although new, area detectors, are making this faster).

With powder diffraction, one has the advantage of speed and convenience. A powdered sample has multiple “crystallites” and, assuming these are randomly distributed, at least one crystal will be oriented correctly to cause diffraction. Data collection times tend to be faster, since only a “one-dimensional” pattern is being collected.

The most-important powder diffraction techniques—which can be simulated by CrystalDiffract—are described below.

Angle-Dispersive Diffraction

In most laboratories, X-rays are generated by firing a beam of electrons at a metal target—usually copper (Cu) or molybdenum (Mo). A characteristic X-ray spectrum is emitted, which is filtered, so that only the strongest, Cu $K\alpha$ peak emerges (this is actually a doublet, comprising $K\alpha_1$ and $K\alpha_2$ peaks, although sometimes the weaker, $K\alpha_2$ peak is also filtered out). This *monochromatic* radiation (with wavelength λ) is then directed at the specimen.

The same can be done for neutron radiation—although not, generally, in the laboratory. Instead, the experiment might be carried out at a nuclear reactor, with the constant-wavelength neutron beam directed from the reactor, to the sample.

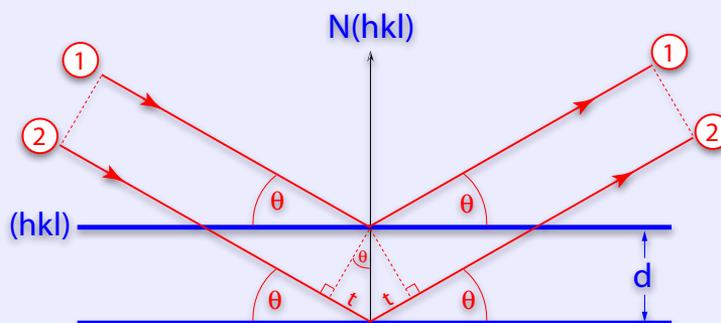
One typically moves the beam, relative to the sample, scanning through a range of angles, θ . There is a reciprocal relationship between θ , and inter-planar distances in the crystal (“d-spacings”), which give rise to diffraction peaks. This is summarized in the famous Bragg Equation:

$$\lambda = 2 d \sin \theta$$

which provides the condition for coherent scattering of the radiation (wavelength λ), directed at an angle θ (the Bragg Angle) with respect to the d-spacing of a set of planes in the crystal.

By measuring scattered intensity as a function of scattering angle, one is in effect measuring the scattering strengths of different sets of planes (with different d-spacings) inside the crystal. Ultimately, this scattering strength is controlled by the arrangements of atoms in different directions in the crystals—and hence one can learn something about the crystal structure from its diffraction properties.

Derivation of the Bragg Equation



Consider a crystal with a set of planes, (hkl), shown here in blue. The interplanar spacing is denoted by d , and the plane normal is $N(hkl)$.

If a beam of monochromatic radiation (wavelength λ), shown here in red, strikes these planes at a glancing angle, θ , then constructive interference between adjacent

wavelets ① and ② occurs when their path difference ($t + t$) is equal to an integral number of wavelengths.

Thus, $n \lambda = 2 t$

where: $t = d \sin \theta$

hence, $n \lambda = 2 d \sin \theta$ (the Bragg Equation).

Energy-Dispersive X-Ray (EDX) Diffraction

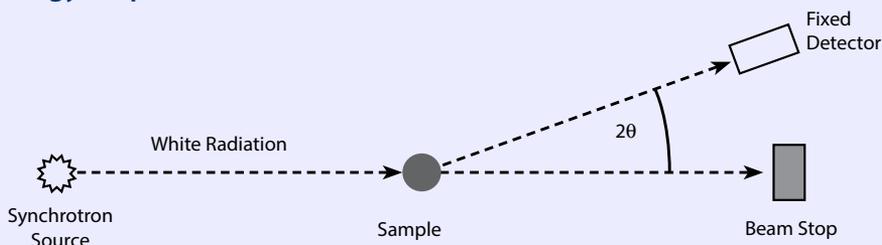
EDX experiments are typically conducted at synchrotron sources. Here, charged particles are accelerated to relativistic speeds, and emit x-rays as they travel around a curved beam path. So-called “White Radiation”, comprising a broad spread of wavelengths, can be generated; this is useful in diffraction experiments because it allows *rapid* measurements, without the need to mechanically scan a detector over a range of angles.

The spaceship-like building housing the new “Diamond” synchrotron facility, at the Rutherford-Appleton Laboratory, near Oxford, England.



From the Bragg equation, $\lambda = 2 d \sin \theta$ (introduced in the previous section), we see that if the wavelength, λ , is varied, then diffraction from a range of d -spacings can be recorded at the *same* θ angle. However, in order to resolve diffraction from different d -spacings, the stationary detector must be able to discriminate between scattered radiation of different *wavelengths*. This is possible because different wavelengths have different *energies* (see box):

Energy-Dispersive Diffraction Basics



In a typical EDX experiment, white radiation is scattered by the sample, and a portion of this is recorded by a stationary detector at a fixed angle, 2θ , from the beam direction.

Since X-rays are a form of electromagnetic radiation, we can relate their energies (E) to their wavelengths, λ using Planck's equation:

$$E = h f$$

(h is Planck's constant and f is frequency).

Rewriting this using the speed of light, c :

$$E = h c / \lambda$$

Using the Bragg Equation ($\lambda = 2 d \sin \theta$):

$$E = h c / (2 d \sin \theta)$$

Because our Bragg angle, θ , is fixed, the only variables here are energy, E , and d . Thus, in an EDX experiment, the energy of a scattered reflexion is inversely proportional to the d -spacing.

Thus, an *energy-dispersive* detector can be used to record an extended diffraction pattern, as a function of energy.

Time-of-Flight Diffraction

Some neutron diffraction experiments use yet another kind of diffraction: a neutron spallation source creates pulses of neutrons with a range of energies. These travel at different speeds, depending on the energy of the neutrons, and are directed down a long "beam line" towards a powder sample.

Diffraction is recorded by neutron detectors arranged around the sample, at a fixed two-theta angle (2θ). The number of pulses is recorded as a function of the time-of-flight, t , of the neutrons (which is typically in the range of a few milliseconds to several hundred milliseconds). As for energy-dispersive diffraction, an extended diffraction pattern can be recorded at a fixed Bragg angle because the sample is subjected to neutrons of different energies, and hence wavelengths.

We can analyse the time-of-flight process by combining De Broglie's hypothesis with Bragg's Law (see box). This gives a linear relationship between the total time-of-flight and the overall flight path. This is why the highest-resolution neutron diffractometers have the longest flight paths (~100m for the HRPD instrument at the Rutherford-Appleton Laboratory).

The ISIS neutron spallation source at the Rutherford-Appleton Laboratory, Oxford, England. Neutrons are generated at the target (the light-blue chamber at the centre of the photo) and directed radially, along beam lines.



Time-of-Flight Diffraction Basics



Consider a typical neutron diffraction experiment with a primary flightpath (the distance from the moderator to the sample) of L_1 and a secondary flight path (sample to detector) of L_2 .

De Broglie's hypothesis relates wavelength, λ , to particle mass & velocity, via Planck's constant, h . For neutron diffraction,

$$\lambda = h / m_n v_n$$

(m_n = neutron mass; v_n = velocity).

Bragg's Law states: $\lambda = 2 d \sin \theta$

Combining these,

$$\lambda = h / m_n v_n = 2 d \sin \theta$$

If the corresponding times of flight are t_1 and t_2 , we have:

$$v_n = (L_1 + L_2) / (t_1 + t_2) = L / t$$

(L = total flight path; t = total time-of-flight)

$$\text{Thus, } h t / m_n L = 2 d \sin \theta$$

$$\text{hence: } t = 2 d L (m_n / h) \sin \theta$$

So the total time-of-flight, t , is proportional to the total flight path, L .

Intensity Simulation

CrystalDiffract calculates diffraction patterns using the types, and positions, of atoms in a unit cell of a crystal. The program assumes an ideal structure (although you can specify size and strain effects later).

Structure Factor

The first step is to calculate the amplitude scattered for each reflexion: the *Structure Factor*, $F(hkl)$. This is derived by summing the amplitudes scattered by each atom in the unit cell, e.g., for X-ray diffraction we have:

$$F(hkl) = \sum_{n=1}^N f_n \exp\{2\pi i (h x_n + k y_n + l z_n)\}$$

Where hkl are the Miller indices of the reflexion, f_n is the *atomic scattering factor* for the n 'th atom in the unit cell, and $\{x_n, y_n, z_n\}$ are its fractional coordinates. These coordinates are assumed to be at rest; in most real structures this isn't the case, and atomic motions are generally described by *atomic displacement parameters* ("thermal ellipsoids"). If these data are present, CrystalDiffract will apply them to the structure factor calculation.

The *atomic scattering factor* depends on the nature of the atom. If your structure is disordered, then CrystalDiffract will use the site occupancy values to determine an average atomic scattering factor (or an average *scattering length*, in the case of neutron diffraction). We shall explore these factors in the next section.

Saving Structure Factors: You can export a table of Structure Factors, via the **File > Export > Structure Factors** command.

Intensity

You may have noticed that the Structure Factor is a complex number: it has amplitude and *phase*. Sadly, it is not possible to record the *phase* of scattered beams in a diffraction experiment—otherwise, analyzing diffraction data would be a much simpler process! Instead, what is measured is the *intensity* of scattering. This is proportional to the square of the amplitude of the Structure Factor, i.e.,

$$I(hkl) \propto |F(hkl)|^2$$

The complete calculation of the intensity needs to take into account other factors, including geometrical considerations and polarization effects, so we write:

$$I(hkl) = s \cdot Lp \cdot |F(hkl)|^2$$

Here, s is a constant that would depend on factors such as the intensity of the

radiation beam and sample absorption effects. The factor Lp comprises the Lorentz factor, and the *polarization* factor, which are described below.

Constant Volume: The Intensity calculation takes into account the density of your structure. Intensities are calculated on the basis of a fixed *volume* of material, allowing you to directly compare different materials which might have very different densities.

Polarization Factor

The polarization factor is relevant for X-ray diffraction, and relates to the fact that X-rays are a form of electromagnetic radiation, with a plane of polarization. The direction of polarisation of an X-ray photon can change as a result of scattering: in general, the polarisation resolved along the new (diffracted) direction is reduced by the cosine of the scattering angle (i.e., $\cos 2\theta$)—but since intensity is proportional to the square of amplitude, the factor to be applied to our calculated intensity becomes:

$$p = \cos^2 2\theta$$

So, for fully-polarized X-rays, at $2\theta = 90^\circ$ we would get no scattering.

In the specific case that the polarisation direction is parallel to the scattering planes, there is no effect, i.e.,

$$p = 1$$

Since laboratory X-ray sources produce unpolarised X-rays (i.e., X-rays polarised equally in all directions), we need to take an average of the two extreme cases:

$$p = (1 + \cos^2 2\theta) / 2$$

where p is our *polarization factor*.

Lorentz Factor

Considering the “cones” of scattered radiation from a powder sample, then a detector of finite aperture will have the potential to count more photons at low (or equally at high) angle, and less as the scattering angle approaches 90° .

The amount of the cone measured is inversely proportional to the sine of the scattering angle 2θ , i.e.,

$$1 / \sin 2\theta .$$

A second term relates the probability of the crystallites having a plane in the correct orientation for Bragg scattering, and this is inversely proportional to the sine of the incident Bragg angle θ , i.e.,

$$1 / \sin \theta$$

Hence we get the equation,

$$L = 1 / (\sin \theta \cdot \sin 2\theta)$$

where L is our *Lorentz factor*.

Electron Diffraction

This factor must be modified slightly for electron powder diffraction, where the sample is expected to be static, and not rotating (as for x-ray or constant-wavelength neutron diffraction).

Lp Factor

Combining the *Lorentz* and *polarization* factors, we can write:

$$Lp = (1 + \cos^2 2\theta) / (2 \sin \theta \cdot \sin 2\theta)$$

Where Lp is the *Lorentz-polarization* factor.

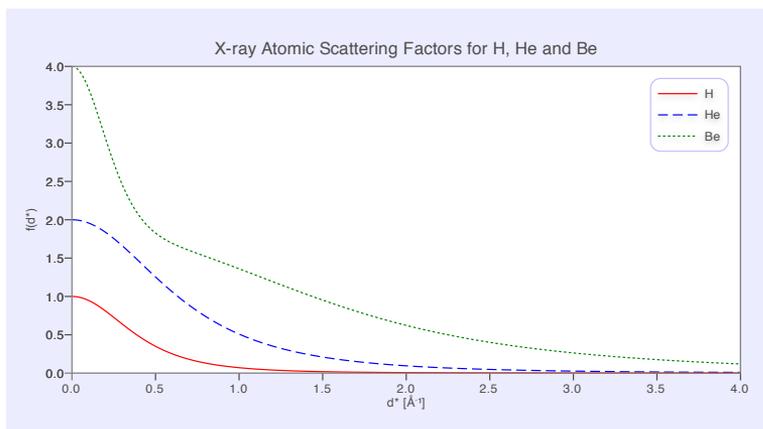
Atomic Scattering Factors

The amplitude scattered by a single atom is measured by its *atomic scattering factor* in the case of X-ray diffraction, or its *neutron scattering length* in the case of neutron powder diffraction.

Neutron scattering lengths represent the amplitude scattered by the *nucleus* of the atom—and since this is so small in comparison with the size of an atom, the amplitude does not vary with the scattering angle, θ .

By contrast, X-ray scattering is highly angle dependent. X-rays are scattered by the electron cloud, and so a “head on” X-ray photon experiences more electron density than a photon that approaches at an oblique angle. One therefore sees a fall-off in amplitude with increasing scattering angle, as shown graphically when plotted as a function of reciprocal d-spacing, d^* , where $d^* = \lambda / 2 \sin \theta$:

X-ray scattering factors for selected elements, as a function of reciprocal d-spacing (and hence scattering angle).

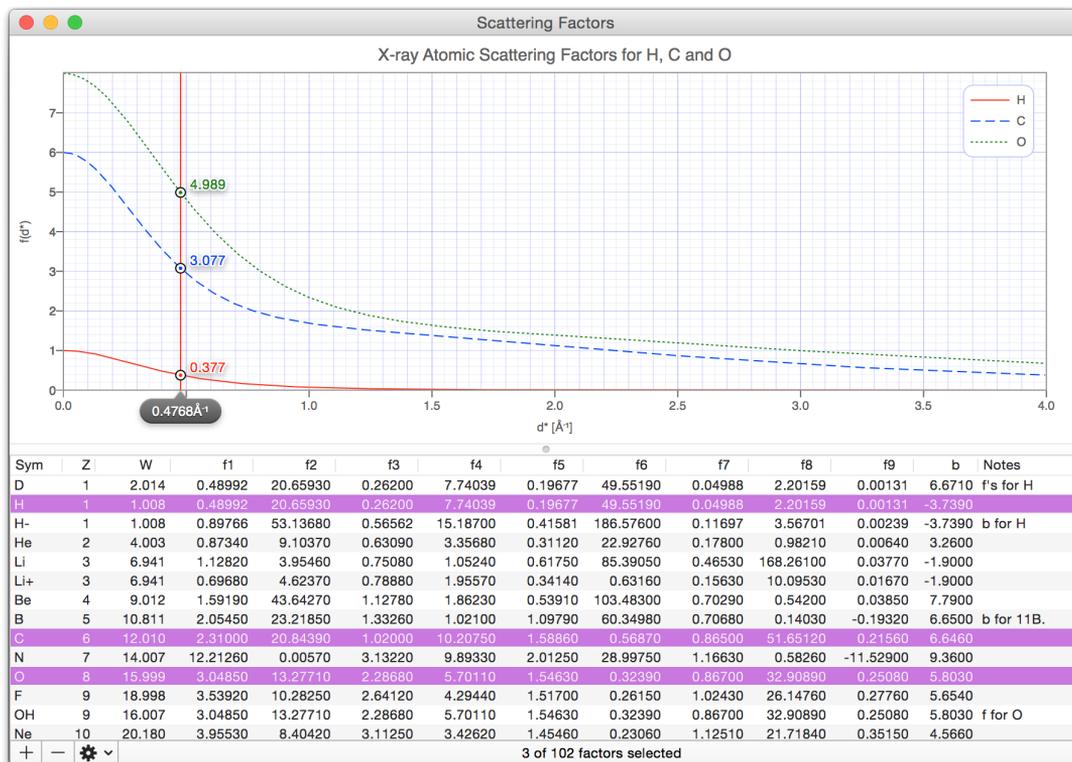


We also see a dependence on the atomic number (i.e., the number of electrons in the atom). Thus, smaller atoms (e.g., H) with fewer electrons scatter less than heavier atoms (e.g., He or Be) with more electrons. This contrasts with neutron diffraction, where there is no simple relationship between scattering lengths and atomic numbers.

Atomic scattering factors and neutron scattering lengths are tabulated in the Scattering Factors window (**Edit > Scattering Factors**). The form of the X-ray scattering factor curve can be represented by a 9-coefficient polynomial, the values $f_1 - f_9$ in the Scattering Factors table. CrystalDiffract uses the standard analytical approximation defined in the *International Tables for Crystallography, Volume C*:

$$f = \sum_{i=1}^4 a_i \exp(-b_i \sin^2 \theta / \lambda^2) + c$$

where f is the value of the atomic scattering factor for a particular $\sin \theta / \lambda$ and a_i , b_1 , a_2 , b_2 , a_3 , b_3 , a_4 , b_4 and c are coefficients. These are tabulated for different elements in the scattering factors table as the variables $f_1 - f_9$, respectively (i.e., $a_1 = f_1$; $b_1 = f_2$; etc.), to avoid confusion with the neutron scattering length, which is commonly represented by the variable b .



The Scattering Factors window, highlighting atomic scattering factors for the elements H, C and O.

The Powder Diffraction Profile

We now have all of the data required to simulate a diffraction pattern: crystal structure, atomic scattering factors (or neutron scattering lengths), the Lorentz factor and the polarization factor.

Diffraction in One Dimension

We can generate intensities for all reflexions in the crystal, up to a pre-determined maximum limit. However, this is effectively a three-dimensional diffraction pattern—not a powder pattern.

The next stage is to merge the 3D pattern into a “one-dimensional” pattern: a *profile* of intensity as a function of d -spacing, and plotted versus your choice of x -axis (e.g., 2θ , $1/d$, time-of-flight, etc.).

Multiplicity

Reflexions with the same d -spacing will end up overlapping in this one-dimensional pattern. CrystalDiffract will *merge* those reflexions which are *symmetry-related* (e.g., the six planes of the form $\{100\}$ for a cubic crystal), but will keep non-symmetry-related reflexions separate, even if they have the same d -spacings.

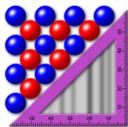
The *multiplicity* (m) of each “reflexion” is the number of symmetry-related reflexions that have been combined to give a single peak. In the case of our cubic example, the multiplicity of a “100” peak is now 6. You can inspect a table of “merged” reflexions in the Reflexions List.

Check Your Symmetry! If you find multiple reflexions with related indices listed for the same d -spacing (e.g., 100 and 010 in a cubic or tetragonal crystal), it is likely that your crystal structure is missing symmetry—most probably, because a default “P 1” spacegroup has been entered, instead of the correct spacegroup. In this case you may want to edit the spacegroup setting in the Crystal Editor, to ensure that your reflexions are correctly merged.

Peak Function

The final step is to plot the results. This requires taking into account the instrumental resolution and peak shape: giving each diffraction peak a characteristic shape and width—possibly modified by sample strain and/or size effects. This results in the diffraction “graph” or “film” shown in the Graphics pane.

In the next chapter, we shall see how you can control the diffraction process.



Chapter 5

Simulating Diffraction

CrystalDiffract gives you extensive control of the simulation of x-ray and neutron powder patterns. You can edit crystal structures directly, change atomic scattering factors, choose different simulation types, and adjust sample and experimental parameters in real time.

This chapter describes how you can control the simulation of a powder pattern for a crystal structure, including the simulation *type*, the *x*-axis display, *wavelength*, *peak shape*, *instrumental broadening* plus *particle size* or *strain* effects. You can also edit the *atomic scattering factors* and *neutron scattering lengths*.

Crystal Structure

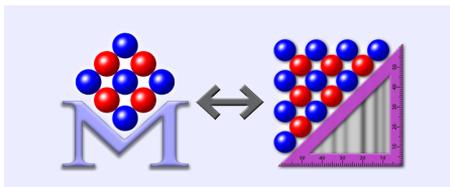
Diffraction relies on the principle of *periodicity* (i.e., lattice planes act as microscopic diffraction gratings). That means that diffraction simulations require you to provide a *crystal* structure—not merely a single molecule isolated in space. You have various ways of supplying the crystal data: either directly, via CrystalMaker; or indirectly, via a saved file. You also have the option of entering crystal data directly into the program.

Sending a Crystal from CrystalMaker X

CrystalMaker® X, our award-winning crystal and molecular structures program, lets you visualize a crystal structure on screen, and send its structure directly to CrystalDiffract, via its **Calculate > Powder Diffraction** submenu.

This gives you the best of both worlds: you can visualize (and check!) the structure in real space, and simulate intensities in diffraction space.

Crystals can be shared between CrystalMaker X and CrystalDiffract.



You can opt to generate a new pattern, replace the frontmost pattern (in the frontmost CrystalDiffract window), or enter a “Live Diffraction Mode” in which any changes to the crystal (in CrystalMaker) cause the (frontmost) diffraction pattern in the corresponding CrystalDiffract window to be updated.

Adding Patterns to Existing Windows: Unlike earlier versions of CrystalMaker and CrystalDiffract, diffraction patterns are now added to the frontmost CrystalDiffract window (instead of a new window being opened up each time CrystalMaker requests a new pattern). If you require a new window, please open this in CrystalDiffract before issuing a powder diffraction command from CrystalMaker.

Sending a Crystal to CrystalMaker X

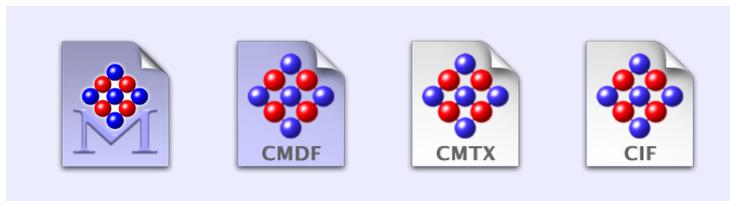
You can also send the crystal structure of any simulated diffraction pattern *back* to CrystalMaker, by using CrystalDiffract’s **Pattern > Visualize Crystal Structure** command.

Loading a Saved Crystal

CrystalDiffract can read from crystal structures saved as CrystalMaker® X documents (file extension `.cmdx`), CrystalMaker® 7–9 (legacy) documents (file extensions `.crystal` or `.cmdf`), or as text files in CMTX (CrystalMaker Text), STRUPLO or the ubiquitous CIF formats. You can load your crystal in the usual way: either by adding directly to the Graphics pane, or by dropping into the Patterns List, or by using the **File > Open** command to open your crystal in a new window.

CIF Warning: Please be aware that CIF files are highly fragile and prone to errors, especially with regard to spacegroup symmetry settings. We strongly recommend that you verify the structure first, in CrystalMaker, before relying on a simulated diffraction pattern—particularly if your CIF file was generated by a third-party program.

Some of the crystal file formats supported by CrystalDiffract.



Creating a New Crystal

You can create a new crystal structure directly in CrystalDiffract. This is a great way to learn about the diffraction process, as you have full control over the structure and can observe how the resulting diffraction pattern changes.

To create a new crystal

Do one of the following:

- ◆ Click the toolbar's **Add** button, then choose: **New Crystal Pattern** from its popup menu.
- ◆ Choose: **File > Add New Pattern > Simulated Crystal**.

CrystalDiffract will build you a dummy crystal and simulate its diffraction pattern, presenting you with a sheet or dialog in which you can edit the structure.

The Crystal Editor showing spacegroup, lattice parameters and site data.

Spacegroup: Origin:

Monoclinic spacegroup (No. 14): Unique axis b; cell choice 1; 4 general equivalent positions.

Lattice Parameters: a [Å] b [Å] c [Å] α [°] β [°] γ [°]

Atoms in the Asymmetric Unit:

On	Label	Occupancy	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂	Uiso	Type
<input checked="" type="checkbox"/>	C(1)	C _{1.0}	0.1536	0.9405	0.5668	0.0128	0.0115	0.0104	-0.0003	0.0019	-0.0007	0.0116	Uij
<input checked="" type="checkbox"/>	C(2)	C _{1.0}	0.2477	0.0184	0.5075	0.0132	0.0124	0.0109	0.0006	0.0020	-0.0001	0.0122	Uij
<input checked="" type="checkbox"/>	C(3)	C _{1.0}	0.3006	0.2045	0.5404	0.0159	0.0144	0.0153	-0.0001	0.0037	-0.0025	0.0152	Uij
<input checked="" type="checkbox"/>	C(4)	C _{1.0}	0.2613	0.3150	0.6347	0.0181	0.0137	0.0170	-0.0020	0.0030	-0.0024	0.0163	Uij
<input checked="" type="checkbox"/>	C(5)	C _{1.0}	0.1687	0.2401	0.6954	0.0170	0.0143	0.0147	-0.0030	0.0029	-0.0008	0.0153	Uij
<input checked="" type="checkbox"/>	C(6)	C _{1.0}	0.1153	0.0550	0.6610	0.0146	0.0140	0.0117	-0.0016	0.0030	-0.0009	0.0134	Uij
<input checked="" type="checkbox"/>	C(7)	C _{1.0}	0.3673	0.7625	0.4367	0.0139	0.0157	0.0101	0.0005	0.0021	0.0013	0.0132	Uij
<input checked="" type="checkbox"/>	C(8)	C _{1.0}	0.3993	0.6598	0.3261	0.0207	0.0244	0.0128	-0.0035	0.0028	0.0047	0.0193	Uij
<input type="checkbox"/>	H(1)	C	0.0899	0.7461	0.5372	0.0134	0.0124	0.0104	-0.0004	0.0024	-0.0011	0.0121	Uij

1 of 21 sites selected

Cancel OK

Editing an Existing Structure

At any time during your work you can edit the crystal structure. You will need to specify which crystal to edit, by selecting the corresponding *pattern* in the Patterns List, before choosing: **Edit > Crystal Structure**. Note that many aspects of the structure, including lattice parameters and site occupancies, can be edited in real-time, via slider controls in the Parameters Pane.

Using the Crystal Editor

The Crystal Editor (a drop-down *sheet* on macOS, or a *dialog* on Windows), provides full details of the crystal to be edited:-

Spacegroup Symbol

A spacegroup symbol defines the arrangement of symmetry elements in the unit cell. This then allows the program to generate the complete contents of the unit cell, from your asymmetric unit. It also allows the program to determine which reflexions are related to each other, by symmetry. Such reflexions can be safely combined, to give a high-*multiplicity* reflexion.

CrystalDiffract gives you a choice of spacegroup formats:

- International Notation (e.g., $I a \bar{3} d$)
- Schoenflies Notation (e.g., O_h^{10})
- Spacegroup Number (e.g., 230)

Spacegroup symbols in *International* notation should be entered in plain-text form, with spaces between the symmetry operators, e.g.,

Examples of different spacegroup formats recognised by CrystalDiffract.

Spacegroup Symbol	Spacegroup Format		
	International	Schoenflies	Number
$P \bar{1}$	P -1	C1 (1)	1
$P m$	P m	Cs (1)	6
$P a \bar{3}$	P a -3	Th (6)	205
$P 2 2 2_1$	P 2 2 21	D2 (2)	17
$P n m a$	P n m a	D2h (16)	62
$R \bar{1} c$	R -3 c	D3d (6)	167
$C 2 / c$	C 2/c	C2h (6)	16
$I 4_1 / a$	I 41/a	C4h (6)	88
$P 4_1 2_1 2$	P 41 21 2	D4 (3)	92
$P 4_2 / m n m$	P 42/m n m	D4h (14)	136
$I a \bar{3} d$	I a -3 d	Oh (10)	230

CrystalDiffract verifies your spacegroup as you type. If the spacegroup is recognised, additional information, including the crystal system and number of symmetry generators, is displayed below. If you then click in a different text field, the spacegroup symbol is redrawn, using the conventional (rich text) formatting.

Origin Setting

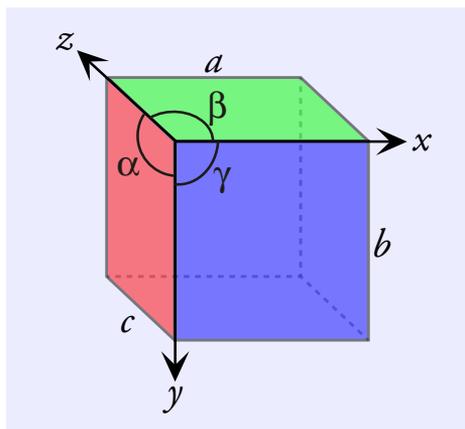
Some spacegroups require you to specify an origin setting, i.e., to choose between one of two possible locations relative to the symmetry elements. Typically there is a “conventional” origin setting (“origin choice 1”), and an unconventional setting (“origin choice 2”)—the latter usually being located on a centre of symmetry.

CIF Warning: Some CIF files are incorrectly formatted, omitting the symmetry generators, and instead relying on a spacegroup symbol. Such files can cause major problems, as there is then no way of specifying the origin setting the authors intended. You may need to use the Edit Crystal window to manually define the origin setting.

Lattice Parameters

You will need to enter six lattice parameters, which define the size and shape of your unit cell: the edge lengths, a , b and c , in Ångström units (the standard length scale for crystallography; $1 \text{ \AA} = 1 \times 10^{-10} \text{ m} = 0.1 \text{ nm}$) and the inter-axial angles, α ($y \angle z$), β ($x \angle z$) and γ ($x \angle y$), given in degrees.

A unit cell with coloured faces, illustrating its lattice parameters.



Tip: CrystalDiffract doesn't constrain your unit cell to the shape expected by the crystal system, e.g., you don't have to specify a cubic unit cell for a cubic crystal. This can be useful if you want to experiment with how changing the unit cell shape will affect peak splittings (e.g., as a result of a displacive phase transition).

Asymmetric Unit

CrystalDiffract doesn't, in general, expect you to list the entire contents of the unit cell. Instead, you should only provide the minimum set of crystal sites which, when combined with the spacegroup symmetry, results in the full unit cell contents. This minimum set is called the *asymmetric unit*.

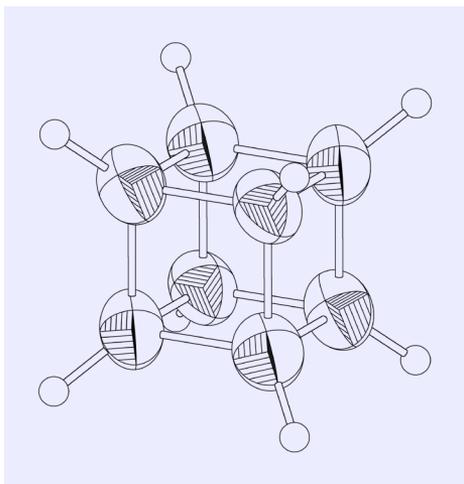
- Each site row has an **On** checkbox, which defines whether or not that site will be included in the intensity calculation. You could, for instance, decide to “turn off” certain sites, so as to determine their influence on the final diffraction pattern.
- You must provide a **Label** for each site, to uniquely distinguish it from the other sites. The label can be any length, and can contain any ASCII character, apart from white space (e.g., you can have “Ca (2)”, but not “Ca 2”).

- You can edit site occupancies by typing a formula into the **Occupancy** field. You can enter any number of element symbols and their corresponding occupancies. The total occupancy must not exceed 1.0. For example, you might enter something like: **Si 0.7 Al 0.3** or: **Ca 0.56 Mg 0.41 Al 0.03**.
- Each site requires three *fractional coordinates* **x, y, z** (i.e., fractions of the cell lengths *a, b* and *c*, respectively), which define the location of the site within the unit cell.

You can, optionally, define atomic displacement parameters for each site. These can be either anisotropic (**U11–U23**) or isotropic (**Uiso**). Use the **Type** popup to specify what type you have. (If you are using anisotropic displacement parameters, CrystalDiffract will calculate the equivalent isotropic parameter and display this in grey.)

Uiso to Biso: The isotropic *displacement* parameter, U_{iso} is related to the isotropic *temperature* factor, B_{iso} by the relation: $B_{iso} = 8\pi^2 U_{iso}$. Please see the *Glossary* for further information on *temperature-, displacement- and Debye-Waller factors*.

CrystalMaker image showing an organic molecule with thermal ellipsoids.



You can sort your data by clicking on a column header. Click again to reverse the sort order. You can also move columns, by clicking-and-dragging their column headers.

Verifying the Structure

If you want to check that your crystal structure is reasonable, the best way to do this is to visualize it in CrystalMaker. Simply select your simulated pattern and choose: **Pattern > Visualize Crystal Structure**. If CrystalMaker is installed, it will be launched, with your structure displayed.

Simulation Type

CrystalDiffract supports one of five types of diffraction simulation, which you can choose from the Diffraction menu:

- Constant-wavelength **X-Rays**
- Constant-wavelength **Neutrons**
- Constant-wavelength **Electrons**
- **Energy-Dispersive X-Rays**
- **Time-of-Flight Neutrons**

Each of these simulations requires additional parameters, which you can specify as follows.

Constant-Wavelength Experiments

To simulate a traditional, constant-wavelength X-ray diffraction experiment (e.g., a laboratory diffractometer):

- ◆ Choose: **Diffraction > X-Rays**.

To simulate a traditional constant-wavelength diffraction experiment (e.g., from a nuclear reactor source):

- ◆ Choose: **Diffraction > Neutrons**.

To simulate electron powder diffraction (i.e., powder rings observed in a transmission electron micrograph):

- ◆ Choose: **Diffraction > Electrons**.

To change the wavelength:

- 1 Choose: **Diffraction > Wavelength**. The *Wavelength* window appears.

The Wavelength window for X-ray powder diffraction. The dual-wavelength option is shown selected.

- 2 If using X-ray diffraction, choose between monochromatic (single-wavelength) or dual-wavelength radiation. (Most traditional X-ray diffractometers use radiation from the target's $K\alpha_1$ and $K\alpha_2$ lines, resulting in peak splitting at higher angles.)
- 3 Enter the wavelength(s) for your radiation, or click one of the **Presets** buttons (e.g., to use standard X-ray wavelengths for the corresponding target material; or, in the case of electron powder diffraction, to specify the simulated beam voltage).
- 4 Click the **OK** button to replot your diffraction profiles using the new wavelength(s).

Energy-Dispersive X-ray Diffraction

To simulate a white-radiation X-ray diffraction experiment (e.g., from a beamline at a synchrotron source):

- ◆ Choose: **Diffraction > Energy-Dispersive X-rays**.

Use the **Instrument** group in the Parameters List to specify the 2θ angle for the beam/detector geometry.

Time-of-Flight Neutron Diffraction

To simulate a neutron time-of-flight diffraction experiment (e.g., from a neutron spallation source such as ISIS in Oxford or SNS at Oak Ridge)

- ◆ Choose: **Diffraction > Time-of-Flight Neutrons**.

Use the **Instrument** group in the Parameters List to specify the 2θ angle for the beam/detector geometry (typically close to 180° for back-scattering) and the total neutron flight path, in metres. You can also specify the resolution function, $\Delta d/d$, for your chosen instrument (please see the section on “Instrumental Broadening” later in this chapter).

Choosing the Display Type

For a given simulation type, you can choose one of six different x -axis options from the **Diffraction** menu:

- 2θ
- d-spacing
- $1/d$
- $2\theta/d$ (“Q-space”)
- Energy (keV)
- Time-of-Flight (ms)

Note that the Energy and Time-of-Flight options are only available for Energy-Dispersive and Neutron Time-of-Flight diffraction simulations only.

When switching between the different display types, CrystalDiffract will recalculate your diffraction profile for any *simulated* patterns. Please remember, though, that observed data can only be plotted according to their original experimental types (i.e., the program does not recalculate x -values for observed patterns).

Setting a Reflexions Limit

By default, CrystalDiffract will limit the maximum number of reflexions used in its intensity calculations: this is to ensure that the vast majority of simulations proceed rapidly. You can override the default options for a particular window, using the **Diffraction > Reflexions Limit** command. You then have the option of specifying a minimum d-spacing (below which reflexions will not be generated), with the option of also specifying a maximum number of reflexions.

If you regularly need to override the default options (e.g., you work with massive structures such as proteins), you can change your global preferences—please see

the section on Preferences later in this chapter.

Peak-Shape Functions

In an ideal diffraction experiment, the shape of a diffraction peak would be determined solely by the sample, reflecting its mean particle size, particle shape and structural state (including strain).

In practise, for most samples the shape of diffraction peaks is mainly determined by the diffraction technique and geometry. For example, neutron diffraction experiments tend to result in peaks with a Gaussian shape, whilst synchrotron diffraction may result in a Pseudo-Voigt peak shape.

CrystalDiffract's **Diffraction > Peak Function** submenu lets you choose between four different shape functions:

Delta

The Delta function is simply a “spike” of near-zero width. This provides a very quick way of showing the positions of many peaks in a complex pattern.

Lorentzian

The **Lorentzian** function has a distinctive splayed appearance: peaks having very wide tails, like the mouth of a trumpet.

Gaussian

The **Gaussian** function is shaped like the profile of a church bell, with a more rounded appearance than the Lorentzian function, and with less extensive “tails”.

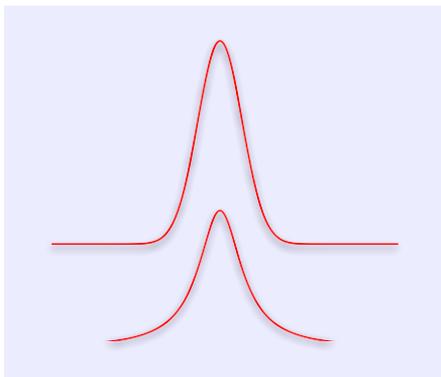
Pseudo-Voigt

The **Pseudo-Voigt** function is a mix between the Gaussian and Lorentzian functions. It is characterized by a mixing parameter, **Eta**, which determines the Lorentzian character of the final function:-

$$\text{Result} = \text{Eta} \times \text{Lorentzian} + (1 - \text{Eta}) \times \text{Gaussian}$$

You can edit the **Eta** parameter using the **Diffraction > Peak Function > Eta** command.

Gaussian (top) and Lorentzian (bottom) profiles for the same diffraction peak. Notice the lower peak maximum for the Lorentzian profile, with its intensity distribution spread over a wide range of x values.



Peak Widths

Powder diffraction peaks have a finite width, because of limited instrumental resolution, small particle size, and strain in the sample.

Instrumental Broadening

The limited resolution of a diffraction experiment may result in diffraction peaks that are substantially broadened. For most practical experiments, this “instrumental broadening” is the major contribution to the widths of observed diffraction peaks.

Constant-Wavelength and Energy-Dispersive Diffraction

For *constant-wavelength* or energy-dispersive experiments, CrystalDiffract lets you specify a fixed instrumental broadening using the **Diffraction > Instrumental Broadening** command. The broadening is specified in terms of the full width at half-maximum for a diffraction peak. (The units depend on the current choice of x -axis: two-theta, d -spacing, reciprocal- d , energy in keV.)

Time-of-Flight Neutron Diffraction

In neutron time-of-flight diffraction experiments, the peak widths vary systematically with increasing time-of-flight, t (and hence, d -spacing). This relationship is summarized by the *instrumental resolution*, which is expressed as the ratio:

$$\Delta d/d = \text{constant} = \Delta t/t$$

You can specify a default resolution in the Preferences window, and also edit the resolution for any experiment using the **Instrument** group in the Parameters Inspector.

Particle Size Broadening

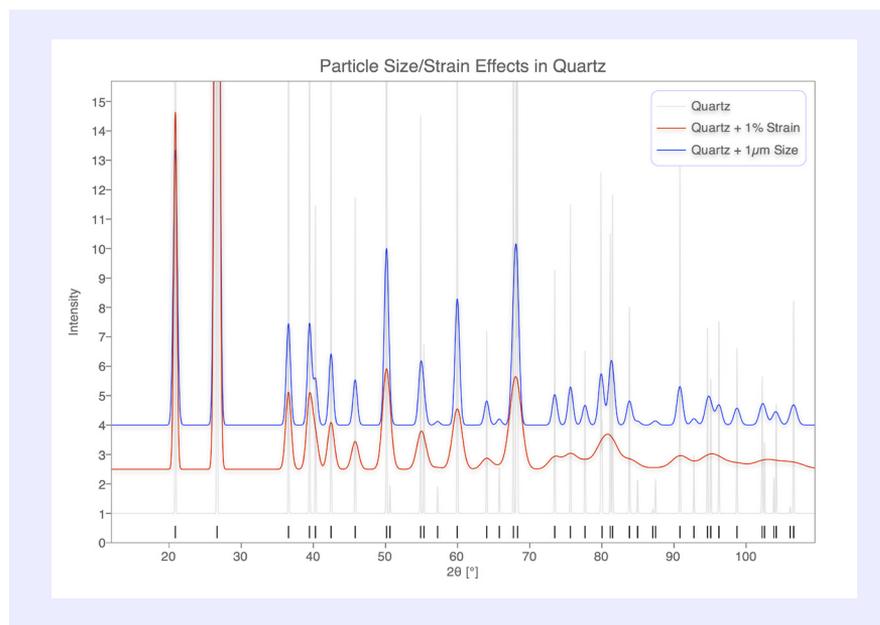
The width of a diffraction peak also depends on crystal size. This is a reciprocal relationship, so for large crystals there is very little peak broadening, but for very small crystals (fractions of a micron in diameter), diffraction peaks can become noticeably broadened.

In a powder sample, we normally refer to a mean particle size, and this can be simulated using the **Particle size** controls in the **Sample** group of the Parameters Inspector.

Strain Broadening

A strained crystal can be thought of as containing regions with slightly different unit cell dimensions. In fact, there is likely to be a continuous spread of unit cell dimensions throughout the sample, resulting in a diffraction pattern with a blurred appearance.

Particle size and strain broadening, simulated for quartz.



The amount of strain can be summarized by a “percent strain”. This is the standard deviation for the variation of cell parameters in the sample. In an ideal crystal there would be one unique cell parameter, whereas in a strained crystal there might be a normal distribution of cell parameters, characterized by a standard

deviation, ranging from zero to a few percent for a very-highly strained crystal.

To adjust the amount of strain in your simulated sample, use the **Iso. Strain** controls in the **Particle Size & Strain** group of the Parameters Inspector.

The Particle Size & Strain group in the Parameters List, showing controls for adjusting particle size and strain.

▼ PARTICLE SIZE & STRAIN

Particle size: 1.00 μm

Iso Strain: 0.000%

Temperature: Use displacement parameters

Preferred Orientation

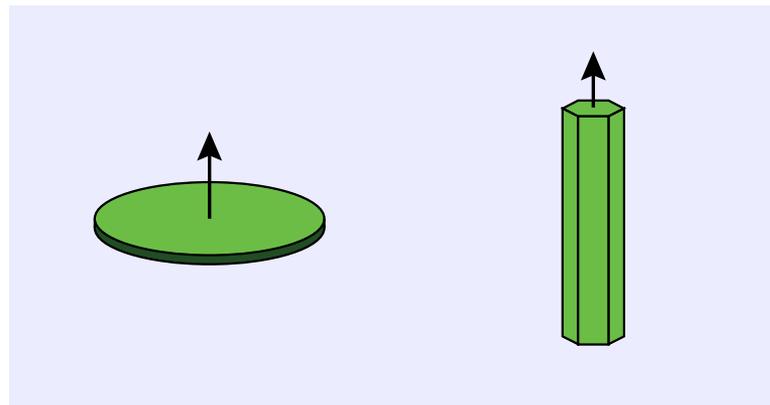
CrystalDiffract lets you simulate the effects of non-random crystallite orientations in your powder sample. Such “preferred orientation” can result in some peaks appearing much stronger than expected, whilst other peaks may be weaker—or entirely absent from the diffraction pattern.

CrystalDiffract can simulate preferred orientation from one of two common scenarios:

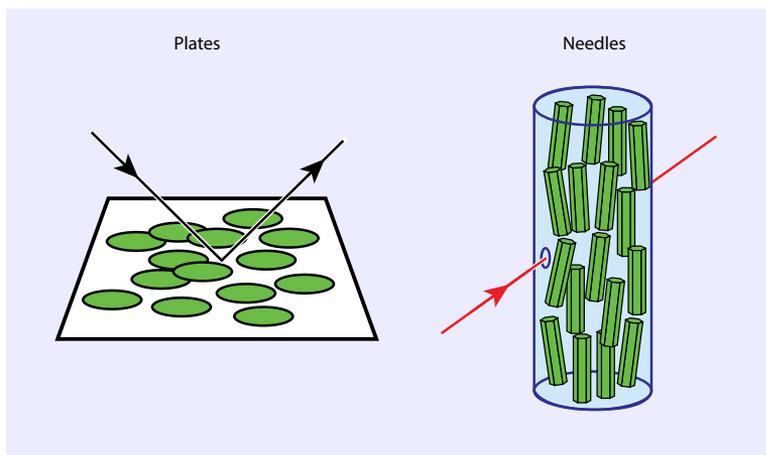
- Plate-like crystallites (e.g., clay minerals) - where the platelets tend to align parallel to the sample holder.
- Needle-like crystallites packed together in a wire or a capillary.

These morphologies and their relationships to the diffraction geometry are illustrated below:-

Crystallite shapes: Plate-like (left) and needle-like (right). The arrows represent the orientation vectors.



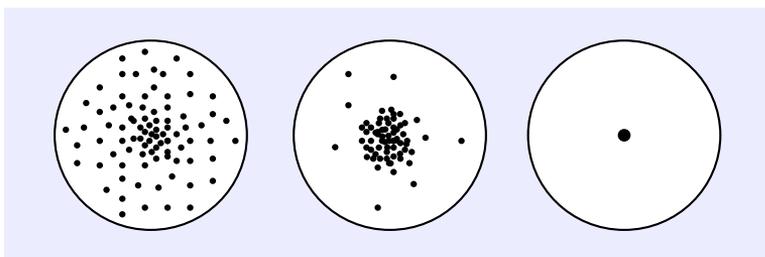
Preferred orientation sample geometries: plate-like crystallites aligned parallel to the sample holder (left); needle-like crystallites aligned inside a capillary tube (right). The arrows represent the incident radiation and its scattering.



The degree of alignment is represented by a fraction, with zero representing a completely-random sample, and 1 being a fully-ordered sample (e.g., all plates exactly parallel). In between the two, there is a statistical distribution of orientations.

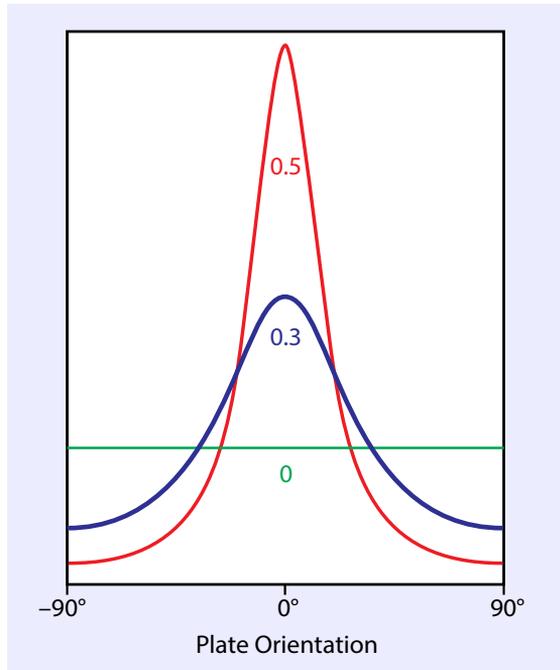
The orientation distribution can best be visualized by thinking of plate normals (or needle axes) plotted on a stereogram or “pole figure”. As the degree of alignment increases, the randomly-aligned poles begin to clump around the centre of the pole figure. The actual distribution of angles varies from -90° to $+90^\circ$, but its standard deviation decreases as the alignment increases.

Pole figures showing progressively-increasing degrees of alignment from random (left) to fully-aligned (right).



CrystalDiffract assumes a normal (Gaussian) distribution of orientations, as first proposed by March in 1932 (*Z. Kristallogr.* 81:285-297). Note that CrystalDiffract’s alignment parameter is equal to: $1 - R$, where R is the March coefficient which measures the degree of randomness.

Pole distributions for different degrees of alignment in a powdered sample of platy crystallites. The alignment parameter varies from 0 (random alignment) to 1 for a perfectly-aligned sample.



CrystalDiffract lets you specify the preferred orientation direction as a lattice vector $[UVW]$ or as a plane normal, $N(hkl)$, using the popup menu control to the right of the orientation text fields. The alignment parameter can be continuously varied, using the slider control:-

Adjusting the alignment parameter using the Preferred Orientation Parameter group.

The screenshot shows the 'PREFERRED ORIENTATION' control panel. It includes a checked checkbox, a 'Geometry' dropdown menu set to 'Plates', 'Orientation' fields for h (0), k (0), and l (1), and an 'Alignment' slider set to 0.25.

Interactive Parameter Control

You can control your simulated diffraction experiment more interactively by using the Parameters Inspector to gradually change one structural variable (e.g., unit cell angle) whilst the diffraction pattern is replotted in real time.

To show the Parameters Inspector:

- 1 If the Inspector is hidden, make it visible by clicking the toolbar's **Inspector** button () or by choosing: **View > Layout > Show Inspector**.
- 2 Click the **Parameters** tab to show the Parameters Inspector.

The Parameters Inspector contains a series of group buttons, each with its own disclosure triangle, and representing different aspect of the diffraction experiment. You can open (expand) a group by clicking its button, to reveal various slider-, text- and button controls.

Instrument

Use this group to change the peak function and adjust instrumental broadening in real time. CrystalDiffract provides a choice of Gaussian, Lorentzian and Pseudo-Voigt peak function, plus a Delta function.

Particle Size & Strain

Use this group to experiment with isotropic size and strain effects. As we have already seen in this chapter, these factors both affect the width of diffraction peaks, but in different ways.

Also included in this group is a checkbox for enabling or disabling atomic displacement parameters. This is a useful way of determining the relative importance of these factors in shaping the overall profile.

Preferred Orientation

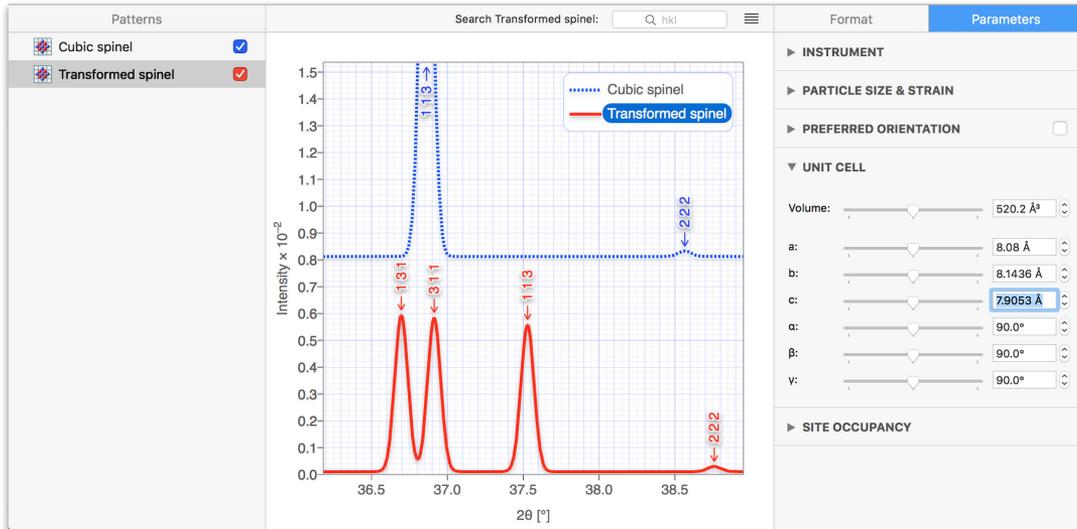
You can choose the particle geometry (plates or needles), the preferred orientation direction and the degree of preferred orientation.

Unit Cell

You can interactively edit the unit cell parameters (edge lengths, a , b , c ; angles α , β , γ) for a *selected* simulated (crystal) pattern, using the Unit Cell parameter group.

You can also apply *isotropic* expansion or compression, by using the **Volume** controls.

Please note that CrystalDiffract does not perform an energy minimization of the structure; one is simply “deforming” the unit cell, whilst keeping atoms in their existing sites, as defined by their fractional coordinates. Nevertheless, this is a useful range of settings when assessing the effect of a phase transition on the diffraction properties.



Editing cell parameters to investigate peak splittings resulting from a phase transition.

Site Occupancies

If a calculated pattern is selected, then you can interactively adjust the occupancies of its individual sites, using this group. Simply choose the site label from the group's popup, then add or remove occupants using the list controls below. You can use the slider controls to interactively change the proportions of the occupants, remembering that the total cannot exceed 1.

Editing the occupancy of the T1 site in a silicate structure.

▼ SITE OCCUPANCY

Site: T1 Include

Occupancy:

Si	<input type="range" value="0.7500"/>	0.7500
Al	<input type="range" value="0.2500"/>	0.2500

+ - ⚙️ Total: 1.0000

Lock occupancy total

To specify the site to be edited:

- ◆ Choose the site label from the **Site** popup.

To add or remove occupants:

- ◆ Use the + or – buttons to add or remove occupants. For example, if you wanted to investigate substituting a new element on a particular site, you might add it here and then adjust the occupancy values.

To equalize occupancy values:

- ◆ Click the Actions button () and choose: **Equalize Occupancies** from its popup menu. All site occupants will now have the same occupancy value (the current site total divided by the number of occupants).

To enter explicit occupancy values:

- 1 Click the Actions button () and choose: **Zero Occupancies** from its popup menu. All site occupancies will now be reset to zero, and the **Lock occupancy total** checkbox will be unset.
- 2 Click on each occupancy text field to select it, and type a new value.
- 3 If required, set the **Lock occupancy total** checkbox, so you can freely adjust slider values.

Please note that, as with the Unit Cell adjustments, CrystalDiffract does not optimize the structure following any of these adjustments. However, it does let you assess the chemical contribution to peak intensities.

Possible Applications

The Parameters Inspector is designed to be educational as well as functional. Here are some possible uses:

- Simulating **structural phase transitions** (e.g., cubic → tetragonal → orthorhombic) by changing cell parameters and watching how diffraction peaks split.
- Simulating the effect of **pressure and temperature** by changing the unit cell volume (isotropic expansion/compression is assumed).
- Visualizing the influence of one **site occupancy** on the final diffraction pattern, by dragging its site occupancy slider from 1 to 0.

- Changing the **composition of a mixture** by varying the proportions of individual phases, perhaps to match an observed diffraction pattern—and hence to determine its approximate composition.
- Understanding how mean **particle size** and/or **sample strain** affects the diffraction pattern.
- Fine-tuning a calculated diffraction pattern to **match an observed pattern**, e.g., by changing Peak Width, Eta value, Zero Error, etc.

Fast Parameter Mode

You can speed up parameter control by ensuring that **Fast Parameter Mode** is enabled in the **Profile** pane of the Preferences panel.

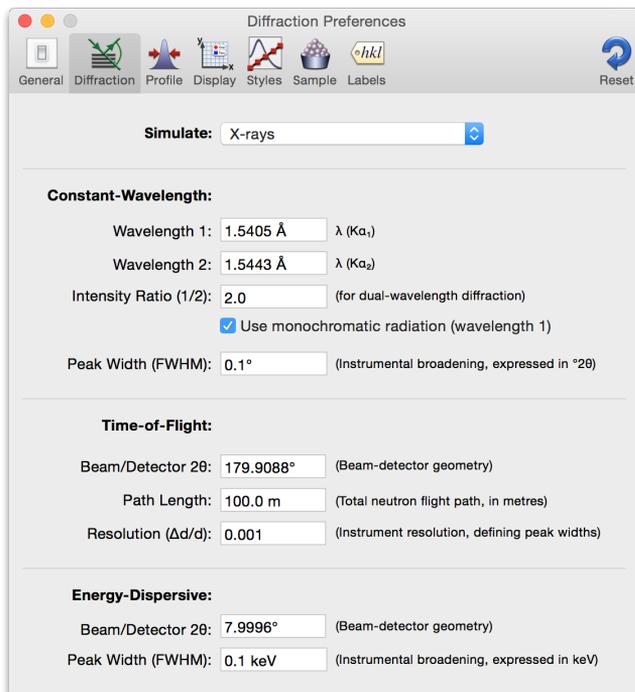
In “Fast Parameter Mode”, CrystalDiffract will temporarily use a smaller number of reflexions whilst you are adjusting simulation parameters (e.g., dragging a slider). Once you have finished your adjustments and the system is idle, CrystalDiffract will revert to the full number of reflexions and replot the simulated diffraction pattern.

Speeding up Simulations: In general, if you find that simulations are taking too long on a particular computer, you can opt to limit the maximum number of reflexions and/or impose a minimum d-spacing. These parameters are included in the Profile pane of the Preferences panel, as illustrated in the next section.

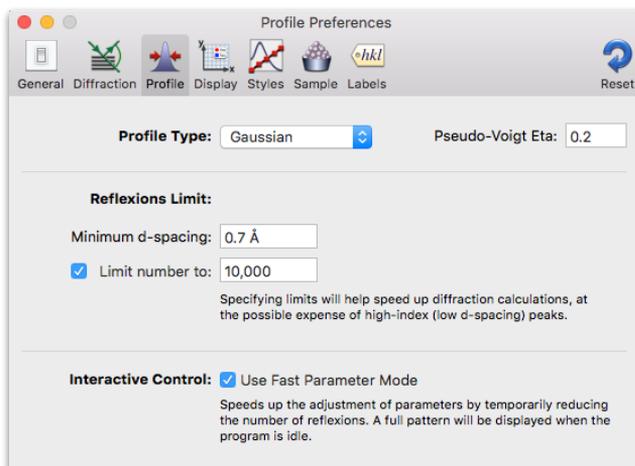
Simulation Preferences

You can set the default parameters for all new simulations using the Diffraction and Profile panes of the Preferences panel. Choose: **CrystalDiffract > Preferences** (Mac), or **Edit > Preferences** (Windows).

Diffraction preferences.



Profile preferences. Note that “Reflexions Limit” and “Interactive Control” sections for speeding up intensity calculations.



Editing Scattering Factors

CrystalDiffract’s Scattering Factors window (**Edit > Scattering Factors**) lets you browse the program’s active table of X-ray scattering factors and neutron scattering lengths. You can edit individual parameters and see the results directly in the graph at the top of the window. You can also add new elements to the table, import tables or data, or export data.

As with the diffraction graph, clicking in the Scattering Factors graph displays a vertical Cursor, which can be moved with the mouse, and allows you to measure displayed profiles.

You can save the graph by dragging-and-dropping it into another program, or you can right-click and choose: **Copy Graphics** or **Export Graphics** from the contextual menu.

You can visualize one or more X-ray scattering factors by selecting their rows in the table (hold down the **shift** or **command** keys to select multiple rows); curves for selected rows are then plotted in the graphics area (which can be resized by dragging the horizontal separator bar).

You can measure data in the graph by clicking to display a cursor, and then dragging this to the point of interest. To customize the graph, copy or export graphics, right-click in the graph and choose an appropriate command from the contextual menu.

To edit an existing element:

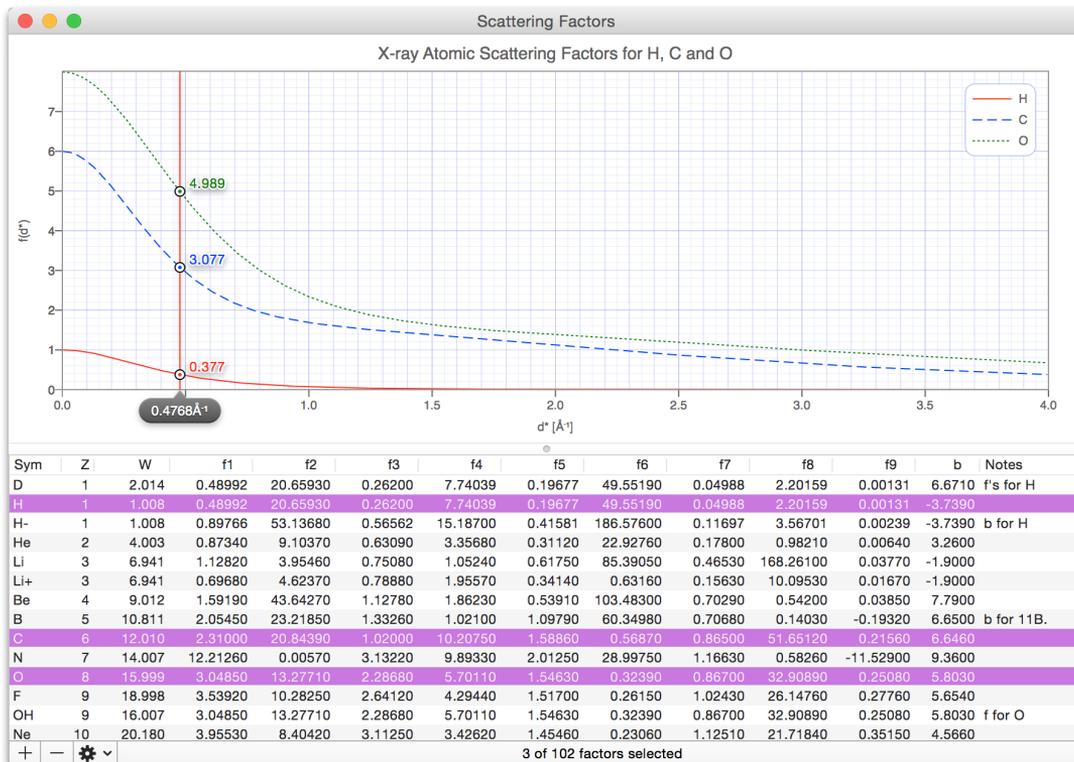
- 1 Select the element to be edited in the table.
- 2 Edit the appropriate parameters for x-ray scattering factors (f1-9) or the neutron scattering length.

To add a new element:

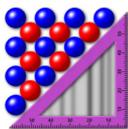
- ◆ Click the + button in the bottom left-hand corner of the window. Note that the “element symbol” you enter cannot contain spaces.

To delete one or more elements:

- 1 Select the row(s) you wish to delete (**shift**- or **command**-click to select multiple rows).
- 2 Click the - button in the bottom left-hand corner of the window.



The Scattering Factors window, showing three element rows selected.



Chapter 6

Working with Observed Data

CrystalDiffract can display the results of real diffraction experiments, letting you compare observed and simulated patterns in the same window. You can adjust scaling, data offsets, smoothing and subtract a background. This chapter explains how to create new observed patterns, load them from text files and scale x - and y -axis values (e.g., to match the simulated intensity scale). More advanced options, including data smoothing and background subtraction, are also covered.

Loading Data

If you have an observed diffraction pattern, you can visualize its data in CrystalDiffract. You can choose to load the data from a plain-text file, or you may type the values directly into the program, as a new observed pattern.

Creating a New Pattern

If you need to, you can create an “observed” pattern directly in CrystalDiffract, without the need for importing data. CrystalDiffract will build a dummy “observed pattern” and present you with an editing window, so you can enter your own values.

Editing a new observed pattern.

Num	x	y
1	1.23	0.0000
2	10.0000	1.0000
3	20.0000	2.0000

+ - 1 of 3 points selected

? Cancel OK

To create a new observed pattern

Do one of the following:

- ◆ Click the toolbar’s **Add** button and choose: **New Observed Pattern** from its popup menu.
- ◆ Choose: **File > Add New Pattern > Observed Data**.

In each case, an editing window appears, letting you enter your x and y values.

Loading a Saved Pattern

CrystalDiffract can load observed diffraction patterns from *plain-text* ASCII data files in either of two formats: XY or Rigaku ASC.

XY Format

An “XY” text file is simply a plain-text file where each line has a pair of numbers, representing one data point: an x -value, and a y -value. The first line of the file can, optionally, be a title line, as the example below shows.

An example XY text file.

```

Analcime: scanned x-ray film.
11.2866 146.13
11.3077 146.22
11.3288 144.79
11.3498 146.19
11.3709 145.4

```

CrystalDiffract expects these files to have one of the following file extensions: “.txt”, “.dat”, “.xy” or “.xye”.

Rigaku/Scintag ASC File

The Rigaku “ASC” file (extension “.asc”) has a more complex format, as it includes diffractometer parameters, sample name, and other proprietary parameters (which are ignored by CrystalDiffract).

An example Rigaku ASC text file.

```

*CLASS = ASCII CLASS
*SAMPLE = Si TEST SAMPLE
*COMMENT = Si Std
...

*COUNT = 3501
14, 12, 7, 17
11, 14, 9, 9
11, 17, 11, 11
8, 11, 17, 17
19, 6, 15, 19
...

```

To open a saved diffraction pattern in a new window

Do one of the following:

- ◆ Drag-and-drop the file onto CrystalDiffract's application icon.
- ◆ Choose: **File > Open**, and use the file dialog to specify your file.

To open a saved diffraction pattern in the same window

Do one of the following:

- ◆ Drag-and-drop the file into the window's Graphics Pane. The new pattern will be displayed, and an entry added to the Patterns List.
- ◆ Drag-and-drop the file into the window's Patterns List. The new pattern will be listed, but will not be plotted until you click its checkbox.
- ◆ Choose: **File > Open in Same Window**, and use the file dialog.
- ◆ Click the toolbar's **Add** button and choose: **Add Pattern From File** from its popup menu.

Use Plain Text: Data files must be simple, plain-text, ASCII files. If you edit the file in a word processor, you should ensure that it is saved in a text-only format (and is not saved as a rich-text or proprietary data format such as Microsoft Word).

Editing and Scaling Data

CrystalDiffract lets you manually edit the data points for an observed pattern, using its built-in data editor. You can modify values, add new data points, or delete existing ones.

CrystalDiffract also lets you apply scaling factors to the entire pattern, e.g., to convert the x -axis units; or to scale the y -axis values so that the intensity range matches that of your simulations.

Editing an Observed Pattern

CrystalDiffract's built-in data editor lets you manually edit data values for an observed pattern. (This is the same editor that appears when you create a new pattern in the program.)

To edit an existing observed pattern:

- 1 Select the diffraction pattern to be edited, using the Patterns List. (Note that only one pattern may be edited at a time.)
- 2 Choose: **Edit > Observed Data**. The data editor sheet or dialog appears.
- 3 Use the + and - buttons to add or remove data points. Click on x or y values

to edit them. (Note that, for editing purposes, you can rearrange your list by dragging-and-dropping rows into new locations in your table.)

- 4 Click the window's **OK** button to replot your diffraction pattern.

Scaling X-Values

Should you require it, you can automatically scale *all* the x -values for your observed pattern. You might need to convert units, or to calibrate “raw” data values.

Example 1: If your observed time-of-flight neutron data were measured in μs (microseconds, $1 \mu\text{s} = 10^{-6} \text{ s}$), and you wanted to plot them in CrystalDiffract, which uses a ms (millisecond, $1 \text{ ms} = 10^{-3} \text{ s}$) scale, then you would need to apply a factor of 0.001 to convert μs to ms .

Example 2: If your observed diffractometer data were measured in step increments, and you wanted to convert these to $^{\circ}2\theta$, you might compute a scale factor based on the known distance between two peaks, and your measured distance on screen. So, if your two peaks were $28.5^{\circ} 2\theta$ apart, and this corresponds to 1200 x -axis units, you would need to apply a factor of $28.5 / 1200 = 0.02375$ to convert your x -axis units to $^{\circ}2\theta$. (You would also probably need to specify an x -offset, which is discussed later in this chapter.)

To rescale all x -values:

- 1 Select the pattern(s) to be scaled, using the Patterns List.
- 2 Choose: **Pattern > Scale X-Values**. A sheet or dialog appears.
- 3 Enter the scale factor you wish to apply to your x -values, and click the **OK** button to replot.

Scaling Intensity Values

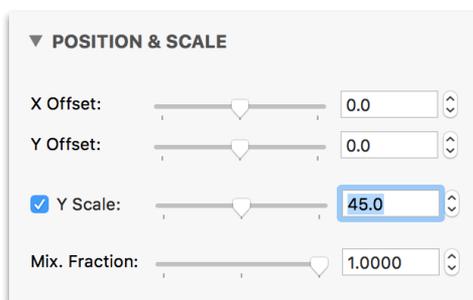
Chances are, your observed intensity scale probably won't match the intensity scale used for simulations. There is no reason why it should: intensities are always recorded in arbitrary units, and depend on the intensity of the radiation used, the sample size, detector efficiency, and many other factors. In contrast, CrystalDiffract's simulated intensities all use a consistent intensity scale—designed to allow you to directly compare different simulated patterns.

In order to compare your *observed* patterns with the simulated patterns, you will probably need to apply a scale factor to the observed data. This is easy to do, and can be adjusted in real time using the Format Inspector.

To apply a scaling factor to the y-values:

- 1 Select the diffraction pattern(s) whose data you wish to edit.
- 2 Open the **Position & Scale** group in the Inspector's **Format** tab.
- 3 Set the **Y Scale** checkbox to enable its editing fields.
- 4 Adjust the **Y Scale** using the slider control or its edit text field.

Adjusting the Y Scale for an observed pattern.



Note that, unlike with the x -axis scaling (described earlier), applying a y -scale factor does not directly change your observed data values. Instead, a scale factor is applied during plotting. This makes it easier to “tweak” the scale factor interactively.

One for All: CrystalDiffract will apply your scale factor to every one of your x values in the selected pattern(s).

Adjusting Data Offsets

You can also use the **Position & Scale** group to apply a zero correction to your x -axis data (perhaps to correct for a systematic error in your diffractometer, as determined from the use of an internal standard), or to your y -axis data (to bring your “baseline” down to a reasonable value for display).

To apply an x- or y-offset to the pattern:

- 1 Select the diffraction pattern(s) whose data you wish to edit.
- 2 Open the **Position & Scale** group in the Format List.
- 3 Adjust the **X Offset** or **Y Offset** controls.

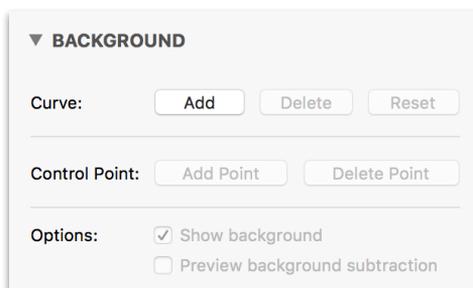
Subtracting the Background

CrystalDiffract lets you define a background function for an observed pattern. You can then subtract this from your data points to reveal the bare diffraction pattern.

To add a background function:

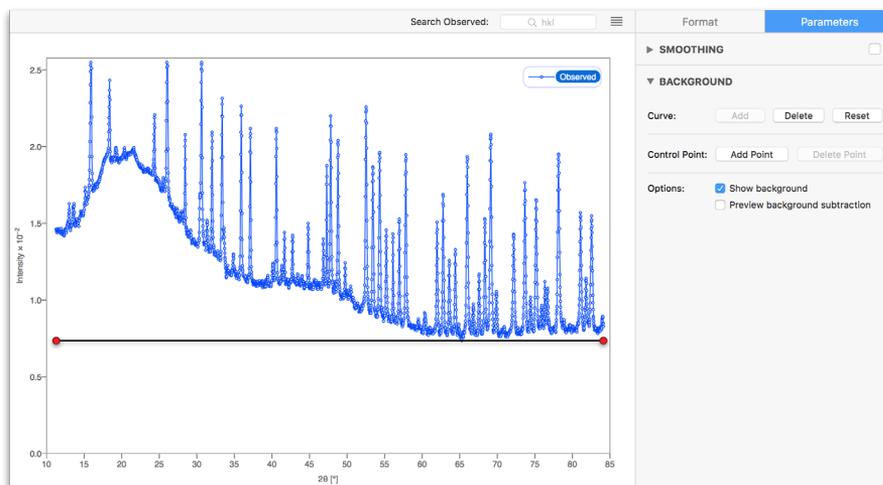
- 1 Select the *observed* diffraction pattern whose background you wish to remove. (You can only select one pattern at a time for this operation.)
- 2 Open the **Background** group in the Parameters Inspector.

The Background group in the Parameters Inspector.



- 3 Click the (*Curve:*) **Add** button. A dark line appears in the Graphics Pane, with two red control points, for editing the shape of the background function.

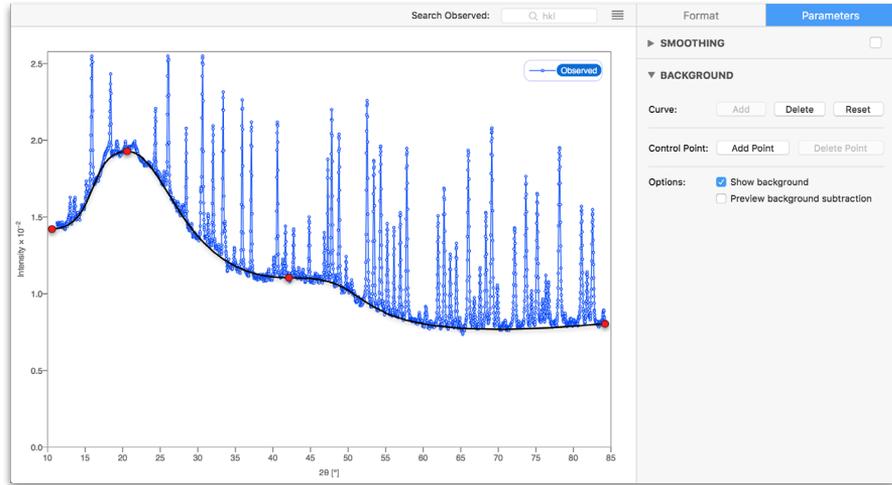
Starting to define a background function (the black line with its two red control points).



To edit the background function:

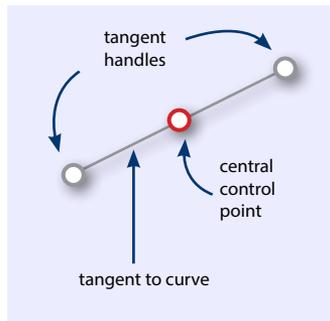
- 1 If the background function isn't visible, ensure that your observed pattern is selected and the **Show background** checkbox (**Background** group) is set.

Observed pattern with a background function defined.



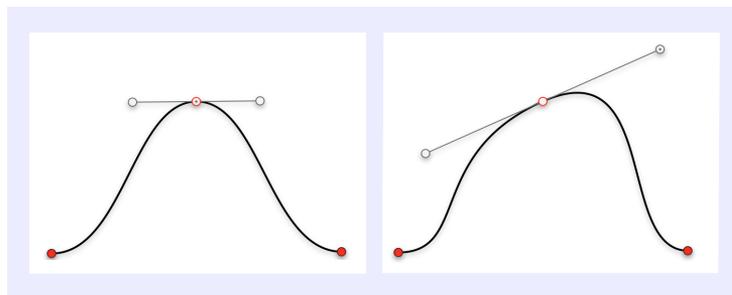
- 2 In the Graphics Pane, click-and-drag a red control point to bend your background function.

Anatomy of a selected background control point.



- 3 Drag a tangent handle (white circle) vertically to change the slope.
- 4 Stretch or contract the tangent handle to change the radius-of-curvature of the background function (at the control point).

Using control handles to adjust the curvature of the background function.



- 5 To add more control points, click the **Add Point** button in the Background group.
- 6 To delete the currently-selected (active) control point, click the **Remove Point**

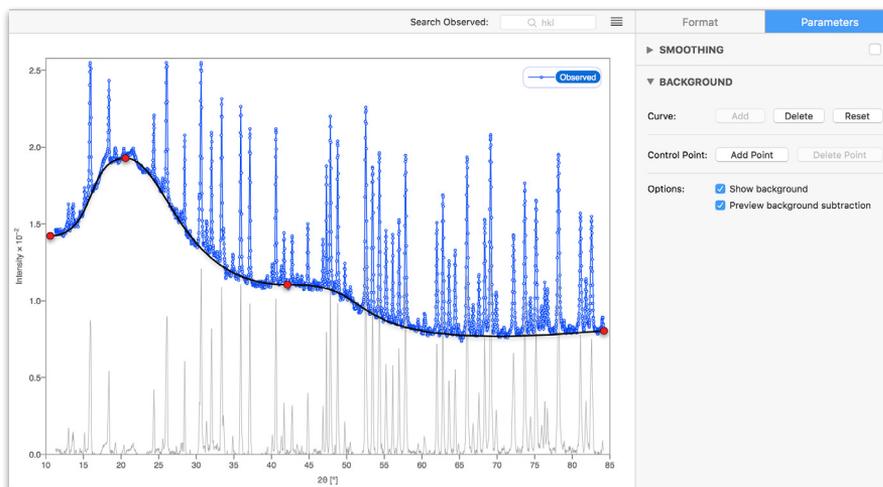
button in the Background group.

Keep it Simple: Try not to add too many control points, as this will slow down your editing and display. Instead, try to make the most of your existing control points, adjusting their positions, curvature, and slope. For most patterns, you will require no more than 4–5 control points.

To preview the background subtraction:

- ◆ Set the **Preview background subtraction** checkbox in the **Background** group. A preview of the background subtraction will be shown, as a separate curve. You can then fine-tune the shape of the background function.

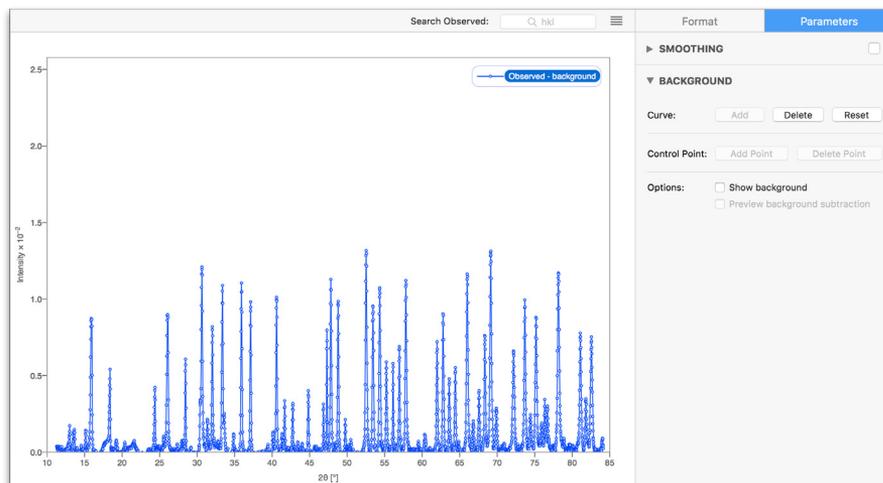
Previewing the background subtraction (grey curve at bottom)



To subtract the background function from your observed data:

- ◆ Unset the **Show background** checkbox in the Background group. The background function will vanish, and your observed pattern will be shown minus the background.

Observed pattern, after subtracting the background function.



To change the colour or line style of the background function:

- ◆ Use the controls in the **Background Overlay** group in the Inspector's **Format** tab.

To remove a background function:

- 1 Ensure that your pattern is selected in the Patterns List.
- 2 Click the (*Curve:*) **Remove** button in the Background group.

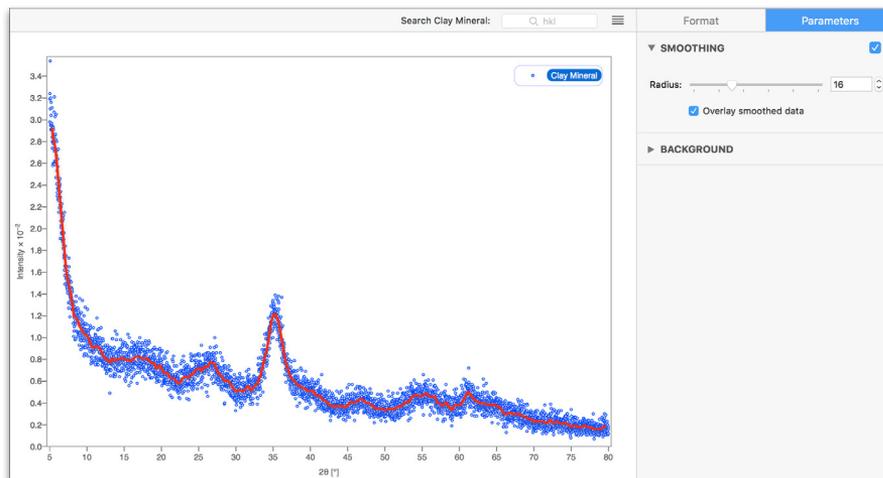
Smoothing Data

CrystalDiffract lets you interactively smooth the *intensity values* in your observed pattern. This should make it easier to locate peaks in otherwise noisy data.

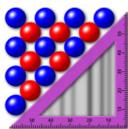
To smooth an observed pattern:

- 1 Select the observed pattern(s) you wish to smooth.
- 2 Open the **Smoothing** group in the Parameters List.
- 3 Set the checkbox. Your observed pattern is replotted, using a bilinear smoothing algorithm.
- 4 Adjust the **Radius** slider and/or text box to control the amount of smoothing.
- 5 To compare smoothed and unsmoothed data (as illustrated below), set the **Overlay smoothed data** checkbox.

Raw data (blue points) with smoothing preview (red curve).



You can adjust the colour and line style of the smoothing overlay using the **Smoothing Overlay** group in the Format Inspector.



Chapter 7

Simulating Mixtures

This chapter outlines how you can create a mixture from separate patterns, edit the proportions of mixture components, and control the visibility of individual mixture components, with or without the simulated mixture.

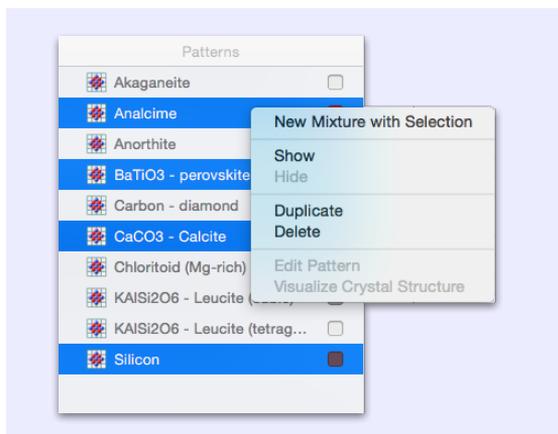
Creating a Mixture

You can simulate a multi-phase mixture by specifying two or more patterns to be the mixture components. CrystalDiffract will create a new, “Mixture pattern”, represented by a “container” group in the Patterns List, into which you can drag other Patterns. The Mixture’s diffraction profile is the sum of the components profiles, scaled by their mixture fractions. You can continue to edit the individual patterns, and the mixture will be updated.

To create a new mixture:

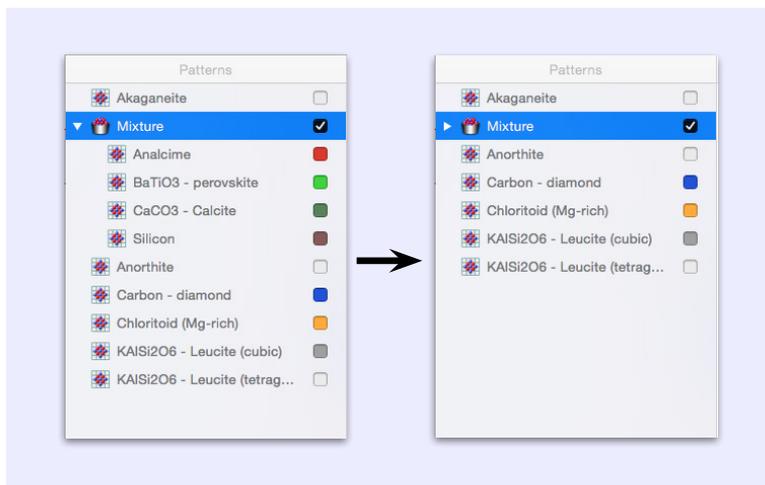
- 1 In the Patterns List, select the patterns you wish to combine (e.g., on the Mac, you would hold down the **command** key and click on each pattern to select it).
- 2 Right-click in the Patterns List to display a contextual menu and choose: **New Mixture with Selection**.

Creating a mixture from a selection in the Patterns List.



A new Mixture pattern is created, with the selected patterns as components. The mixture pattern is represented as a “container” or “folder” object in the Patterns List. You can use its disclosure triangle to show or hide the constituent patterns.

The Mixture group in the Patterns List, showing “open” (left) and “closed” (right) forms.

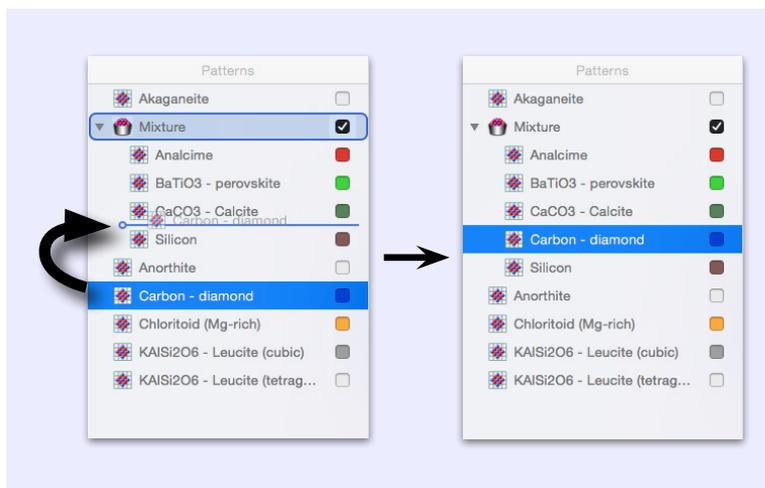


Please note that, when plotted, the diffraction patterns for the mixture components are scaled according to their respective mixture proportions.

To add components to an existing mixture:

- ◆ Drag-and-drop patterns into the Mixture group.

Adding a new component to an existing mixture by dragging-and-dropping its pattern into the mixture group.



To remove components from an existing mixture:

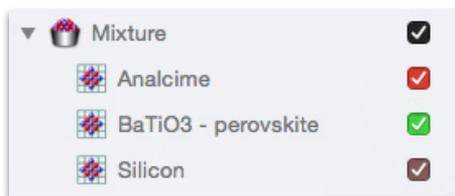
- ◆ Drag the patterns out of the mixture group.

Multiple Mixtures & Patterns: Unlike earlier versions of the program, CrystalDiffract 6 lets you visualize multiple mixtures in the same window. You can also visualize individual patterns and mixture patterns: you are no longer restricted to *either* all-mixture *or* all-separate modes.

Displaying Mixture Components

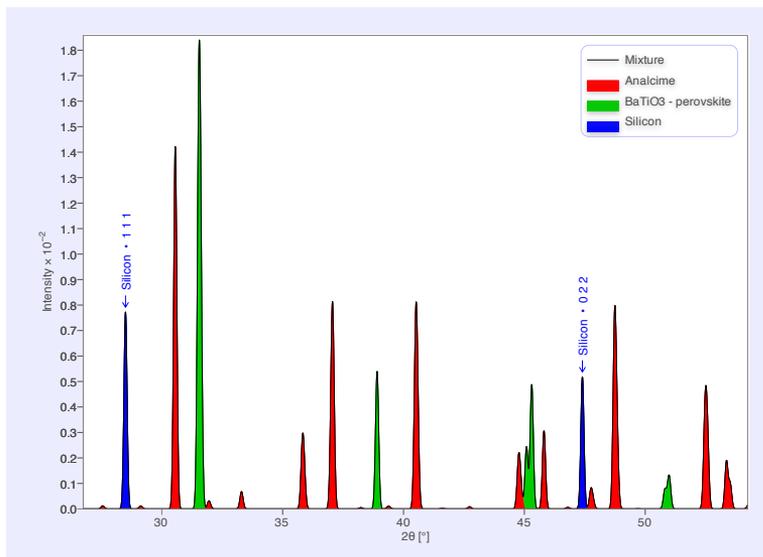
When you simulate a mixture in CrystalDiffract you are, in effect, creating a new pattern. This is represented as a disclosure (“folder”) group in the Patterns List, with the individual mixture components inside. To display the individual components, simply enable their visibility checkboxes.

Patterns List showing a visible mixture and its three component patterns.



To make your individual patterns stand out from the simulated mixture pattern, you might want to change their plot styles (using the **Plot** group in the Parameters List), specifying a solid fill, as the example below shows:

Simulated diffraction patterns for a multi-phase mixture (dark line) and its individual components (red, green and blue).



Editing Mixture Proportions

CrystalDiffract lets you edit the composition of your mixture by changing the relative proportions of the phases involved.

Mixture Composition

You specify the relative *volume fractions* of the phases in a mixture. This makes it easier to relate your simulated mixture to a real-life experiment, where you may mix known volumes of different phases.

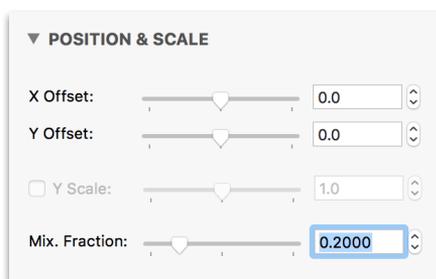
You can edit the composition in one of two ways:

- Editing an individual pattern's mixture fraction (and letting the program recalculate the remaining phase proportions); or
- Editing the Mixture group as a whole (which gives more precise control).

To edit an individual component:

- 1 Using the Patterns List, select the pattern whose phase proportion you want to change.
- 2 Ensure that the pattern's visibility checkbox is set (i.e., it is plotted on screen) as, for safety's sake, CrystalDiffract does not let you edit settings for hidden patterns.
- 3 Open the **Position & Scale** group in the Format Inspector. (If the Inspector is hidden, choose: **View > Layout > Show Inspector**.)
- 4 Adjust the **Mix. Fraction** slider control to set the relative phase proportion for this component.

Editing the mixture fraction for an individual component.

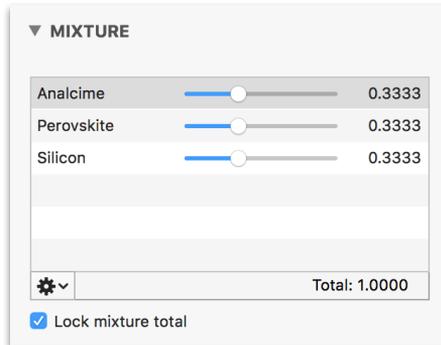


CrystalDiffract will automatically adjust the proportions of any other phases, to ensure that the overall sum of mixture components remains constant. If you need more precise control, you will need to edit the Mixture group directly, as described below.

To edit the entire mixture:

- 1 Select the mixture you wish to edit (e.g., by clicking its name in the Patterns List).
- 2 Open the **Mixture** group in the Parameters Inspector.
- 3 Adjust the slider controls, or use the text fields, to set the phase proportions for individual mixture components.

Adjust the phase proportion for one selected pattern (which is part of a mixture).

**To enter explicit phase proportions (e.g., $A_{0.7} B_{0.2} C_{0.1}$):**

- 1 Click the Actions popup button () in the Mixture Group and choose: **Zero Phase Proportions** from its menu. All phase proportions will now be reset to zero, and the **Lock mixture total** checkbox will be unset.
- 2 Click on each component's text field to select it, and type a new value.
- 3 If required, set the **Lock mixture total** checkbox, so you can freely adjust slider values.

To reset phase proportions:

- ◆ Click the Actions popup button () and choose: **Equalize Phase Proportions** from its menu. The mixture composition is reset, so that all components have the same phase proportions.

Deleting a Mixture

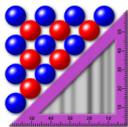
If you want to delete a mixture (rather than just hiding it), you need to choose whether you also want the mixture's components to be deleted.

To delete a mixture and its components:

- 1 Select the mixture in the Patterns List.
- 2 Right-click on the mixture and choose **Delete** from the contextual menu.

To delete a mixture but retain its components:

- 1** In the Patterns List, ensure that the relevant mixture group is open (click its disclosure triangle to reveal the mixture components).
- 2** Drag all of the components outside the mixture group. This is best accomplished by selecting all components, and then dragging the group outside the mixture.
- 3** Select the (now empty) mixture pattern, and choose **Delete** from the Patterns List contextual menu.



Chapter 8

Manipulation

CrystalDiffract makes it easy to scroll or zoom diffraction patterns with multi-touch or mouse control—plus high-resolution magnification via the Loupe.

This chapter explores the ways in which you can scroll and zoom the plot, automatically stack multiple graphs, magnify part of the plot using the Loupe, toggle pattern visibility and rearrange plot order using the Patterns List, plus control plot-, sample- and diffraction parameters using the Format and Parameters Lists in the Inspector.

Scrolling and Zooming

CrystalDiffract lets you set an explicit plot range by specifying minimum and maximum x -values. A more-interactive way of doing this is to use the mouse, clicking-and-dragging through the plot range. Even more intuitive is multi-touch: if your computer has a suitable trackpad (e.g., most Macs) you can use standard gestures to scroll or zoom.

Setting an Explicit Plot Range

You can use the Plot Range window (**View > Plot Range**) to specify explicit minimum and maximum values for the x -axis:

Using the Plot Range command.

X-axis range from: to:

Scrolling with the Mouse

There are three ways to scroll with the mouse:

- Click and drag the plot area left or right.
- Use the Toolbar **Scroll** buttons to scroll left or right, one screen at a time.

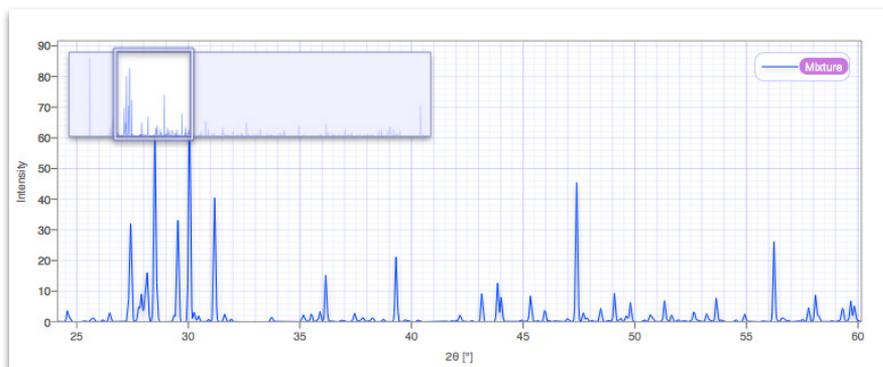
(Customize your toolbar to show these buttons.)

- Use the Scroller.

Scrolling with the Scroller

Most graphics programs use scrollbars to let one scroll an over-size image inside a fixed frame. CrystalDiffract has something better: a “Scroller” panel which shows a preview of the entire diffraction pattern, extending beyond the limits of the current plot range (which is indicated in the “thumb” rectangle).

The Scroller in action.



The Scroller is normally hidden, for clarity, but it appears (in the top left-hand corner of the Graphics pane) whenever the plot range is being changed: when scrolling or zooming.

To show the Scroller

Do one of the following:

- ◆ Using the mouse, click-and-drag in the Graphics pane.
- ◆ Use a multi-touch gesture (see next section) to scroll or zoom.

To use the Scroller

Do one of the following:

- ◆ Click-and-drag the Scroller thumb to change the plot range.
- ◆ Click-and-drag the left- or right edges of the thumb to adjust the width of the plot range; or
- ◆ Click and hold down mouse on either side of the thumb to continuously scroll the pattern (as with a traditional scrollbar).

To hide the Scroller:

- ◆ Move the mouse outside the Scroller rectangle. After a few seconds the Scroller will vanish.

To disable the Scroller:

- 1** Choose: **CrystalDiffract > Preferences** (Mac) or **Edit > Preferences** (Windows). The Preferences panel appears.
- 2** Choose the **General** tab.
- 3** Locate the **Overlays** group and uncheck the **Scroller** checkbox.

Manipulating the Pattern with Multi-Touch

CrystalDiffract supports scrolling and zooming, using multi-touch: this is where a trackpad detects movements of two fingers and interprets these as one of two types: a “pinch” gesture or a “slide” gesture.

Multi-Touch on Modern Computers

Multi-touch is available on all portable Macs, and desktop computers with a Magic Trackpad. Windows computers have not traditionally had such elegant hardware integration—but trackpads are now widely available on laptop PCs and can be added to desktop PCs.

Pinch-to-zoom

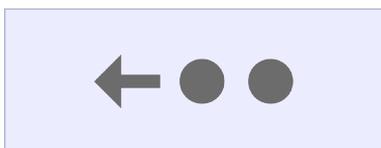
Use a standard “pinch” or “expand” gesture to contract or expand the horizontal range. CrystalDiffract detects your initial touch position on the trackpad, so you can change the centre of the zoom by repositioning your fingers on the trackpad.

“Expand” trackpad gesture.

**Slide-to-scroll**

If your system is configured to accept two-fingered scrolling, you can scroll the diffraction pattern left or right by sliding two fingers horizontally across the trackpad.

Slide-to-scroll trackpad gesture.



Slide-to-scale

If your system is configured to accept two-fingered scrolling, you can scale the diffraction pattern left or right by sliding two fingers vertically, up or down the trackpad.

Slide-to-scale trackpad gesture.

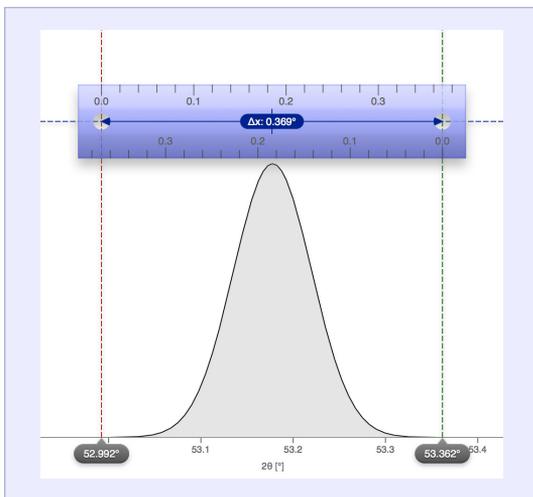


Multi-Touch Gestures: You can use the pinch-to-zoom and slide-to-scale gestures to control the Loupe (as described later in this chapter).

Zooming with the Ruler

You can use the ruler's vertical and horizontal cursors to define a smaller plot range—much like a traditional “marquee” tool:

Defining zoom limits with the ruler



To zoom with the ruler:

- 1 Make the ruler visible (e.g., click the toolbar's **Ruler** button).
- 2 Click and drag the ruler's vertical cursors to define your intended x -axis range.
- 3 Click and drag the ruler's horizontal cursor to define your intended y -axis maximum.
- 4 Right-click in the ruler to display a contextual menu and choose: **Zoom to Cursors**.

Automatic Scaling

CrystalDiffract provides a pair of Autoscale commands which can be accessed from the toolbar:

- Autoscale Y (\updownarrow): rescales the y -axis to fit any peaks in the current plot range.
- Autoscale X and Y (\leftrightarrow): resets the x -range so that all diffraction patterns are clearly displayed.

Automatic Scrolling Limits

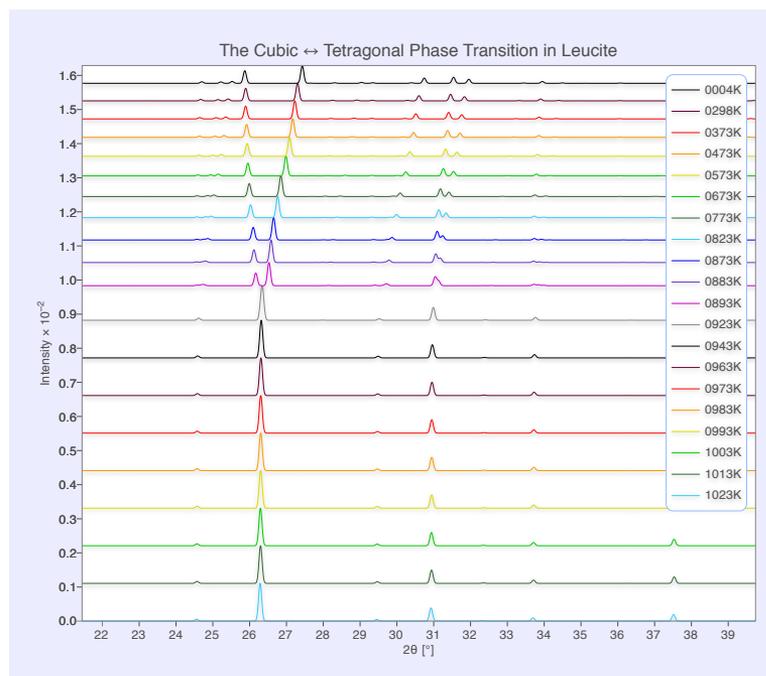
Whilst you have the flexibility to specify an arbitrary plot range (using the **Set Range** command), CrystalDiffract pre-calculates the maximum scroll range, based on the current data range. For example, an observed pattern with datapoints in the x -range 20 – $80^\circ 2\theta$ can only be scrolled between $x = 20^\circ$ and $x = 80^\circ$. However, if additional patterns with wider x -ranges were added to the plot, the scroll range would expand.

When working with simulated data, CrystalDiffract calculates a scroll range based on the first and last diffraction peaks.

Stacking Patterns

If you are working in **Graph** mode and want to compare multiple patterns, you can automatically stack the patterns.

Auto-stacked
diffraction patterns.



To stack graphs:

- ◆ Choose: **View > Stacked**. The graphs will be automatically positioned on the screen to avoid any overlaps.

To manually adjust the stacking amount:

- 1 Select a pattern, by clicking on its title in the Patterns List.
- 2 Open the **Position & Scale** group in the Format Inspector.
- 3 Adjust the **Y Offset** controls to move the graph vertically.

To change the stacking order:

Do one of the following:

- ◆ Click and drag patterns in the Patterns List to change the stacking order.
- ◆ Use a **Sort** command from the Patterns List Actions menu, e.g., **Sort by Title**.

Tip: To reverse the direction of the sort, press **shift** as you select a **Sort** menu command.

To undo (collapse) any stacking:

- ◆ Choose: **View > Collapsed**.

Logarithmic Intensity Display

By default, CrystalDiffract plots intensities on a linear scale. This works well for most patterns, but if you need to easily see very-weak and very-strong reflexions on the same screen, you might want to switch to a *logarithmic* scale.

To use a logarithmic intensity scale:

Do one of the following:

- ◆ Choose: **View > Logarithmic Y-Axis**.
- ◆ Click the **Use Log Scale** button () in the toolbar.

To revert to a linear intensity scale:

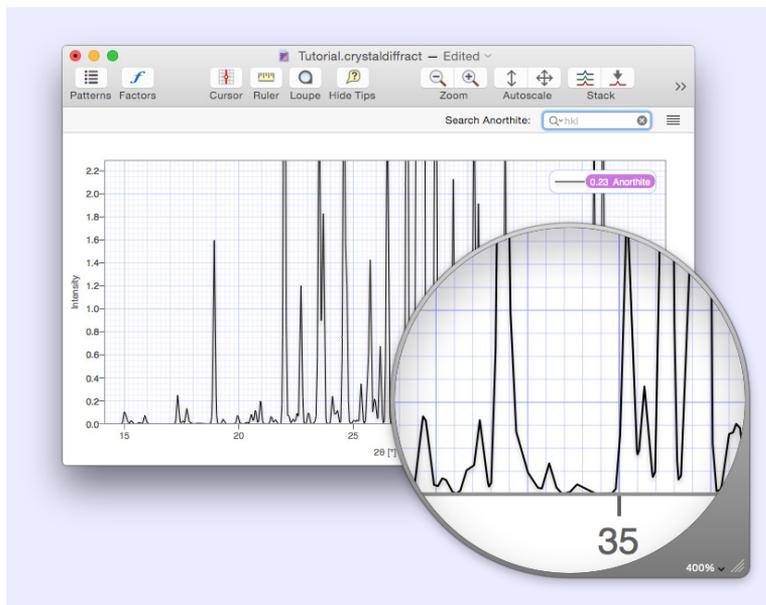
Do one of the following:

- ◆ Choose: **View > Linear Y-Axis**.
- ◆ Click the **Use Linear Scale** button () in the toolbar.

Using the Loupe

The Loupe tool lets you examine part of your plot in detail, without having to change the overall scale. This makes it easier to compare multiple patterns across their entire x-range.

The Loupe in action.



To show the Loupe:

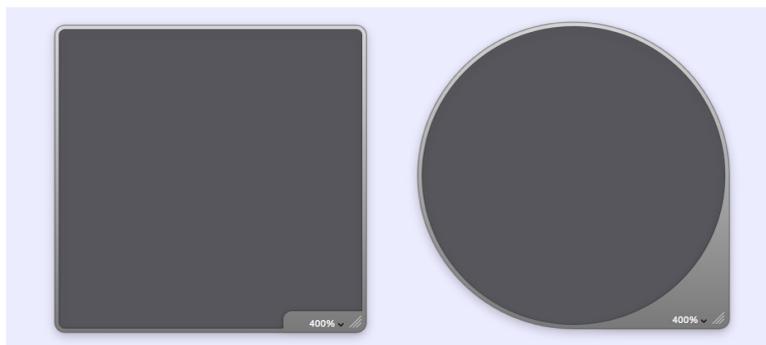
Do one of the following:

- ◆ Click the **Loupe** button (🔍) in the Toolbar.
- ◆ Choose: **View > Overlays > Show Loupe**.

To switch to a Square Loupe:

- ◆ Choose: **Square** from the Loupe popup menu or its contextual menu.

The square and circular loupes.



To change the size of the loupe:

Do one of the following:

- ◆ Use a multi-touch “pinch” or “expand” gesture.
- ◆ Drag the resize box, in the bottom right-hand corner of the Loupe.
- ◆ With the **control** key held down, press the up or down arrow keys (Mac).

To change the loupe magnification:

Do one of the following:

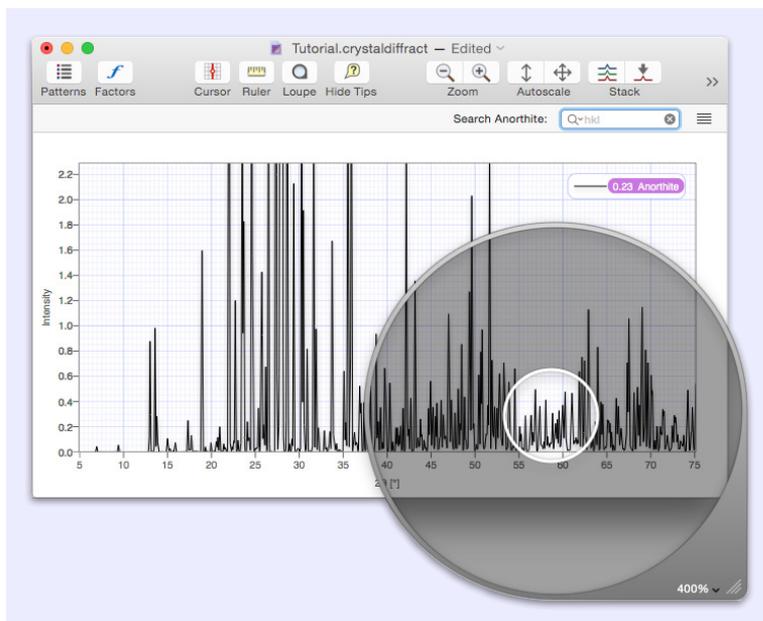
- ◆ Position the mouse pointer inside the Loupe, then use a two-fingered vertical slide gesture (up to increase the scale; down to reduce the scale)
- ◆ Choose a fixed scale from the Loupe’s popup menu or its contextual menu.
- ◆ With the **command** key held down, press the up or down arrow keys (Mac).

To reposition the Loupe:

Do one of the following:

- ◆ Click-and-drag the Loupe frame. The magnified image is updated as you move the Loupe.
- ◆ Use the arrow keys on your keyboard (press **shift** for faster movement).
- ◆ Click-and-drag the Loupe lens. The magnified image vanishes and is replaced by representation of the magnified image area: a see-through circle or square cutout. This makes it easier to position the Loupe over the exact region of interest.

The Loupe showing its magnified image area (inner circle) with the un-magnified diffraction pattern visible within.



To hide the Loupe:

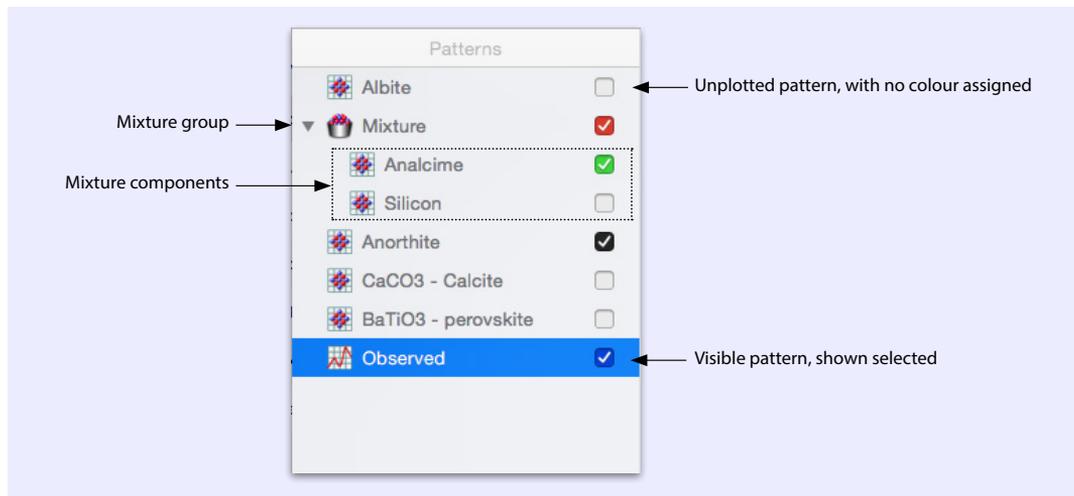
Do one of the following:

- ◆ Click the **Loupe** button in the Toolbar.
- ◆ Choose: **View > Overlays > Hide Loupe**.
- ◆ Choose: **Hide Loupe** command from the Loupe's contextual or popup menus.

Using the Patterns List

The Patterns List provides access to all diffraction patterns in a window, letting you control pattern selection, visibility, plotting order and mixtures. You can rename patterns, duplicate or delete them. The Actions popup menu () has additional commands for automatic sorting or colouring.

The Patterns List can be resized by clicking-and-dragging its right-hand edge.



The Patterns List explained.

To show the Patterns List:

Do one of the following:

- ◆ Click the **Patterns** button in the Toolbar.
- ◆ Choose: **View > Layout > Show Patterns List**.

To add patterns to the list:

Do one of the following:

- ◆ Click and drag one or more diffraction files from your operating system into the Patterns List.

- ◆ Click the toolbar's **Add** button and choose: **Add Pattern from File** from its popup menu.
- ◆ Choose: **File > Open in Same Window**, then use the navigation dialog to specify the file you want to add.

To show or hide a diffraction pattern:

- ◆ Use the pattern's visibility checkbox to toggle its plot state.

To delete one or more diffraction patterns:

- 1 Select the patterns you wish to delete by **shift**- or **command**-clicking on them in the Patterns List.
- 2 Right-click in the Patterns List and choose: **Delete** from the contextual menu.

To duplicate a diffraction pattern:

Do one of the following:

- ◆ Hold down the **option/alt** key, then click-and-drag the pattern to duplicate it.
- ◆ Select a pattern, then right-click to display the contextual menu and choose its **Duplicate** command.

To copy a diffraction pattern to a different window:

- ◆ Drag the pattern from the Patterns List, into a different CrystalDiffract window.

To change the order in which patterns are plotted:

Do one of the following:

- ◆ Click-and-drag pattern names to rearrange them.
- ◆ Use the **Sort** submenu in the Patterns List contextual menu to automatically sort the patterns by title, colour, visibility or kind (simulated versus observed). To reverse the direction of the sort, hold down the **shift** key as you select the menu command.

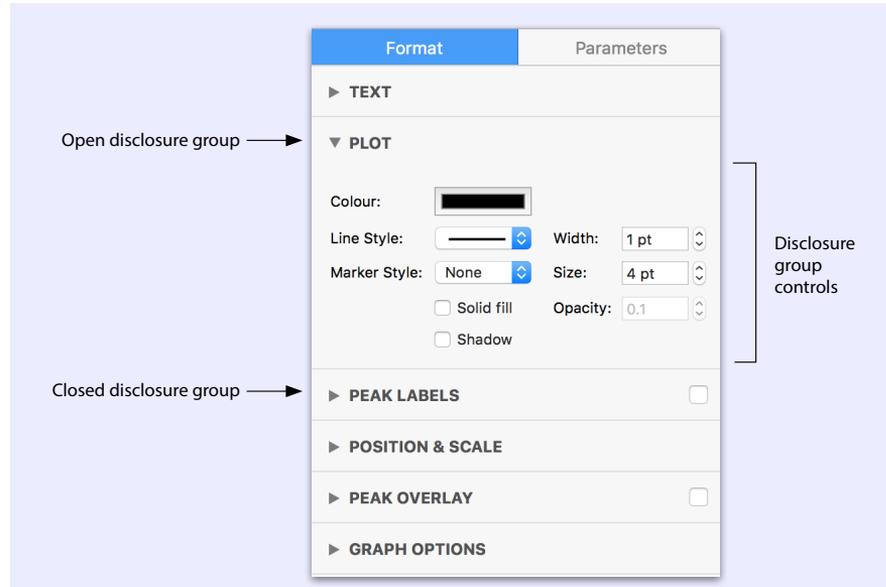
To automatically colour the patterns:

- ◆ Right-click in the Patterns List to display a contextual menu, then choose a command from its **Colorize** submenu:
 - Choose: **Reset Colours** to have CrystalDiffract apply contrasting colours to your patterns.
 - Choose: **Apply Spectrum Colours** to apply a smooth colour range from red to blue. (To reverse the colouring, from blue-to-red, hold down the shift key as you select the menu command.)

Using the Format Inspector

Clicking the **Format** tab in the Inspector pane reveals an inspector with extensive controls for changing colours, line and marker styles, labelling, scaling and data offsets. As with the Parameters Inspector, formatting controls are divided into groups, controlled by disclosure buttons.

Anatomy of the Format Inspector.



To show the Format Inspector:

- 1 Ensure the Inspector pane is visible by clicking the **Inspector** button in the toolbar, or by choosing: **View > Layout > Show Inspector**.
- 2 Click the **Format** tab.

To expand or collapse individual groups:

- ◆ Click the relevant group button to show or hide its controls.

To expand or collapse all groups:

- ◆ Right-click in the Inspector and choose **Expand List** or **Collapse List** from the contextual menu.

Fixed Width: The Inspector has a fixed width and cannot be resized horizontally. CrystalDiffract will automatically hide any disclosure groups that are not relevant to the current pattern selection.

Graph Parameters

CrystalDiffract gives you extensive control over the way in which your diffraction patterns are plotted in “Graph Mode”, including colour, line style/width, marker size/width, shadows, opacity and fill-from zero.

Quick Style Adjustments

You can use the **Pattern** menu to change plotting styles for any *selected* patterns in *Graph Mode*.

To change the pattern colour:

- ◆ Choose: **Pattern > Colour** and use the colour picker.

To change the line width:

- ◆ Choose a new width from the **Pattern > Line Width** submenu.

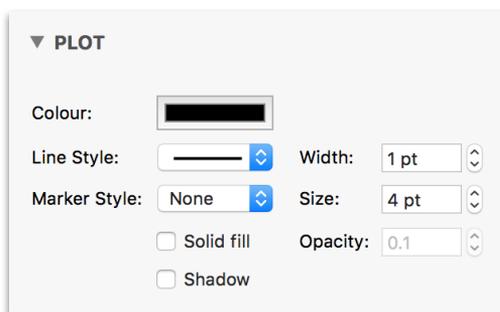
To change line and marker styles:

- ◆ Use the **Pattern > Line Width** and **Marker Style** submenus.

Plot Control

Pattern graph styles are summarized in the **Plot** group in the Format Inspector. You can change the colour, line style/width, marker sizes/styles, etc., for any *selected* patterns in *Graph Mode*.

The Plot group in the Format Inspector.



By default, CrystalDiffract uses markers for observed patterns and lines for simulated patterns, but you are free to change these.

Slow Shadows: If you have very many data points, using shadows can increase plotting times and make the program appear sluggish.

Gridlines

By default, CrystalDiffract shows gridlines for any graphs that you display in the Graphics Pane or in the Scattering Factors window. These help to give a sense of scale, but can be customized: you can change the colours (and opacities of the gridlines), or hide them completely.

To show or hide gridlines:

Do one of the following:

- ◆ Set the **Show gridlines** checkbox in the **Graph Options** group.
- ◆ Choose: **View > Grid > Show/Hide Gridlines**.
- ◆ Right-click in the graph and choose **Show/Hide Gridlines** from the contextual menu.

To change the grid colour/opacity:

- ◆ Use the colour checkbox in the **Graph Options** group of the Format Inspector.
- ◆ Choose: **View > Grid > Grid Colour**.

Coloured Graph Background

By default, CrystalDiffract displays graphs over a white window background, but the graph area itself is transparent. This is not usually a problem, but if you wish to copy graphics into another program, you may wish to make the graph area opaque, to improve clarity (e.g., if the graph is to be displayed over a coloured or textured background).

To use a coloured graph background:

- 1 Set the **Use solid fill** checkbox in the **Graph Options** group of the Format Inspector.
- 2 Use the **Colour** button to choose a fill colour.

Save a Preference: If you regularly use solid-fill graph backgrounds, you can record this as a preference, using the **Display** pane of the Preferences dialog.

Film Parameters

CrystalDiffract lets you display diffraction patterns as simulated “film strips”, in which the intensity of a diffraction peak is mapped to a colour (or greyscale value), and drawn as a vertical stripe.

Film Style

When working “Film Mode” you can choose from a number of different film simulations, including the traditional greyscale “negative” or “positive”, or more colourful displays in which the intensity is represented by a scale of colours. Use the **View > Film Type** command to change the display.

Film Gamma

CrystalDiffract also lets you tweak the way in which your simulated “film” responds to intensity, in terms of a Gamma parameter. Unlike modern digital detectors, traditional photographic film has a characteristic, *non-linear* response, with a smooth “roll-off” in sensitivity at higher intensities. This makes it easier to record weaker reflexions in the presence of stronger ones.

Definition of Gamma

A film-like intensity response can be expressed using the optical density formula:

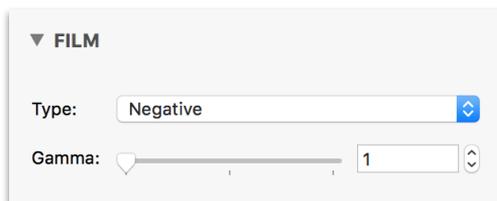
$$\text{Optical density} = \log(\text{opacity}) = \gamma \log(I)$$

$$\text{Thus: } \text{opacity} \propto I^\gamma$$

Where: “opacity” is the relative darkness of the reflexion seen on film or on the computer screen; and I is the intensity of the reflexion and γ (“gamma”) is a constant.

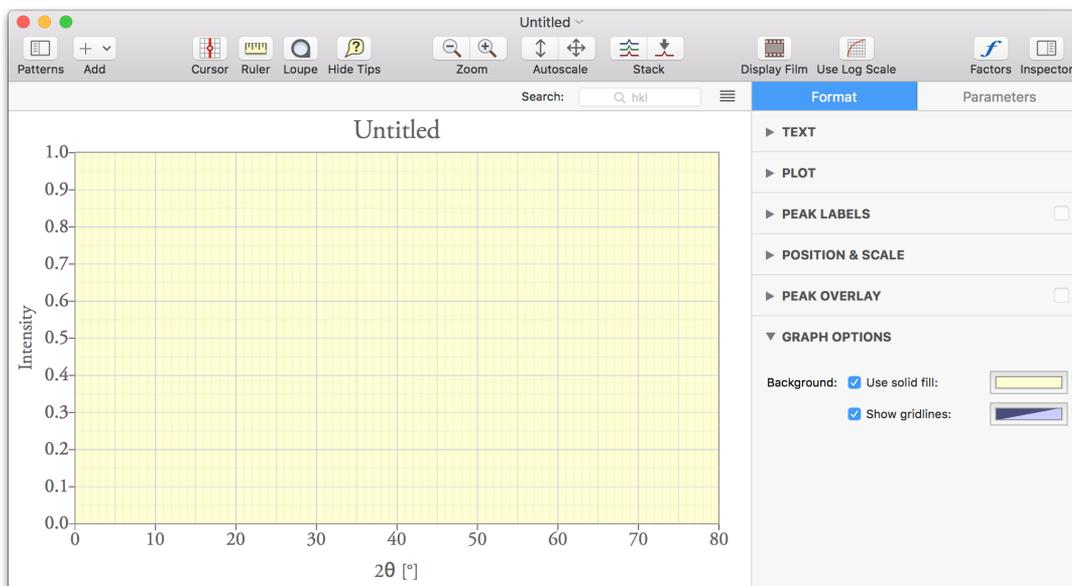
Gamma values greater than 1 help to “compress” the higher intensities (highlights) and emphasize the weaker reflexions. A typical gamma value might be around 2.2, but you can use even higher values if you wish CrystalDiffract allows you to specify the value of “Gamma”, using the **Film** group in the Format Inspector:

The Film group in the Format Inspector.



Adding embellishments

You can annotate your plot by adding a title at the top, and a legend inside the plot area. The legend can also be used to display intensity values, as measured at the cursor position. You can control the text font and size, enable or disable gridlines, and change the grid colour.



Gridlines and a coloured graph background: options available from the Graph Options group in the Format Inspector.

Plot Title

By default, the Graphics pane is untitled. However, when preparing output for illustrations or publication, you might want to have a graph title appear above your plot. You can easily preview this in the program, as follows:

To show or hide the plot title:

Do one of the following:

- ◆ Click the **Plot title** checkbox in the **Text** group of the Format Inspector.
- ◆ Choose: **View > Annotation > Show/Hide Plot Title**. If this is a new title, you will be prompted to enter text in a sheet or dialog.

To edit the plot title:

Do one of the following:

- ◆ Double-click the title.

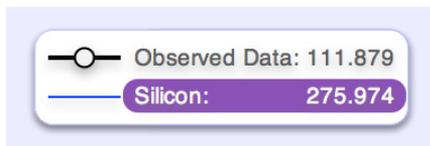
- ◆ Choose: **View > Annotation > Set Plot Title**.

You can then edit the text in the editing sheet or dialog that appears.

Legend

The Legend appears in the top right-hand corner of your graphs, and within each film strip in “film mode”. It has two purposes. Firstly, it helps identify which profile relates to which pattern. Secondly, when measuring your plot using the Cursor tool, the y -values for each pattern are shown next to their respective names in the Legend. This makes it much easier to read data in a crowded plot.

The Legend



To show or hide the legend:

Do one of the following:

- ◆ Click the **Legend** checkbox in the **Text** group of the Format Inspector.
- ◆ Choose: **View > Annotation > Show/Hide Legend**.

Text Sizes and Styles

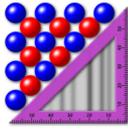
CrystalDiffract uses a consistent set of font sizes for the Graphics pane. For example, axis labels, tickmark labels, legend and title displays. You can control the master font and text size at any time, using the standard fonts command on your system. The individual sizes of the text elements will be adjusted, based on your settings.

Changing text font and size:

Do one of the following:

- Use the font controls in the **Text** group of the Format Inspector.
- Choose: **Edit > Show Fonts** and use the fonts panel.

Global Font Size: The title and legend text are drawn at a scale that is relative to other text in your plot. This makes it easier for you to control text styles, since you only need to set a single font/size for the entire plot.



Chapter 9

Measurement

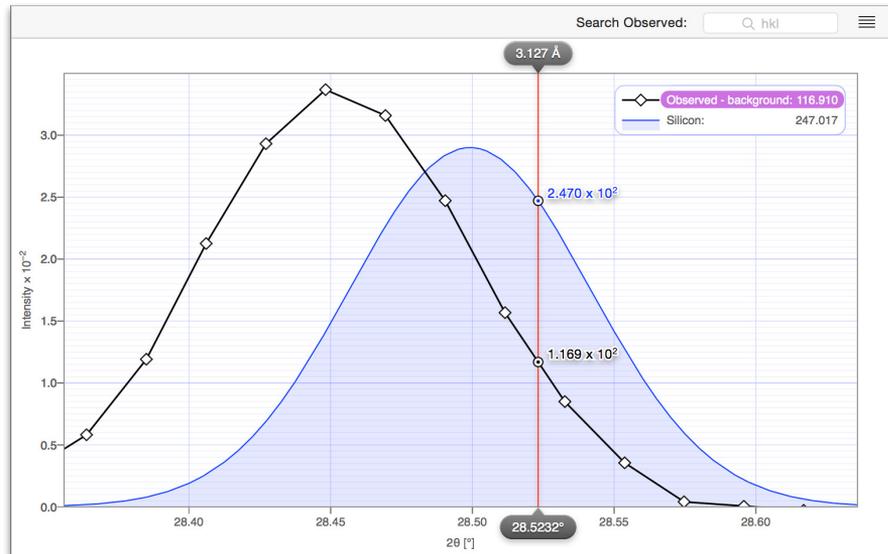
CrystalDiffract lets you measure observed and simulated diffraction data in various ways: measuring points, distances, identifying peaks, or browsing them in the Reflexions List. This chapter describes how to use screen tools including the Cursor and Ruler; how to show live peak information as you move the cursor across the screen, reflexion labels—and how to search for specific reflexions.

The Cursor Tool

The Cursor lets you measure x - and y -values for all displayed patterns. A thin red line marks the cursor's position on the graph or film, with its x -value shown above and below the display area in screen units (below), and in Ångstroms (above).

If you have any patterns displayed, then the y -value at the cursor position is labelled directly on the pattern and on the right-hand side of the Legend, as illustrated below:

The cursor in action. Y-values are displayed on the graph and in the Legend.



To show or hide the cursor:

Do one of the following:

- ◆ Click, and release the mouse, in the graph area.
- ◆ Click the **Cursor** button in the Toolbar.
- ◆ Choose: **View > Overlays > Show/Hide Cursor**.

To move the cursor

Do one of the following:

- ◆ Click and drag the cursor with the mouse.
- ◆ Use the left- or right-arrow keys on your keyboard.

Measure Scattering Factors: You can use the Cursor to measure atomic scattering factors, in the Scattering Factors window. Just click anywhere in that window's Graphics Pane to display the cursor; click again to hide the cursor, or right-click in the window and use the contextual menu to toggle the cursor's visibility.

Using the Ruler

The Ruler lets you measure horizontal distances on the plot, as well as letting you reset the plot range to the displayed cursors.

The Ruler provides a translucent overlay which you can click-and-drag to reposition. When selected, the ruler shows a pair of vertical cursors passing through its two holes. These are intersected by a horizontal cursor, which records the vertical position. You can use the cursors to precisely position the ruler.

To show or hide the ruler:

Do one of the following:

- ◆ Click the **Ruler** button in the toolbar.
- ◆ Choose: **View > Overlays > Show/Hide Ruler**.

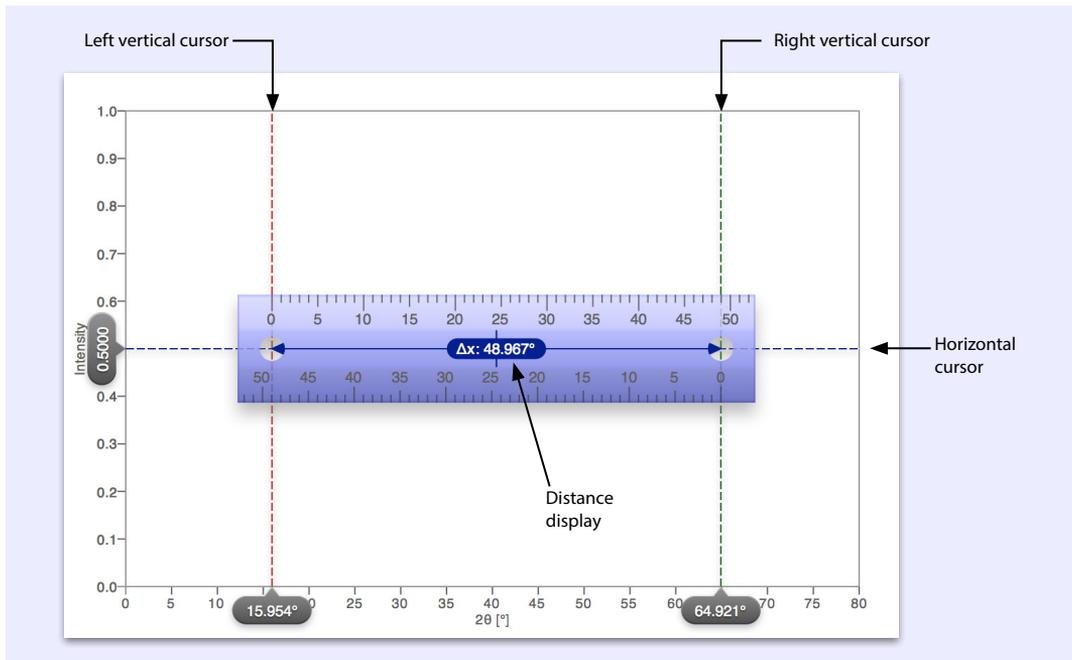
To move the ruler:

Do one of the following:

- ◆ Click and drag the ruler with the mouse.
- ◆ Click-and-drag a vertical (red or green dashes) or horizontal (blue dashes) cursor.

To resize the ruler:

- ◆ Click-and-drag a vertical cursor.



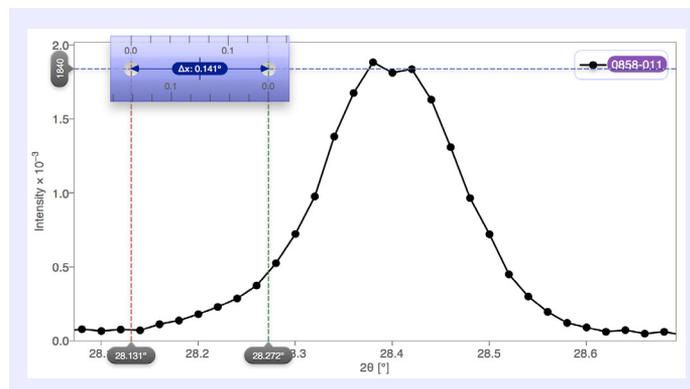
The Ruler in its selected state.

To measure a peak's Full-Width at Half Maximum (FWHM):

Before you start, for the maximum precision, make your window as large as possible and adjust the scale so that the peak fills the screen.

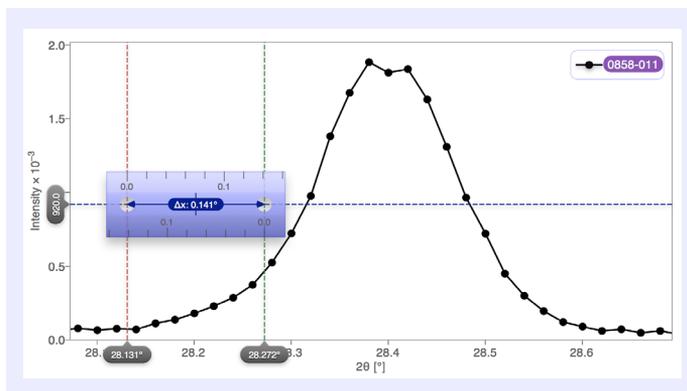
- 1 Position the ruler's horizontal cursor at the top of the peak. Note that the ruler measurement is hidden when you move the ruler, which makes it easier to locate the centre line. You may also wish to slide the ruler to one side, so you can clearly identify the peak maximum, as shown below:

Measuring the peak maximum.



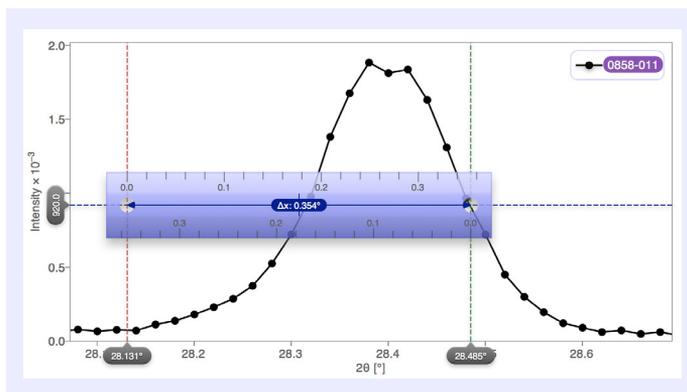
- Right-click on the ruler and choose: **Move Down 50%**. The ruler moves vertically downwards to half the peak maximum.

The Ruler at half peak-maximum.



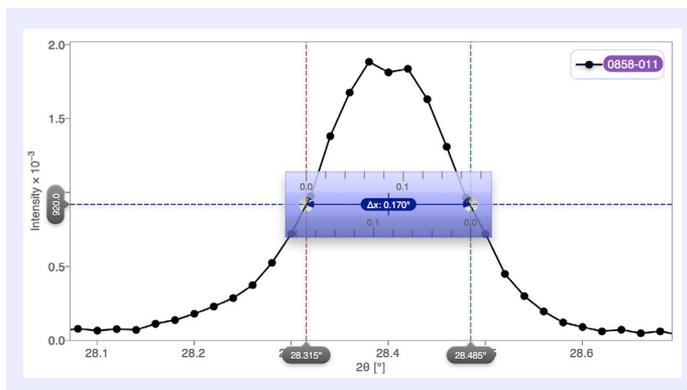
- Click and drag the ruler's right vertical cursor (green dash line = "starboard") so it intersects the right-hand shoulder of the peak:

Locating the peak's right-hand shoulder.



- Click and drag the ruler's left vertical cursor (red dash line = "port") so it intersects left-hand shoulder of the peak. The FWHM is now displayed in the ruler.

Measuring the peak width at half-maximum.



To zoom the plot range:

- 1 Adjust the ruler's vertical cursors to specify the minimum and maximum x -axis limits.
- 2 Adjust the ruler's horizontal cursor to specify the maximum y -axis limit.
- 3 Right-click on the ruler to display its contextual menu, then choose: **Zoom to Cursors**.

To copy the ruler measurement(s):

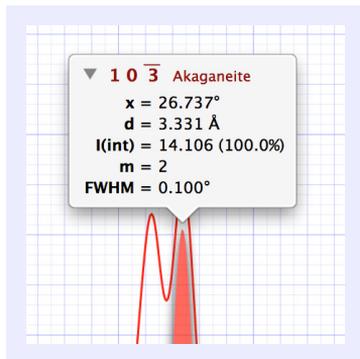
- ◆ Right-click on the ruler and choose a command from the contextual menu, either: **Copy Δx** or **Copy Intensity**.

Colouring & Transparency: You can change the ruler's colour and transparency. Right-click on the ruler and choose the **Change Colour** command.

Peak Tips

Peak Tips are little “tool tips” that appear when the mouse lies close to the position of a *simulated* peak.

Peak tips in action: identifying a peak in a multi-phase diffraction simulation.



Peak tips can appear in one of two states: collapsed, or expanded—as indicated by the “disclosure triangle”. In their default, collapsed form, peak tips identify the Miller indices of the reflexion giving rise to the peak, and the name of the pattern. If you expand the peak tip (by clicking inside it), additional data are shown, including the x -value, d -spacing, integrated intensity, $I(\text{int})$ and multiplicity (m).

To enable or disable peak tips:

Do one of the following:

- ◆ Click the **Show/Hide Tips** button () in the toolbar.
- ◆ Choose: **View > Overlays > Show/Hide Peak Tips**.

To show a peak tip for a simulated pattern:

- ◆ Move the mouse pointer so it lies over the reflexion of interest. The reflexion should be shown highlighted, and a peak tip is shown.

(Note that if several reflexions overlap, CrystalDiffract will identify the most-intense reflexion.)

To toggle the peak tip's state:

- ◆ Click anywhere inside the peak tip.

To get more information about a reflexion:

- 1 Position the mouse over the peak, so it appears highlighted.
- 2 Double-click the highlighted peak. The Reflexions List appears, with the corresponding list row selected.

To change your Peak Tip preferences:

- ◆ Use the **General** pane of the Preferences window to control whether Peak Tips should be automatically enabled.

Highlighted Peaks Too Weak?

If you find that your highlighted peaks appear to be too weak (when compared to the overall profile), it is likely that your structure has missing symmetry: most likely, an incomplete spacegroup. You should check the Reflexions List (described later in this chapter) to see if multiple reflexions exist at the same d-spacing, and with symmetry-related indices.

For example, a cubic crystal might be expected to show a peak at 100 that incorporates contributions from the six *symmetry-related* sets of planes, 100, $\bar{1}00$, 010, $0\bar{1}0$, 001 and $00\bar{1}$.

However, if for some reason cubic symmetry had not been specified for that crystal, e.g., the structure had been entered with a default spacegroup of “P 1”, then there is no way in which CrystalDiffract can relate a plane such as 100 to one in a different orientation such as 010 or 001.

(CrystalDiffract will not automatically merge reflexions simply because they have the same d-spacing; that might cause confusion when trying to follow structural behaviour with changing temperature or pressure, when independent reflexions “cross over” each other as the unit cell distorts.)

Thus, a *metrically-cubic* lattice with a non-cubic, “P 1” spacegroup, would result in a set of “independent” reflexions at the same d-spacing, each with its own, superimposed peak tip. For example, we would find 100, 010 and 001 all listed separately. This would result in a displayed peak tip (e.g., 100) appearing much weaker than expected.

The solution in this case would be to review the crystal structure, using the **Edit > Structure** command, and enter the correct, *cubic*, spacegroup.

Peak Overlays

If you have a complex diffraction pattern with many overlapping peaks, it can be useful to show the individual peaks as an “overlay”, above the diffraction profile.

To show or hide the peak overlay:

Ensure you have selected the appropriate pattern(s), then do one of the following:

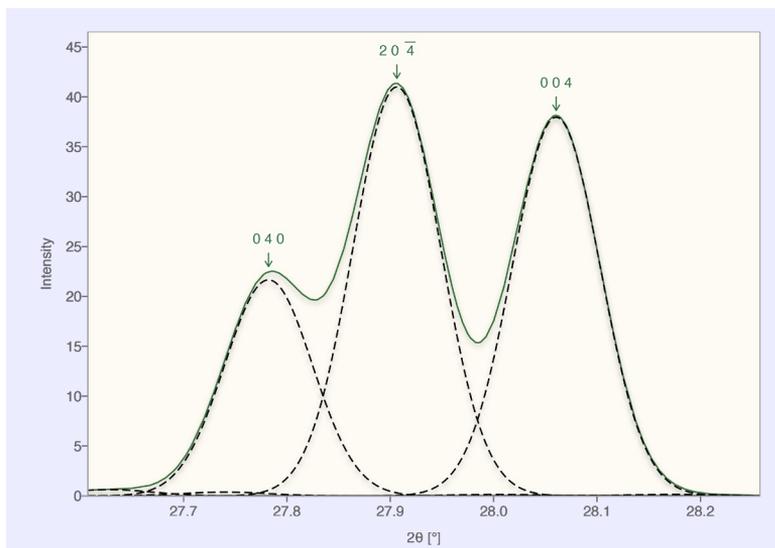
- ◆ Choose: **Pattern > Show/Hide Peak Overlay**.
- ◆ Use the checkbox in the **Peak Overlay** group of the Format Inspector.

To change how the overlay is plotted:

- ◆ Use **Peak Overlay** group in the Format Inspector to edit the overlay colour, line style, and line width.

Slow Overlays: Showing peak overlays may have a significant impact on plotting speed. You should only use the overlay for high-magnification work with a select few patterns.

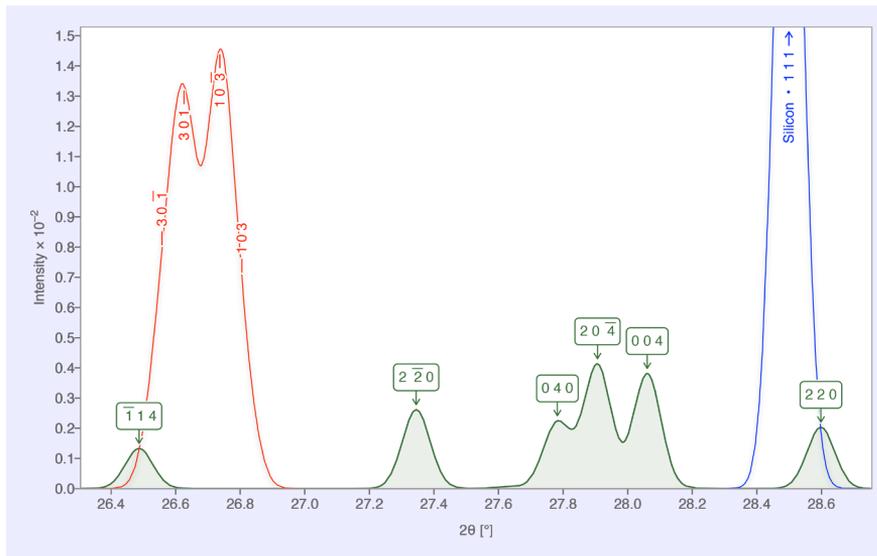
Peak overlay (black dashes) showing the form of individual diffraction peaks, compared with the overall profile (green curve).



Labelling Peaks

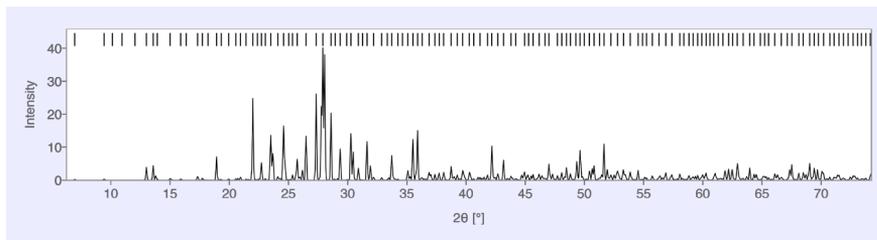
You can display information labels for *simulated* diffraction peaks, in order to emphasize their locations, Miller Indices, d-spacings, or other information.

Different kinds of peak labelling for a multi-pattern display, with frames, shadows, arrows and rotated text.



CrystalDiffract lets you choose the label content and provides a variety of different label options. These include adding lines, arrows, outlines or shadows to the label text. Alternatively, you can omit any text and simply use lines or arrows to indicate peak positions. Labels can be positioned “floating” above the diffraction profile, or fixed to the top or bottom of the graph, as the following example shows:

Labels without text, indicating peak positions in a complex profile.



Labelling can be applied to specific patterns, and you can control how many labels to be shown. Generally, one wishes to label the more intense peaks, rather than every single peak. This can be achieved in one of two ways: you can either specify a minimum intensity “threshold”, below which peaks will remain unlabelled.

Another option is to specify that any *overlapping* peak labels should be hidden: CrystalDiffract will plot labels for the strongest (most-intense) diffraction peaks, hiding any labels for weaker peaks that overlap the stronger labels.

Labelling can be controlled via the Pattern menu (for selected patterns) or, for more control, via the **Peak Labels** group in the Format Inspector:

Labelling options, displayed in the Format Inspector.

To show labels:

Select the pattern(s) to be labelled then do one of the following:

- ◆ Choose: **Pattern > Show Labels**.
- ◆ Set the **Show Labels** checkbox in the **Peak Labels** group of the Format Inspector.

To control how many peaks should be labelled:

Select the pattern(s) to be labelled, then do one of the following:

- ◆ Choose: **Pattern > Label Threshold** and select a higher threshold value (to label fewer peaks), or a lower value (to label more peaks).
- ◆ Adjust the **Threshold** setting in the **Peak Labels** group of the Format Inspector.

To change the label content:

Select the pattern(s) to be labelled, then do one of the following:

- ◆ Choose: **Pattern > Label Content** and select the items to be shown.
- ◆ Set the relevant **Content** checkboxes in the **Peak Labels** group of the Format Inspector.

To change the label position, colour or style:

- 1 Select the pattern(s) to label.
- 2 Use the colour, position and style controls in the **Peak Labels** group of the Format Inspector.

To hide overlapping labels:

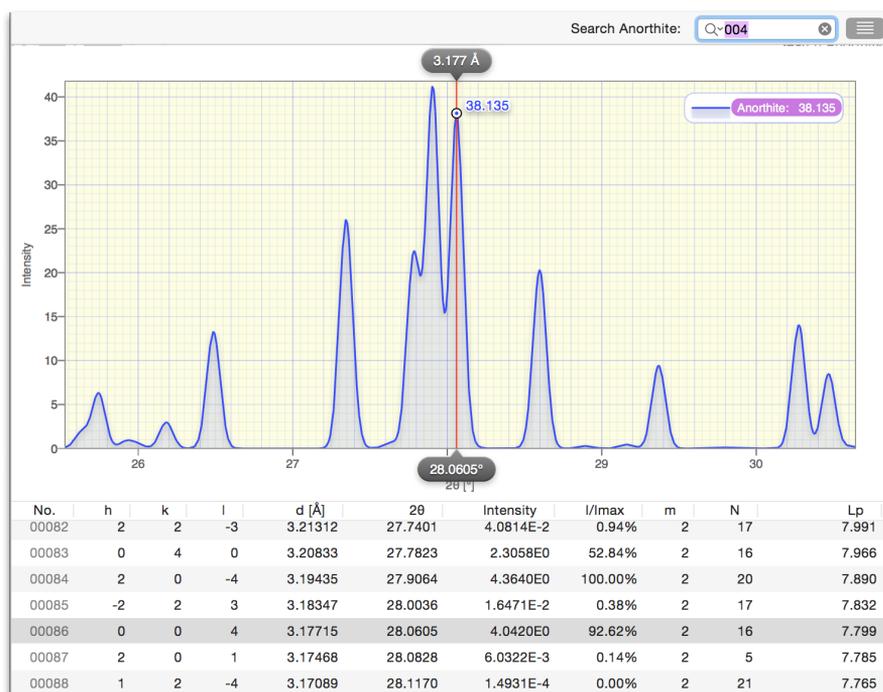
- ◆ Set the **Hide Overlapping Labels** option (in the **Peak Labels** group of the Format Inspector). Any labels for weaker peaks that overlap labels for stronger peaks will be hidden.

Slow Labels: Displaying many peak labels will increase the time taken to plot. For best results, choose a high threshold, so that only the most-intense peaks are labelled. Alternatively, specify the “Hide Overlapping Labels” option, to hide any (weaker) peak labels that overlap with stronger labels.

Reflexions List

CrystalDiffract lets you search for a reflexion in the currently-selected pattern. The result is shown in the Graphics Pane, and in a Reflexions List. You can use the Reflexions List to browse and sort all simulated reflexions for the current, selected pattern.

Searching for the 004 reflexion in a feldspar mineral. The reflexion's position in the Graphics pane is shown by the Cursor and its entry is highlighted in the Reflexions List below.



The reflexions list shows all of the *symmetrically-distinct* reflexions used to simulate the currently-selected diffraction pattern (CrystalDiffract uses the spacegroup symmetry to identify symmetrically-related reflexions, and these are merged to give a single reflexion with a *multiplicity* greater than one). The columns (which you can re-order by clicking-and-dragging the column headers) provide the following data:

- **No.** - The reflexion number, starting with the highest d-spacing reflexions first.
- **hkl** - The Miller Indices of the reflexion. Note that CrystalDiffract merges symmetrically-related reflexions into a single entry, so in the example above, the selected reflexion 004 includes a contribution from 00 $\bar{4}$. In cases where more than 2 reflexions are related, CrystalDiffract chooses the simplest arrangement of indices, e.g., in a cubic crystal, the reflexion labelled 100 would include contributions from six sets of planes, 100, $\bar{1}00$, 010, $0\bar{1}0$, 001 and $00\bar{1}$.
- **d [Å]** - The d-spacing, in Ångström units.
- **Intensity** - The *integrated intensity* of the reflexion, that is, the complete intensity scattered by the sets of planes contributing to this reflexion, as measured over the full peak area. Please note that this is not the same as the *peak height*.
- **I/Imax** - The *relative intensity* of the reflexion, expressed as a percentage, and relative to the most-intense reflexion for this pattern.
- **m** - The *multiplicity* of the reflexion. This is the total number of *symmetrically-related* reflexions that have been combined into this row. For example, the selected row in the Reflexions List pictured above shows an 004 reflexion with $m = 2$. This is because, for the purposes of diffraction, 004 is related to $00\bar{4}$, and hence the two reflexions have been combined. A cubic crystal might show a 100 reflexion with a multiplicity of 6, indicating contributions from the six symmetrically-related reflexions 100, $\bar{1}00$, 010, $0\bar{1}0$, 001 and $00\bar{1}$.
- **N** - The sum of the squares of the Miller Indices for the reflexion, i.e., $N = h^2 + k^2 + l^2$.
- **Lp** - The *Lorentz-Polarization* factor for this reflexion (see *Chapter 4: Principles of Diffraction*).

To show the Reflexions List:

Select the pattern of interest, then do one of the following:

- ◆ Choose: **View > Layout > Show Reflexions List**.
- ◆ Click the Reflexions List button () to the right of the **Search** field.

To sort the list:

- ◆ Click a column header button to sort according to that parameter; click again to reverse the direction of the sort.

To resize the Reflexions List:

- ◆ Click and drag the (thin) divider line, between the Graphics pane and the Reflexions List.

To view data for a particular pattern:

- ◆ Select the pattern whose data you wish to view (e.g., by clicking on its name in the Patterns List, or by choosing its name from the **Search** menu in the Search Bar).

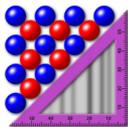
To find a reflexion:

- ◆ Type the Miller Indices for your reflexion in the Search field and press the **return** or **enter** keys on your keyboard. The position of your reflexion is then indicated by the Cursor, and highlighted in the Reflexions List.

Quick-Type Indices: For low-index reflexions, you can type the indices without spaces, e.g., "111". For higher-index reflexions, ensure you enter spaces between the indices, e.g., "10 2 5".

To scroll the pattern to a reflexion in the list:

- ◆ Double-click on the reflexion's row in the list.



Chapter 10

Output

CrystalDiffract lets you save your work in a self-contained CrystalDiffract document, and record your program preferences. You can also export diffraction profiles, tables of diffraction data, structure factors, atomic scattering factors, crystal structures and graphics.

This chapter guides you through the various ways in which you can export diffraction and crystal structural data, including direct structural visualization in CrystalMaker. The chapter also explains how to copy graphics to the clipboard or save them to a graphics file.

Demonstration Version

File output is supported by the full-feature version of CrystalDiffract, but not in the free Demonstration Version. If you are using the software enough to require file output, please purchase a licence.

CrystalDiffract Document

The full-feature version of CrystalDiffract lets you save your work in a self-contained document, with file extension “.diffract” or “.crystaldiffract”. This contains all details of any crystal structures, the simulated reflexions and your plot settings—ready for rapid display next time you open the document.

CrystalDiffract documents are cross-platform compatible between the Mac and Windows versions of the program. However, they are not compatible with earlier versions of the software.

Legacy Document Support

CrystalDiffract 6 for Mac and Windows can open, but not save, documents created by the previous version of the software, CrystalDiffract 5 for Mac or CrystalDiffract 1 for Windows.

Exporting Diffraction Data

CrystalDiffract lets you export diffraction data in various formats. You can simply save the plotted diffraction pattern as a series of (x, y) values, in a text file. For simulated data, you also have the option of exporting a complete reflexions list or, for more specialized use, you can export a table of Structure Factors.

Diffraction Profile

You can export the *displayed* profile for any selected pattern to a text file. This will then comprise a simple x, y listing of data points (in the case of an “observed” pattern) or profile points (for a simulated pattern). Note that the exported profile will match that plotted on screen, and may differ from the original data (e.g., if you have applied any offsets, x -axis scaling, mixture scaling, etc.).

Part of a Profile file for spinel.

```
30.9913 2.0897e-01
31.0038 2.2743e-01
31.0163 2.4840e-01
31.0288 2.7238e-01
31.0413 2.9996e-01
31.0538 3.3189e-01
31.0663 3.6915e-01
31.0788 4.1313e-01
31.0913 4.6602e-01
31.1038 5.3192e-01
```

To export a diffraction profile:

- 1 Select the pattern whose profile you wish to export.
- 2 Choose: **File > Export > Profile**.

Reflexions List

You can export diffraction data—as visible in the Reflexions List—to a plain text file. The file contains all reflexions used by the program, with their intensities, multiplicities and Lorentz-polarization (L_p) factors. The list is sorted using the same way as the on-screen Reflexions List.

Part of a Reflexions List file for spinel.

h	k	l	d(hkl)	2-Theta	Intensity	I/Imax	m	N	L_p
1	1	1	4.66499	19.0076	5.74246e+00	32.0%	8	3	17.609
0	2	2	2.85671	31.2843	5.78224e+00	32.2%	12	8	6.179
1	1	3	2.43621	36.8626	1.79489e+01	100.0%	24	11	4.324
2	2	2	2.33249	38.5650	2.22031e-01	1.2%	8	12	3.914
0	0	4	2.02000	44.8299	1.05848e+01	59.0%	6	16	2.795
1	3	3	1.85368	49.1048	1.48332e-02	0.1%	24	19	2.274
2	2	4	1.64932	55.6807	1.72168e+00	9.6%	24	24	1.708
1	1	5	1.55500	59.3842	8.19314e+00	45.6%	24	27	1.477
3	3	3	1.55500	59.3842	5.95402e-01	3.3%	8	27	1.477
0	4	4	1.42836	65.2661	1.41208e+01	78.7%	12	32	1.200
1	3	5	1.36577	68.6613	6.48416e-01	3.6%	48	35	1.078
2	4	4	1.34667	69.7751	2.73966e-02	0.2%	24	36	1.043
0	2	6	1.27756	74.1568	5.62998e-01	3.1%	24	40	0.926

To export a table of simulated reflexions:

- 1 Select the (simulated) pattern whose reflexions you wish to export.
- 2 Choose: **File > Export > Diffraction Data.**

Structure Factors

If you are interested in a more detailed diffraction output, you can save a table of Structure Factors for all symmetrically-distinct reflexions. The resulting file includes d-spacings, phase angles, intensities—plus the real and imaginary parts of the Structure Factor: F(Re) and F(Im), respectively.

Part of a Structure Factors file for spinel.

ref no.	h	k	l	d(hkl)	Phase	Lp	F(Re)	F(Im)	Intensity
[1]	1	1	1	4.6650	0.000	17.61	55.70	-0.00	5.74246e+00
[2]	0	2	2	2.8567	180.000	6.18	-77.04	0.00	5.78224e+00
[3]	1	1	3	2.4362	180.000	4.32	-114.74	-0.00	1.79489e+01
[4]	2	2	2	2.3325	180.000	3.91	-23.23	0.00	2.22031e-01
[5]	0	0	4	2.0200	0.000	2.80	219.17	-0.00	1.05848e+01
[6]	1	3	3	1.8537	0.000	2.27	4.55	-0.00	1.48332e-02
[7]	2	2	4	1.6493	0.000	1.71	56.54	0.00	1.72168e+00
[8]	1	1	5	1.5550	0.000	1.48	132.63	-0.00	8.19314e+00
[9]	3	3	3	1.5550	0.000	1.48	61.93	0.00	5.95402e-01
[10]	0	4	4	1.4284	0.000	1.20	273.25	-0.00	1.41208e+01
[11]	1	3	5	1.3658	180.000	1.08	-30.89	-0.00	6.48416e-01
[12]	2	4	4	1.3467	0.000	1.04	9.13	-0.00	2.73966e-02
[13]	0	2	6	1.2776	0.000	0.93	43.90	0.00	5.62998e-01

To export a table of structure factors:

- 1 Select the (simulated) pattern whose structure factors you wish to export.
- 2 Choose: **File > Export > Structure Factors.**

Scattering Factors

You can export your current table of atomic scattering factors & neutron scattering lengths by using the **Export** button in the Scattering Factors window (**Edit > Scattering Factors**).

The resulting output file gives the element symbols, atomic numbers (Z), atomic weights (W), the nine coefficients of the X-ray scattering factor function (a_{1-4} , b_{1-4} , c), the neutron scattering length (b), and any notes.

	Z	W	f1 a(1)	f2 b(1)	f3 a(2)	f4 b(2)	f5 a(3)	f6 b(3)	f7 a(4)	f8 b(4)	f9 c	b
D	1	2.014	0.489918	20.659300	0.262003	7.740390	0.196767	49.551899	0.049879	2.201590	0.001305	6.6710
H	1	1.008	0.489918	20.659300	0.262003	7.740390	0.196767	49.551899	0.049879	2.201590	0.001305	-3.7390
He	2	4.003	0.873400	9.103700	0.630900	3.356800	0.311200	22.927601	0.178000	0.982100	0.006400	3.2600
Li	3	6.941	1.128200	3.954600	0.750800	1.052400	0.617500	85.390503	0.465300	168.261002	0.037700	-1.9000
Li+	3	6.941	0.696800	4.623700	0.788800	1.955700	0.341400	0.631600	0.156300	10.095300	0.016700	-1.9000
Be	4	9.012	1.591900	43.642700	1.127800	1.862300	0.539100	103.483002	0.702900	0.542000	0.038500	7.7900
B	5	10.811	2.054500	23.218500	1.332600	1.021000	1.097900	60.349800	0.706800	0.140300	-0.193200	6.6500
C	6	12.010	2.310000	20.843901	1.020000	10.207500	1.588600	0.568700	0.865000	51.651199	0.215600	6.6460
N	7	14.007	12.212600	0.005700	3.132200	9.893300	2.012500	28.997499	1.166300	0.582600	-11.529000	9.3600
O	8	15.999	3.048500	13.277100	2.286800	5.701100	1.546300	0.323900	0.867000	32.908901	0.250800	5.8030
F	9	18.998	3.539200	10.282500	2.641200	4.294400	1.517000	0.261500	1.024300	26.147600	0.277600	5.6540
OH	9	16.007	3.048500	13.277100	2.286800	5.701100	1.546300	0.323900	0.867000	32.908901	0.250800	5.8030

Part of a Scattering Factors output file.

Exporting Crystal Data

CrystalDiffract lets you export the crystal structure used to simulate a powder pattern. The structure can be saved as a text file or you can opt to visualize the structure directly, in CrystalMaker.

To visualize the crystal in CrystalMaker:

- 1 Select the (simulated) pattern whose crystal structure you wish to visualize.
- 2 Choose: **Pattern > Visualize Crystal Structure**. If you have installed CrystalMaker on your system, that program will be launched, and one unit cell of the crystal shown. You can use CrystalMaker to change the range of atoms, the model type, and provide full 3D visualization.

To save the crystal as a CIF file:

- 1 Select the (simulated) pattern whose crystal structure you wish to visualize.
- 2 Choose: **File > Export > Crystal Structure > CIF**.

To save the crystal as a CrystalMaker text file:

- 1 Select the (simulated) pattern whose crystal structure you wish to visualize.
- 2 Choose: **File > Export > Crystal Structure > CrystalMaker Text**.

Exporting Graphics

CrystalDiffract provides very-high quality vector graphics from both the Graphics pane (for diffraction patterns) or the Scattering Factors window (for graphs of atomic scattering factors). Graphics can be copied to the clipboard or dragged-and-dropped into another program:

To copy graphics to the clipboard:

- ◆ Right-click in the Graphics pane and choose: **Copy Graphics**.

To copy graphics into another program:

- ◆ Click in the Graphics pane outside the plot rectangle, then drag the image into another program.

Saving Image Files

You can also save graphics to file, in a wide variety of formats. For example, CrystalDiffract for Mac current supports the following:

- Vector Graphics: PDF and EPS
- Pixel Graphics: TIFF, PNG, JPEG, BMP and GIF

To export graphics to a graphics file:

Do one of the following:

- ◆ Right-click in the Graphics pane (or in the Graphics pane of the Scattering Factors window) and choose: **Export Graphics** from the contextual menu.
- ◆ Choose: **File > Export > Graphics**.

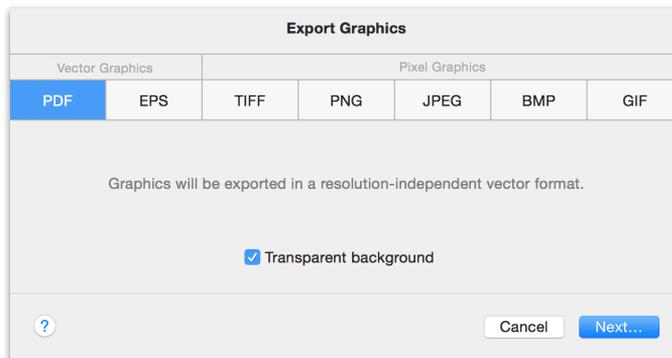
Using the Graphics Exporter

Use the Graphics Exporter panel to choose your output file format and any format-specific parameters such as:

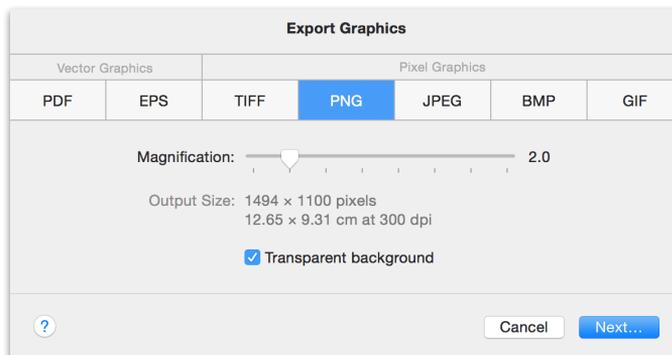
- **Magnification** (for pixel formats): higher magnification levels provide better quality, and help to compensate for the fixed resolution (non-scaleable) of pixel graphics.
- **Transparent background.** Some file formats (e.g., PDF, EPS, TIFF and PNG) provide for transparency. You can choose to either impose the same white background as the screen graphics, or use a transparent background instead.

Use Coloured Backgrounds: Use the **Graph Options** group of the Format Inspector to specify a coloured background; use set the **Use solid fill** display preference to record this as a default. If you require the entire graphic (outside the graph area) to be opaque, uncheck the **Transparent Background** option in the Graphics Exporter panel.

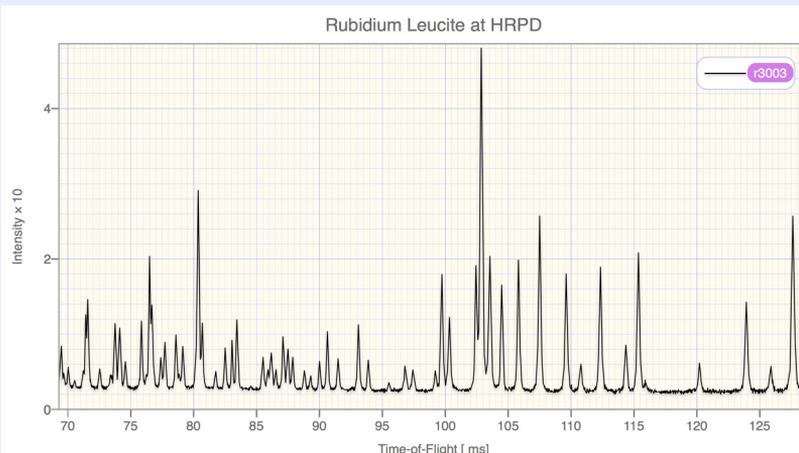
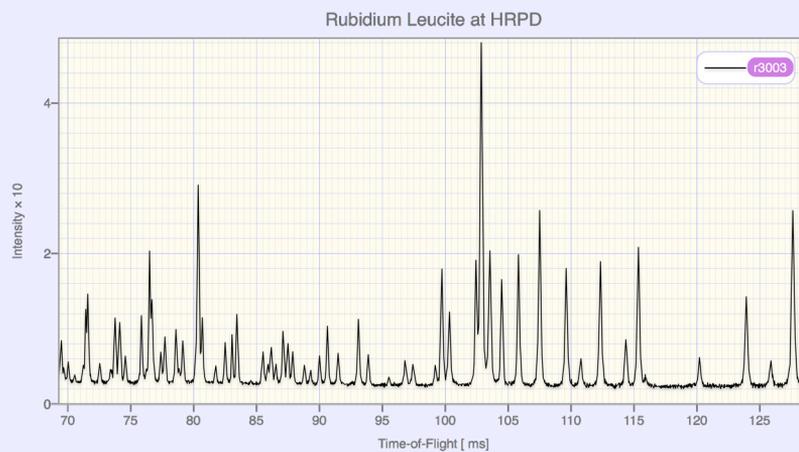
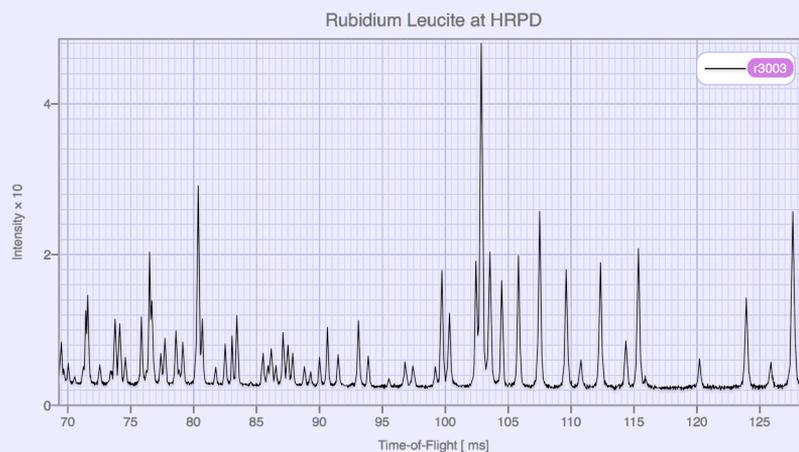
Exporting (PDF) vector graphics. Note the transparent background option.



Exporting (PNG) pixel graphics. A magnification option allows for higher-quality output.



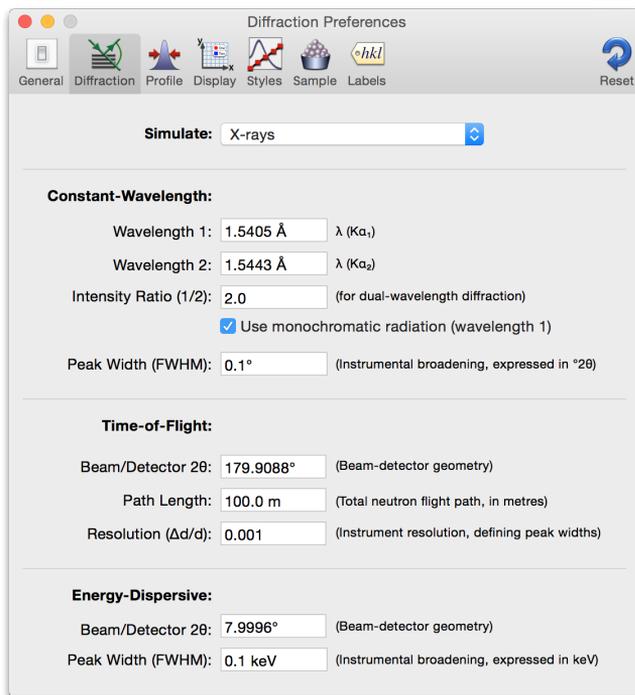
Examples of the same graphic exported with different background options. From top-to-bottom: Transparent background; solid-fill *graph* background; opaque background plus solid-fill graph background.



Editing Preferences

The full-feature version of CrystalDiffract gives you extensive control over how the program works. You can assign default settings for use with new windows, including your preferred simulation types, wavelengths and plot styles.

The Preferences panel.

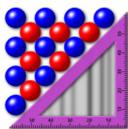


To show the Preferences panel:

- ◆ Choose: **CrystalDiffract > Preferences** (Mac) or **Edit > Preferences** (Windows).

To reset your preferences to factory values:

- ◆ Click the **Reset** button in the Preferences panel's titlebar.

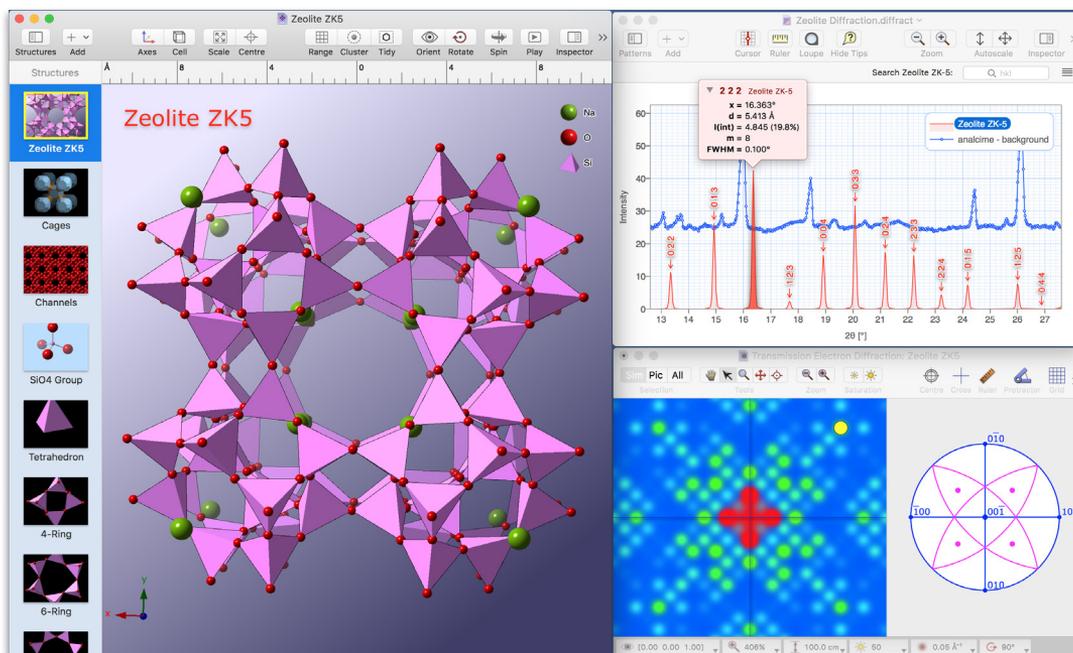


Chapter 11

The CrystalMaker Office

Different tasks require different interfaces. You wouldn't control a train with a steering wheel, and you wouldn't expect to fly a plane using a paddle. The same applies to scientific software: our "CrystalMaker Office" is modular, with components designed to give you the best user experience and performance, whilst ensuring a quick and easy workflow. We believe this is far superior to a single, bloated, "Jack-of-all-trades" application.

This chapter introduces you to the other components in the office: CrystalMaker X and SingleCrystal.

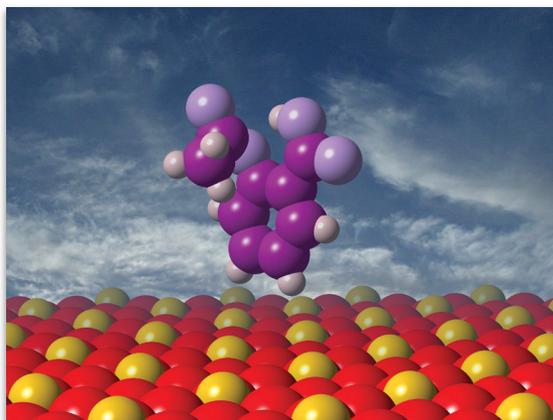


Crystal structure views of zeolite ZK5 in CrystalMaker X (left), with corresponding diffraction simulations in CrystalDiffract (top right) and SingleCrystal (bottom right).

Introduction to CrystalMaker X

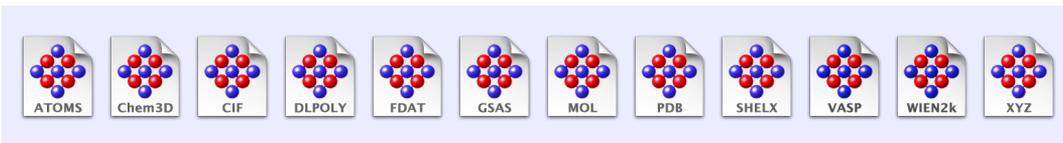
To really make the most of CrystalDiffract, you'll want CrystalMaker® X: our flagship program for crystal & molecular structures visualization and modelling. CrystalMaker features an elegant and intuitive user interface, available in optimized versions for Mac (including macOS High Sierra) and Windows.

An organic molecule and an oxide surface, as visualized in CrystalMaker.

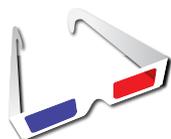


Easy Data Processing

CrystalMaker provides easy drag-and-drop import of text data files in some 40 formats, including CASTEP, CIF, DL_POLY, GULP, GSAS, MOL, PDB, SHELX, VASP, Wien2K, XYZ and more. Just drag-and-drop any files into the program for instant structure display—no cumbersome file import dialogs or convoluted menu commands. Ideal for multi-structure work, or when you need to quickly check a file. A wide variety of file export formats is also provided.



Just some of the many file formats supported by CrystalMaker X.



Spectacular Graphics

CrystalMaker provides superb, photo-realistic graphics, including stunning 3D stereo graphics in colour—with annotation and high-resolution output.

Molecular Modelling

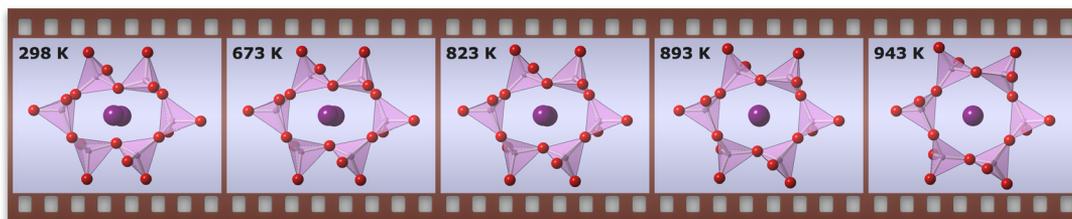
CrystalMaker offers point-and-click molecule building with energy minimization. Hydrogen atoms can be added automatically to any molecular structure.

Crystal Engineering

CrystalMaker lets you actively design new structures, combining multiple models in the same window, move atoms, add bonds, and explore molecular docking, interface boundaries and whatever else your imagination permits.

Animations and Video

CrystalMaker X is unique in providing interactive, rotatable animations, which can be saved, or exported as video files for presentations or the internet.



Structural behaviour visualized in a CrystalMaker animation: simply drag-and-drop files into the same document, synchronize their structures and press "Play". The resulting animation can be saved as a movie.

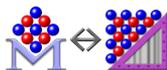
3D Model Export, iBooks and iPad

CrystalMaker can export any structure as a self-contained 3D file, suitable for manipulation in other 3D programs—or as the starting point for a 3D printer.

CrystalMaker supports the COLLADA 3D interchange format, as used by iBooks on the iPad. Simply drag-and-drop your CrystalMaker-authored 3D models into the free iBooks Author application on your Mac, connect your iPad, press "Preview", and you have fully-rotatable 3D structures on your iPad!

Structures Library

CrystalMaker X comes with an integrated library of fully-annotated files, ready for immediate display. The library, which is searchable, is presented in its own browser window. It comprises over 1000 structures, including all the major rock-forming minerals, essential organic and chemical structures, plus many advanced materials. Ideal for teaching and as a handy reference for research workers.



Diffraction Link

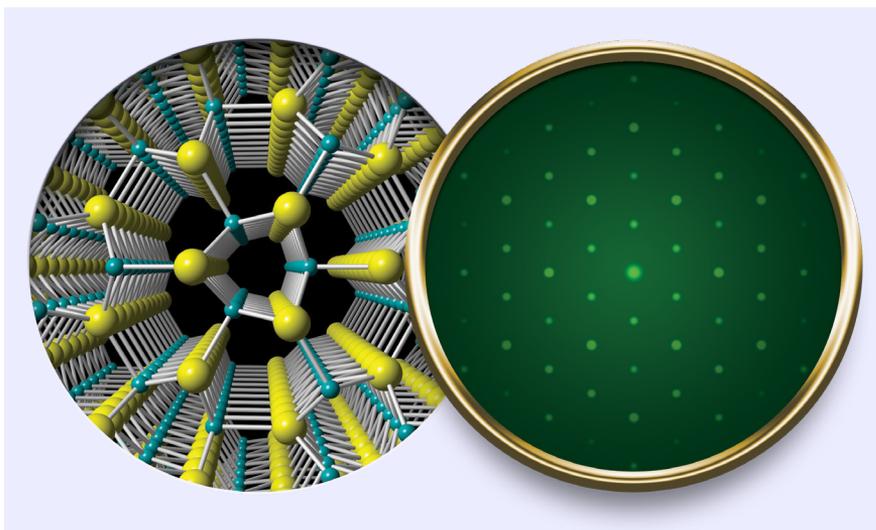
CrystalMaker lets you share your structural data with CrystalDiffract, without the need to save files, switch applications, and load data:

- In CrystalMaker, choose: **Calculate > Diffraction Pattern > Powder** and the corresponding powder pattern will be simulated in CrystalDiffract.
- In CrystalDiffract, choose: **Pattern > Visualize Crystal Structure** and the corresponding crystal structure will be displayed in CrystalMaker.

Single-Crystal Diffraction

CrystalMaker can be extended to provide single-crystal diffraction simulation and analysis, thanks to our SingleCrystal program. This reads from CrystalMaker binary files and from graphics files (e.g., JPEG images), letting you compare images of real diffraction patterns with simulated patterns. The program features easy auto-indexing of TEM diffraction patterns, plus a “live rotation link” with CrystalMaker: rotate your structure in CrystalMaker and see its diffraction pattern rotate in SingleCrystal (or vice versa).

Live diffraction link between a crystal structure in CrystalMaker and its corresponding diffraction pattern in SingleCrystal.



SingleCrystal has advanced stereographic projection capabilities: display poles and traces for lattice planes or vectors, with the option of showing symmetry-related planes, with extensive customization.

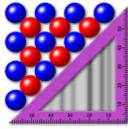
Flexible Licensing and Online Sales

We provide a wide range of licensing options, including Personal, Group and Campus (Site) options. Our *annual site licences* allow unprecedented flexibility, including all products and all platforms for one low, annual fee.

All our software is available for purchase from our award-winning online store,

<http://crystalmaker.com/sales>

Order direct for the best prices, fastest service—and digital delivery: start using your software quickly, with all resources available to download.



Glossary

Anisotropic Displacement Parameter

A description of the thermal vibration or other state of motion, of an atom in a crystal. The uncertainty in the atomic position is represented by a three-dimensional shape, the surface of which defines a fixed probability of the atom being located within. For harmonic motions of the atom, this is an ellipsoid—a “thermal ellipsoid”. This is represented by a symmetric tensor, with six unique components (U_{ij}) giving both the magnitude, and the direction of the atom’s displacement from its ideal position—hence the term “atomic displacement parameters”. This is also referred to as the “anisotropic temperature factor”.

Asymmetric Unit

The smallest group of atoms required to uniquely define a crystal structure.

To define a crystal, one could list all the atoms inside one unit cell. However, if the crystal has some symmetry then some of these atoms will be related to other atoms by the operation of symmetry elements. So, a more concise way of defining a crystal structure is to specify the crystal symmetry (i.e., its spacegroup), and then give the smallest subset of the unit cell (i.e., the asymmetric unit) which, when combined with the symmetry “generates” the remainder of the unit cell.

Atomic Displacement Parameter

See Thermal Ellipsoid.

Atomic Scattering Factor

X-rays are scattered by the electrons of an atom. Because the diameter of electron clouds is similar to the wavelength of x-rays, the amount of scattering varies with angle. The amplitude of x-ray radiation scattered by a single atom is defined by the atomic scattering factor (ASF). Its angular dependence is summarized in a 9-coefficient polynomial equation.

Values for the ASF coefficients may be found in the International Tables for Crystallography, or in: Doyle & Turner (1968) *Acta Crystallographica* A24:390-397.

Bragg Equation

The most important equation in the history of diffraction was derived by W L Bragg in 1912 and relates the angle at which radiation is scattered by a set of lattice planes to the inter-planar separation (d-spacing) and the wavelength of the radiation:

$$\lambda = 2 d \sin \theta$$

where λ is the wavelength, d is the d-spacing of the lattice planes, and θ (the Bragg Angle) is the inclination of the incident radiation beam to the surface of the lattice planes.

Bravais Lattices

The 14 unique three-dimensional lattice types—as derived by the German crystallographer, Bravais, in the mid-nineteenth century. He showed that, when considering all combinations of lattice types and crystal systems, there was a total of 14 unique lattice, which have become known as the “Bravais Lattices”.

Cell Parameters

See Lattice Parameters.

Crystal

A crystal is a solid material which has a regular internal arrangement of atoms, which sets limits on its range of chemical compositions, and gives it a characteristic crystal shape.

The internal arrangement of atoms can be resolved into an endlessly-repeated unit cell, which contains one or more atoms. The relative arrangement of atoms within the unit cell defines the structure’s symmetry, as summarized by the crystallographic spacegroup.

Crystal System

A crystal system relates to the shape and symmetry of the unit cell, and in particular, to the relative lengths and the angles between the cell edges.

For example, the cubic system is defined by a cube-shaped unit cell, where the cell edges a , b and c are mutually perpendicular, and have the same lengths. On the other hand, the triclinic system is characterized by a “distorted” unit cell, in which the cell edge lengths and their interaxial angles may all be different. The 7 unique crystal systems are summarized below:-

Crystal System	Unit Cell Dimensions	Essential Symmetry	Bravais Lattices
Triclinic	$a \neq b \neq c$ $\alpha \neq \beta \neq \gamma$	None	P
Monoclinic	$a \neq b \neq c$ $\alpha = \beta = 90^\circ \neq \gamma$	One 2-fold (diad) axis	P, C
Orthorhombic	$a \neq b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	Three 2-fold (diad) axes	P, C, I, F
Tetragonal	$a = b \neq c$ $\alpha = \beta = \gamma = 90^\circ$	One 4-fold (tetrad) axis	P, I
Cubic	$a = b = c$ $\alpha = \beta = \gamma = 90^\circ$	Three 3-fold (triad) axes	P, I, F
Trigonal (*)	$a = b = c$ $\alpha = \beta = \gamma \neq 90^\circ$	One 3-fold (triad) axis	R
Hexagonal	$a = b \neq c$ $\alpha = \beta = 90^\circ; \gamma = 120^\circ$	One 6-fold (hexad) axis	P

(*) Note that trigonal crystals can be described in terms of an hexagonal unit cell, although their maximum symmetry is 3-fold, not 6-fold.

Debye-Waller Factor

The Debye-Waller factor describes the amount by which scattered intensities are reduced by thermal vibrations of atoms. It has the form:

$$\exp(-B_{\text{iso}} \sin^2 \theta / \lambda^2)$$

where θ is the scattering angle, λ is the wavelength, and B_{iso} is the atom's *temperature factor* (related to the atom's atomic displacement parameter by the relationship: $B_{\text{iso}} = 8 \pi^2 U_{\text{iso}}$).

Delta Function

A delta function is an infinitely-narrow peak, with a finite intensity. For CrystalDiffract, lines are drawn at the positions of each calculated peak, with heights corresponding to the calculated, integrated intensity.

The delta function may be useful because it:

- clearly indicates peak centres for a complex structure;
- allows fast plot scrolling, even on slow machines.

Diffraction

Diffraction is the process by which waves interact with objects and are disturbed, or bent, from their previous course (e.g., the movement of water ripples around

the end of a pier).

In X-ray crystallography, the diffraction of certain electromagnetic waves (X-rays) from atoms in a crystal is studied.

In neutron diffraction, a high-energy beam of neutrons (nuclear particles with no charge) has wave-like properties and can also be diffracted from atoms in a crystal lattice.

D-Spacing

The term “d-spacing” refers to the inter-planar separation for a set of lattice planes: that is, the distance between successive planes, measured along the plane normal (at 90° to each lattice plane).

Planes of atoms in a crystal act as miniature diffraction gratings, and their d-spacings control the angles at which radiation is scattered, as summarized in the famous Bragg Equation of 1912.

Energy-Dispersive

A relatively-new type of diffraction involves using “white” radiation that has a spread of wavelengths.

Whereas a fixed-wavelength experiment requires one to mechanically scan a detector through a range of $\theta/2\theta$ angles (see the Bragg Equation), using “white” radiation allows the use of a stationary detector, with diffraction from a range of d-spacings being recorded at the same θ angle. The different scattered beams have different wavelengths, and hence energies - hence this form of diffraction is referred to as “energy-dispersive”.

CrystalDiffract allows you to specify the 2θ value for the diffraction experiment, using the **Diffract > Energy** command. You can also adjust this interactively, using the Parameter list.

Eta

The Eta (η) parameter describes the shape of a Pseudo-Voigt peak profile. It varies between 0 (a pure Gaussian peak) and 1 (a pure Lorentzian peak).

Form Factor

See Atomic Scattering Factor.

Fractional Coordinates

For crystal structures it is conventional to define atomic coordinates relative to the unit cell dimensions; these relative coordinates are expressed as fractions of the unit cell dimensions along the crystallographic x, y, and z axes.

For example, if an atom in a cubic crystal has orthogonal coordinates $\{x = 2 \text{ \AA}, y = 4 \text{ \AA}, z = 6 \text{ \AA}\}$ and the unit cell parameter $a (= b = c)$ is 10 \AA , then its fractional coordinates are $\{0.2, 0.4, 0.6\}$.

Gaussian

The Gaussian function is a mathematical function, characterized by a bell-shaped peak (“bell curve”), without the wide tail of a Lorentzian function.

General Equivalent Position

Crystallographic symmetry includes lattice translations (defined by the lattice type), plus additional symmetry operations associated with each lattice point. These symmetry operations, which may include mirror or rotational symmetry, can be summarized by listing the relative coordinates to which a single atom is mapped.

For example, consider a crystal whose unit cell contains just a single symmetry element: a mirror plane parallel to the x and z axes, and passing through the y axis at the origin. The mirror plane will reflect (or map) a point at $\{+x, +y, +z\}$ to the position at $\{+x, -y, +z\}$. In other words, the positions $\{+x, +y, +z\}$ and $\{+x, -y, +z\}$ are symmetrically-equivalent - and are referred to as “general equivalent positions” for this crystal.

Graphs and Films

CrystalDiffract can display diffraction patterns in either “graph” mode, or in “film” mode.

CrystalDiffract graphs represent how the intensity of radiation diffracted by a crystal varies according to scattering angle (2θ), time-of-flight, or some other parameter, plotted along the graph’s horizontal (x) axis. The intensity is plotted along the graph’s vertical (y) axis. The “taller” the peaks, the more intense the diffraction.

In film mode, CrystalDiffract simulates the appearance of a photographic film (negative) which has been wrapped around a crystal exposed to radiation. Reflexions appear as dark lines on the grey film background. The darker the line, the more intense the reflexion.

Indexing Peaks

This is the process of assigning Miller Indices to diffraction peaks, to signify which sets of planes caused which reflection.

Intensity

Crystals are regular, ordered structures, and contain billions of atoms arranged in regularly-spaced lattice planes. These planes act as diffraction gratings for

radiation whose wavelength is similar to the lattice plane spacing (e.g., x-rays and neutrons can be diffracted by crystals).

The strength of diffraction from a particular set of lattice planes is termed the intensity of reflection. This depends on the type and arrangement of atoms parallel to the lattice planes; atoms lying between the lattice planes are “out of phase” and reduce the intensity of the scattering.

Knowing the orientation of a lattice plane (which is determined by its Miller Indices), and details of the crystal structure (its lattice parameters, symmetry, and asymmetric unit), CrystalDiffract can calculate the intensity for any reflexion, based on a fixed volume. (Note that in a real experiment, the *absolute* intensity will depend on a multitude of factors, including the radiation intensity, sample volume, detector efficiency, etc.)

Isotropic Displacement Parameter

A representation of the thermal (or other) motion of an atom, assuming equal motions in all directions. The radius of the sphere is the atom’s mean-square displacement, $\langle u^2 \rangle$, written U_{iso} (in units of \AA^2).

N.B., the isotropic *displacement* factor, U_{iso} , is related to the atom’s isotropic *temperature* factor, B_{iso} , by a factor of $8\pi^2$, i.e.,

$$B_{\text{iso}} = 8 \pi^2 U_{\text{iso}} .$$

Lattice

A lattice is defined as a regular arrangement of points throughout space. This is a mathematical concept, used to describe the regular repetition, throughout 3-d space of individual atoms, or groups of atoms (i.e., the asymmetric unit).

The basic building brick of a crystal, the unit cell, has lattice points at each of its eight corners. It may also have lattice points at the centres of certain faces, or at the centre of the unit cell. This arrangement of lattice points within the unit cell defines the lattice type of a crystal: *P* (Primitive); *I* (Body-centred, from the German Innenzentrierte; “inner-centred”); *A*-, *B*- and *C*-face centred; *F* (all-face-centred) and *R* (Rhombohedral).

Lattice Parameters

Six numbers which uniquely define the size and shape of the unit cell. These are the lengths of the unit cell edges, a , b , c ; and the inter-axial angles α , β , and γ .

Lattice Plane

A lattice plane is a plane of lattice points in a three-dimensional structure. The orientation of a lattice plane can be given in terms of its intercepts along the unit cell axes xyz . These are summarized using Miller Indices, (hkl).

Lattice Vector

A lattice vector is defined as a vector between two lattice points. The orientation of the vector can be expressed in terms of its coefficients U , V , and W , parallel to the unit cell vectors \mathbf{a} , \mathbf{b} , and \mathbf{c} , respectively, giving the vector equation:

$$U\mathbf{a} + V\mathbf{b} + W\mathbf{c}$$

which is generally written in a shorthand form as:

$$[UVW]$$

Note that the unit cell vectors are not necessarily mutually orthogonal, neither may they be of equal length. So, for example, in an orthorhombic crystal ($a \neq b \neq c$), the lattice vector $[100]$ probably has a different length from the vector $[010]$.

Lorentzian

The Lorentzian function is a mathematical function, characterized by trumpet-like peaks, with very wide tails. The Lorentzian function generally provides a good description of the shape of neutron diffraction peaks.

Lorentz Factor

A geometric factor that affects the intensity of a reflexion. This takes into account various factors, including (for a rotating sample) the time taken for a reflexion to be in a scattering position.

Miller Indices

Miller Indices are sets of three integers, designated h , k , l , which uniquely define the orientation of a lattice plane, relative to a crystal's unit cell.

A lattice plane's orientation can be defined in terms of its intersections with the three crystallographic axes, x , y , and z . If a unit cell has edge lengths a , b , and c , and the first lattice plane from the origin cuts these axes at a/h , b/k , and c/l , then the Miller Indices of that plane are: $(h \ k \ l)$.

Monochromatic

Monochromatic radiation is radiation characterized by a single x-ray or neutron wavelength (e.g., 1.54 Å). Many synchrotron x-ray sources and neutron diffractometers produce monochromatic radiation.

In contrast, traditional laboratory x-ray tubes usually produce dual-wavelength radiation. The two wavelengths correspond to $K\alpha_1$ and $K\alpha_2$ x-ray emission peaks from the target material.

Multiplicity

“Multiplicity” refers to the number of X-ray or neutron reflexions that contribute to a single peak observed in a powder pattern. Multiple reflexions with the same *d*-spacing become superimposed in a one-dimensional x-ray powder pattern, giving rise to a single peak, with higher intensity. The total number of reflexions contributing to that peak is the peak’s “multiplicity”.

Neutron Scattering Length

Neutrons are scattered by atomic nuclei, which are much smaller than neutron wavelengths—so the scattering is essentially independent of angle. The amplitude scattered by an atomic nucleus is represented by the neutron scattering length.

Percent Strain

A measurement of the amount of strain in a powdered crystalline sample. The percent strain is the standard deviation for the variation of cell parameters in the sample (in an ideal crystal there would be one unique cell parameter, whereas in a strained crystal there might be a normal distribution of cell parameter values, characterized by a standard deviation, ranging from zero for the ideal crystal to a few percent for a very-highly strained crystal).

Polarization Factor

A factor that takes into account the reduction in intensity caused by the polarization of (X-ray) radiation when scattered by electrons.

Preferred Orientation

A non-random distribution of crystallites in the sample (e.g., the tendency of platy crystals to lie flat parallel to the specimen holder, or for needles to clump together parallel to their axes). Preferred orientation causes some reflexions to be scattered more strongly, whilst other reflexions will appear weaker—if at all.

Primitive Lattice

The most basic type of lattice, where the unit cell has lattice points only at its corners. Since each corner is shared between eight unit cells, this gives a total of $8 \times \frac{1}{8} = 1$ lattice point per unit cell.

Pseudo-Voigt

The Pseudo-Voigt function is a mathematical function used to describe the shape of diffraction peaks. It is defined as the weighted sum of a Lorentzian and a Gaussian peak. The Lorentzian fraction is defined by the Eta (η) parameter.

Reflexion

A peak of intensity, caused by the scattering of a beam of radiation, by planes of atoms in a crystal.

Rhombohedral

This is a special kind of Primitive lattice, where the body diagonal has three-fold symmetry and the inter-axial angles are all non-orthogonal.

Site Occupancy

The term “site occupancy” refers to the chemical content of a crystallographic site. In an ideal structure, a site in the crystal might contain one specific chemical element per site, e.g., a copper (Cu) atom on site 1 and a zinc (Zn) atom on site 2. Sometimes, however, crystals show disordered structures and there might be a statistical probability of different atoms occupying the same sites. For example 50% Cu and 50% Zn on sites 1 and 2. The site occupancy for both sites would then be written $\text{Cu}_{0.50}\text{Zn}_{0.50}$.

Space Group

A space group is a three-dimensional arrangement of symmetry elements, positioned within a crystal's unit cell, which relates those atoms in the asymmetric unit (“motif”) to the rest of the atoms in the unit cell.

A space group is represented by a spacegroup symbol: a descriptive tag which may or may not give further information about the actual arrangement of symmetry elements. Crystallographers commonly use the “International Notation” of spacegroup symbols, examples of which include “*P* 1”, “*P* 2/*c*” and “*I* a $\bar{3}$ *d*”. The first character of the symbol (e.g., “*P*” or “*I*”) is the lattice type; the remaining characters relate to symmetry operators (“2” is a 2-fold rotation axis; “*c*” is a *c*-glide plane perpendicular to the 2-fold axis; etc.)

Structure Factor

A Structure Factor, $F(hkl)$ describes the amplitude and phase of radiation scattered by one unit cell of material for the set of lattice planes (*hkl*). The Structure Factor is a complex number, with real (*A*) and imaginary (*B*) parts:-

$$F(hkl) = A(hkl) + i B(hkl)$$

The phase angle, ϕ , is given by the ratio of the imaginary to real parts:-

$$\tan \phi = B / A$$

The Intensity of the scattered radiation for the reflection (*hkl*) is proportional to the square of the amplitude, $|F|^2$.

Systematic Absence

Reflexions which are forbidden because of lattice-type or spacegroup symmetry are known as systematic absences. Not all zero-intensity reflexions are systematic absences; however, all systematic absences have zero intensity.

We divide systematic absences into two main categories:

1. Lattice Absences. These are caused by the lattice type, and can be predicted using the rules below.

Lattice Type	Absent Indices	Example Absences
P	None	None
A	(k + l) odd	001, 010, 101
B	(h + l) odd	100, 001, 110
C	(h + k) odd	100, 010, 011
I	(h + k + l) odd	100, 111, 201
F	h, k, l mixed even & odd	100, 110, 21

2. Spacegroup Absences. These are (non-lattice) absences, caused by internal spacegroup symmetry such as glide planes or screw axes.

A third category, Other Absences, includes reflexions which are absent for reasons other than the predicted lattice type or spacegroup symmetry. These may be caused by:

- Atoms on “special positions” within the lattice.
- Genuine zero intensity resulting from the structure—so-called “accidental absences”.
- “Missing symmetry”: the true symmetry of the structure is higher than that claimed, resulting in additional systematic absences.

Thermal Ellipsoid

Thermal ellipsoids (also referred to as “atomic displacement parameters”, or “temperature factors”) are used to describe the relative magnitude, and orientation, of atomic motions (or positional disorder) in a crystal.

In most structures the atoms are not at rest, but vibrate about their mean positions. A statistical analysis of these motions, assuming harmonic vibrations, confines an atom centre to a probability surface whose three-dimensional representation is, in its most general case, an ellipsoid (described by anisotropic displacement parameters), sometimes approximated by a sphere (described by an isotropic displacement parameter).

Time-of-Flight

A type of neutron diffraction experiment in which scattered intensities are recorded as a function of the time taken for neutrons to reach a fixed detector.

A neutron spallation source (such as ISIS at the Rutherford-Appleton Laboratory, near Oxford, England) creates pulses of neutrons with a range of energies. These travel at different speeds, depending on the energy of the neutrons, and are directed down a long “beam line” towards a powder sample.

Diffraction is recorded by neutron detectors arranged around the sample, at a fixed 2θ angle. The number of pulses is recorded as a function of the time-of-flight of the neutrons (which is typically in the range of a few milliseconds to several hundred milliseconds).

As for energy-dispersive diffraction, an extended diffraction pattern can be recorded at a fixed angle because the sample is subjected to neutrons of different energies, and hence wavelengths.

Unit Cell

The basic building brick of a crystal structure.

The unit cell is a parallelepiped with edges parallel to the three crystallographic axes, x , y , and z . The edges along the x , y , and z directions define the crystallographic basis vectors, a , b , and c , respectively. The inter-axial angles are, by convention, called α ($= y^{\wedge}z$), β ($= x^{\wedge}z$) and γ ($= x^{\wedge}y$).

The axial dimensions and inter-axial angles uniquely define the three-dimensional periodic nature of the crystal's lattice, and are known as the lattice parameters.

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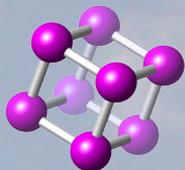
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