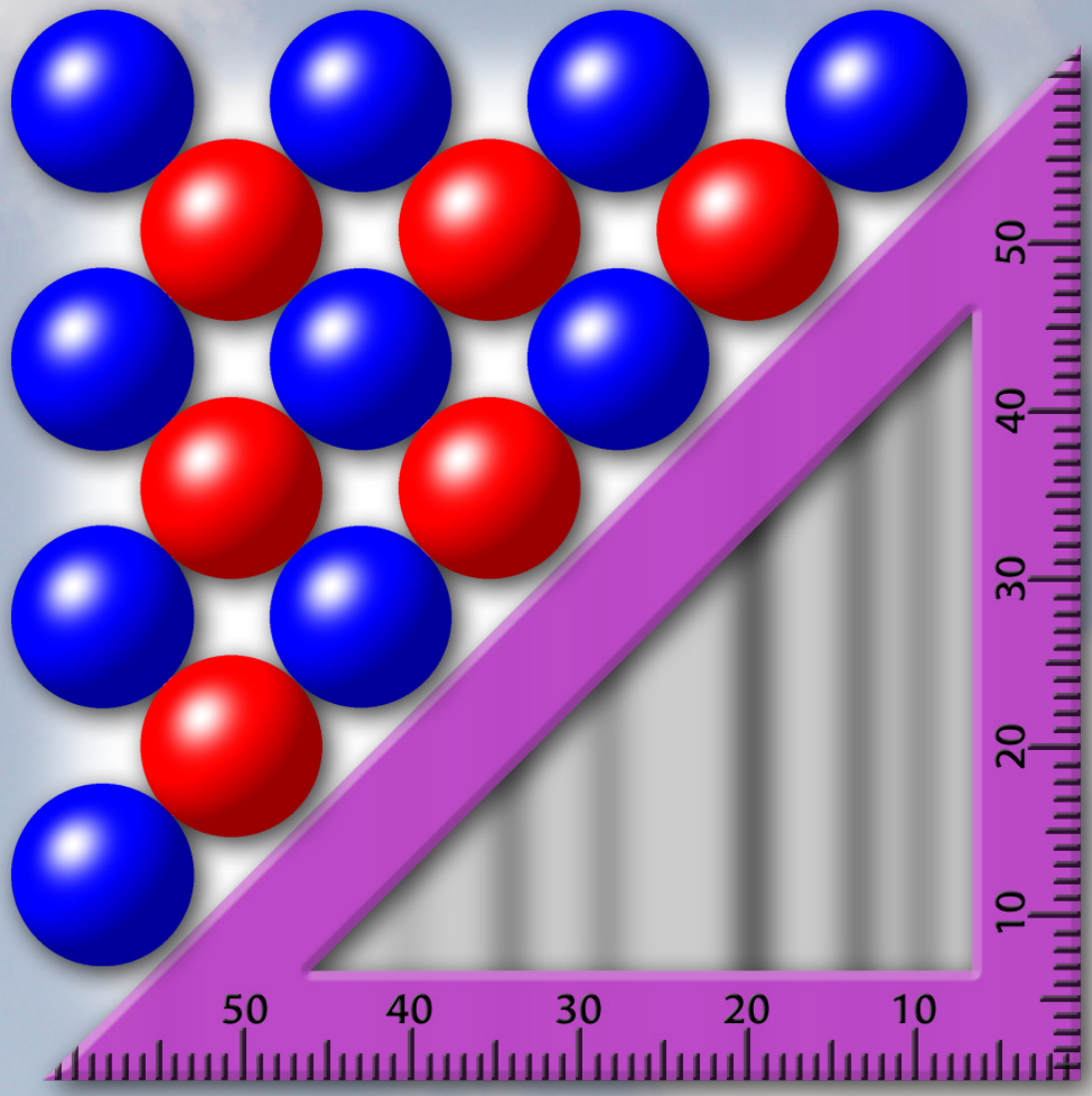
CrystalDiffract®

Interactive Powder Diffraction Software



Tutorial

Version 6 for Mac & Windows

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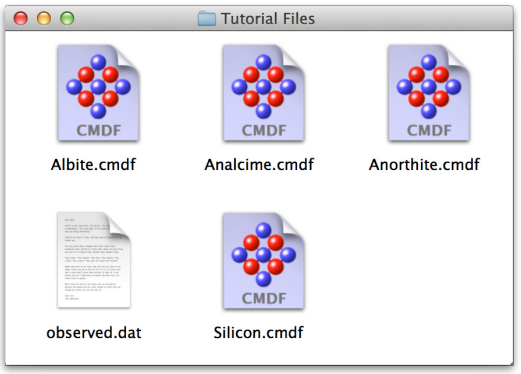
CrystalDiffract

Tutorial

Welcome! This tutorial is designed to quickly familiarise you with the key features of CrystalDiffract 6 in just 30 minutes. Please follow the numbered steps to build your understanding. Use the *Tutorial Files* folder included with the program: this contains four crystal structure files plus a text file with observed data values.

The Tutorial Files are included with the application package.



Full-Feature Version. This tour is designed for the full-feature (paid) version of CrystalDiffract. Some features (such as the ability to save files) are disabled in the free "Demonstration" version.

The Mystery of the Volcanic Crystals

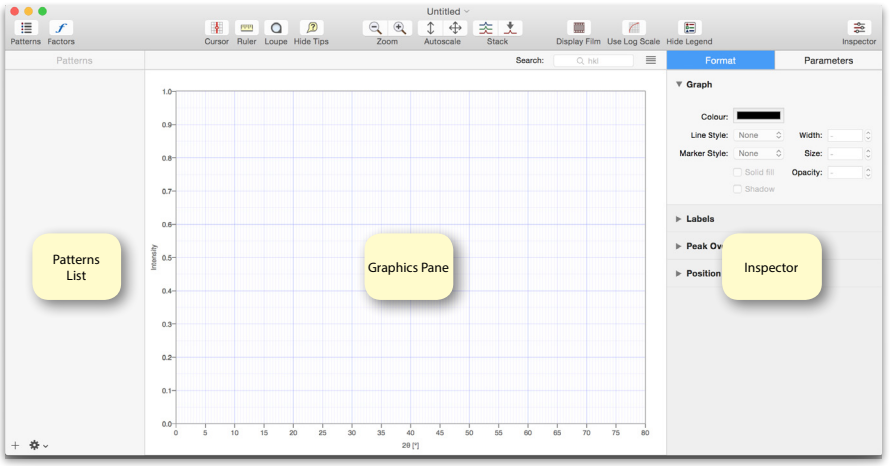
Geologists are investigating a chain of ancient volcanoes between Scotland and Northern Ireland, in the United Kingdom. Large white crystals have been found in a fine-grained rock, thought to be the remains of a lava flow. They may provide clues about the evolution of the Earth's crust early in its history. But first, the crystals need to be identified. X-ray diffraction was carried out, and your task will be to help the geologists identify these crystals.

The Naming of Parts

We need to familiarise ourselves with the CrystalDiffract user interface: our virtual X-ray laboratory.

1 Launch CrystalDiffract. A new window should appear on screen. If a window doesn't appear, choose: **File > New Window** to create one.

You work from left-to-right, loading diffraction patterns in the **Patterns List**, reviewing their profiles in the **Graphics Pane**, and adjusting their Format and Parameters in the **Inspector** on the right.



An empty CrystalDiffract window, showing the three main panes.

Loading Patterns

Our geologists have provided an observed diffraction pattern for us to work with: a simple *xy* text file, `Observed.dat`. This was measured using X-ray diffraction, using a Guinier *film* camera. Silicon was added as an internal standard.

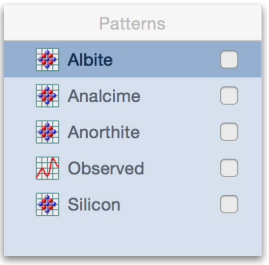
The geologists have some hunches about the white crystals, based on the local mineralogy. They have whittled their search down to three possibilities: *Albite* ($NaAlSi_3O_8$), *Anorthite* ($CaAl_2Si_2O_8$) or *Analcime* ($NaAlSi_2O_6.H_2O$). These are all aluminosilicate minerals, the first two of which are different species of *feldspar:* the most common mineral in the Earth's crust. The third, *Analcime*, is quite rare (but is related to *zeolite* structures, popular with inorganic chemists) and is associated with *watery conditions* in volcanic melts. Our geologists are getting quite excited!

2 Switch to your file system, and locate the *Tutorial Files* folder (this should be inside the *Examples* folder that came with the program). Drag the *Tutorial Files* folder into the Patterns List.

> **Tip:** If your Patterns List isn't visible, click the **Patterns** button in the toolbar, or choose: **View > Layout > Show Patterns List**.

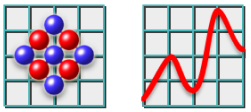
Your Patterns List should now look like this:

The Patterns List, showing loaded simulated patterns (crystal icons) and an observed pattern.



Where did these items come from? Each list entry represents an individual diffraction pattern. Notice that all-but-one of the patterns has a "crystal" icon. These represent *simulated* diffraction patterns: CrystalDiffract generates ideal patterns, using the crystal structures provided in the source files (our geologists thoughtfully provided *CrystalMaker*® crystal structure files for those materials). The Observed pattern has a different icon, to emphasize that this is a *real* pattern.

Icon for simulated (;eft) and observed (right) pattern.



We will later compare the various simulated patterns with our Observed pattern, in order to characterize it. Before we do that, we'll learn a bit more about using CrystalDiffract.

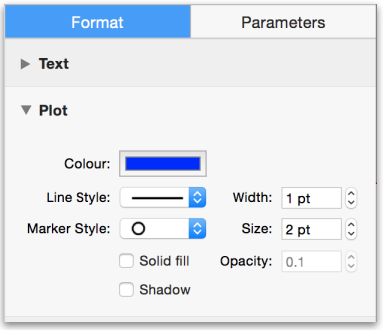
3 Locate the **Inspector**, on the right-hand side of the window. Ensure that the **Format** tab is selected and that the **Plot** group is open (if not, click the **Plot** button to reveal its controls).

(If the Inspector isn't visible, click the **Inspector** button in the window toolbar, or choose: **View > Layout > Show Inspector**.)

4 Click the **Colour** button and choose a new colour for the Observed data. We'll use a blue colour here.

5 Use the **Marker Style** popup to specify a circle for each data point:

Changing the plot colour and line style.



Manipulating the Pattern with Multi-Touch

We're now ready to explore our Observed diffraction pattern. We'll experiment with scrolling and zooming, using **multi-touch.**

> **Multi Touch.** Multi-touch is available on all portable Macs, and desktop computers with a Magic Trackpad. In addition, trackpads are now widely available on laptop PCs and can be added to desktop PCs. If your computer does not have a trackpad, or does not support multi-touch, you will need to skip this section.

6 **Pinch-to-zoom.** Use a standard "pinch" gesture to contract or an "expand" gesture to expand the horizontal plot range.

(Note that 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" gesture.

7 **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 over the trackpad.

"Slide-to-scroll" gesture.

8 **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 over the trackpad.

"Slide-to-scale" gesture.

Manipulating the Pattern with the Mouse

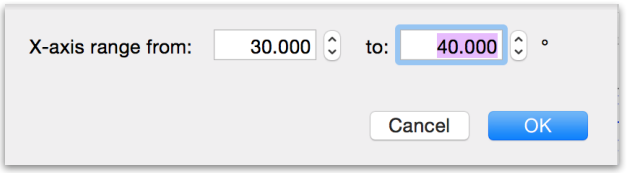
If you don't have multi-touch, you can still manipulate your diffraction pattern using the mouse or keyboard:

9 **Drag-to-scroll**. Click in the Graphics pane with your mouse, and hold the mouse button down so that the "hand" cursor appears. Now drag to the left or the right to scroll your pattern.

10 **Mouse-wheel scaling**. Position the mouse pointer over the Graphics pane, then use the mouse wheel to adjust the vertical scale.

11 **Set Plot Range**. Choose: **View > Plot Range** to display the Plot Range sheet (Mac) or dialog (Windows). Enter the range limits 30–40° and click the **OK** button to continue.

Using the Plot Range command.



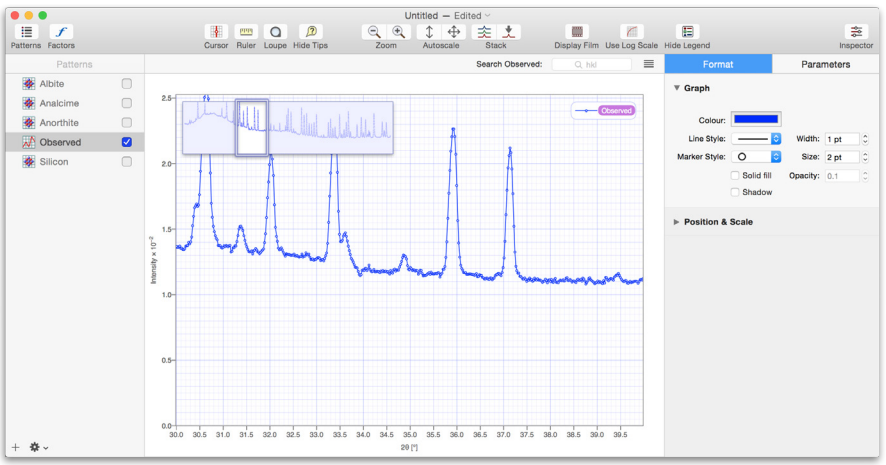
Tip: The toolbar can be customized to add additional scroll buttons, should you require them.

Scrolling with the Scroller

Most graphics programs use scrollbars to let one scroll an over-size image within a fixed frame. CrystalDiffract has something better: a "Scroller" panel which shows the full diffraction pattern.

12 **Showing the Scroller**. Using the mouse, click-and-drag in the Graphics pane. The Scroller appears in the top left-hand corner.

The Scroller in action.



13 **Using the Scroller**. Click and drag the Scroller thumb to reposition the diffraction pattern. (You can also click on either side of the thumb to continuously scroll the pattern.)

A few seconds after you have released the mouse button, the Scroller will vanish, thereby avoiding the screen "clutter" of traditional scrollbars.

14 Click the **Autoscale** button () in the toolbar to reset the plot range.

Subtracting the Background

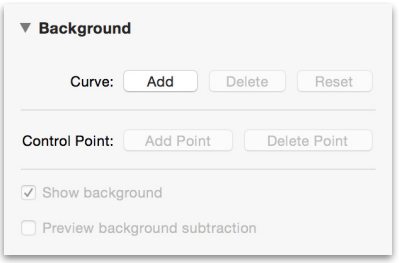
Notice the prominent and uneven background for the Observed pattern? This is caused by the sample being mounted on *Mylar* film and measured in transmission mode. We wish to remove the background, which will then make it easier to compare the pattern's peaks with simulated data, for characterization purposes.

15 Ensure that the observed pattern is shown selected in the Patterns List. (If it isn't selected, simply click its title to select it.)

16 Ensure that the Inspector is visible, then click on the **Parameters** tab to show the Parameters Inspector.

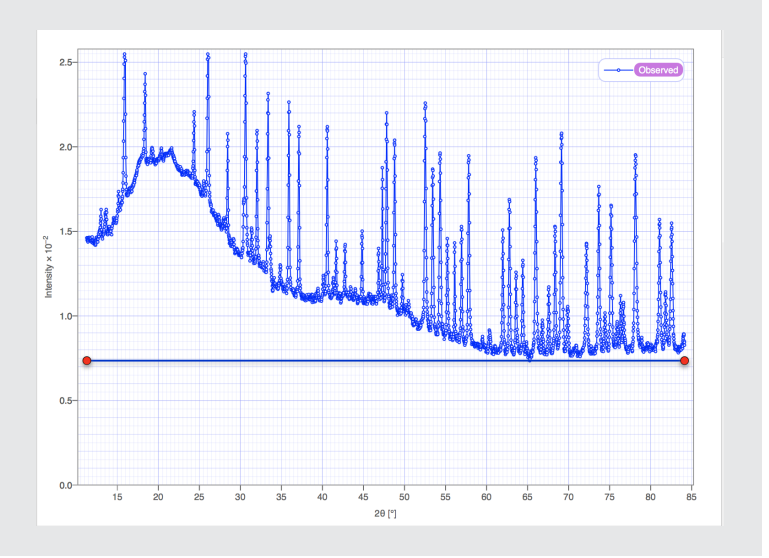
17 Click on the **Background** group button to reveal its controls:

Background controls.



18 Starting at the top of the group ("Curve:"), click: **Add** to add a new background function to your pattern. A dark line appears, with two red control points:

The background curve is represented by the thick dark line, with two red control points.

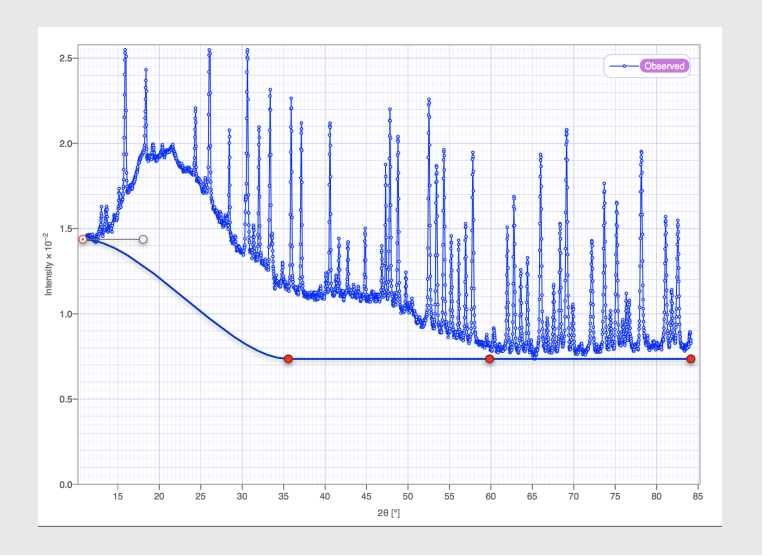


Given the uneven nature of our present pattern, we'll want to add more control points:

19 Click the ("Control Point:") **Add Point** button twice, to add two more control points to your background function. Now the fun starts!

20 Click-and-drag the left control point up and to the left:

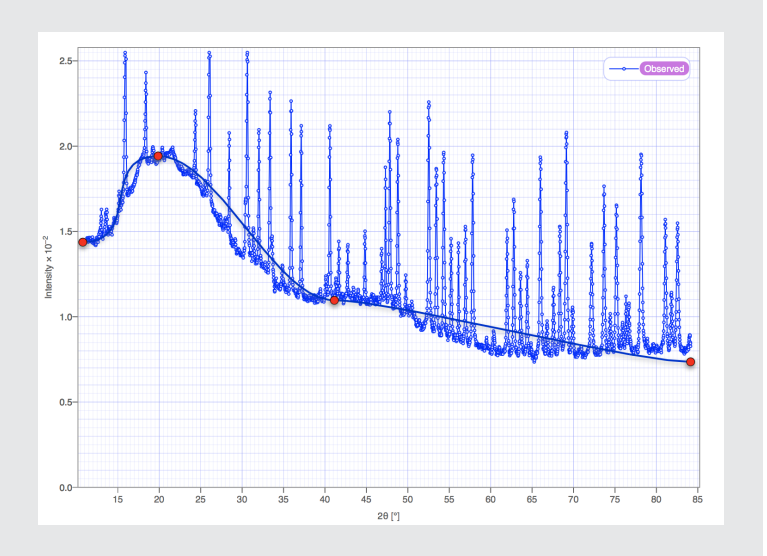
Moving the first control point up to the observed pattern.



(Ignore the tangent control handles: we'll deal with those later.)

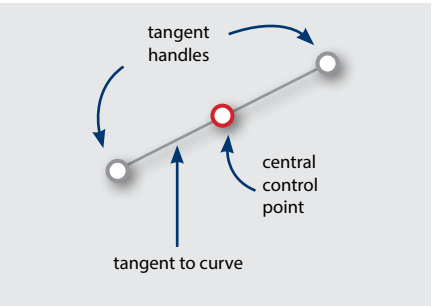
21 Move the other three control points up to the observed pattern:

All control points are now on the observed pattern.



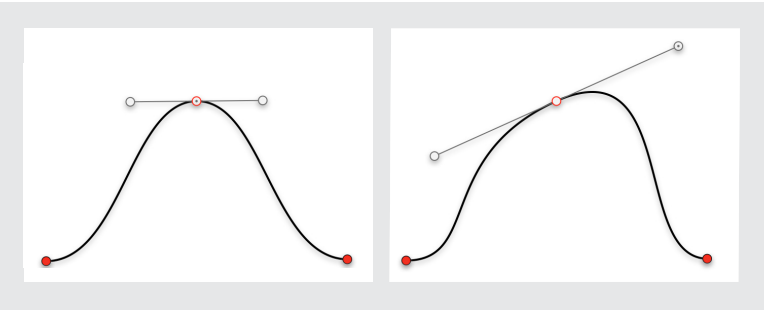
We shall now use the control handles to adjust the curvature of our background function around each control point.

Anatomy of a control point.



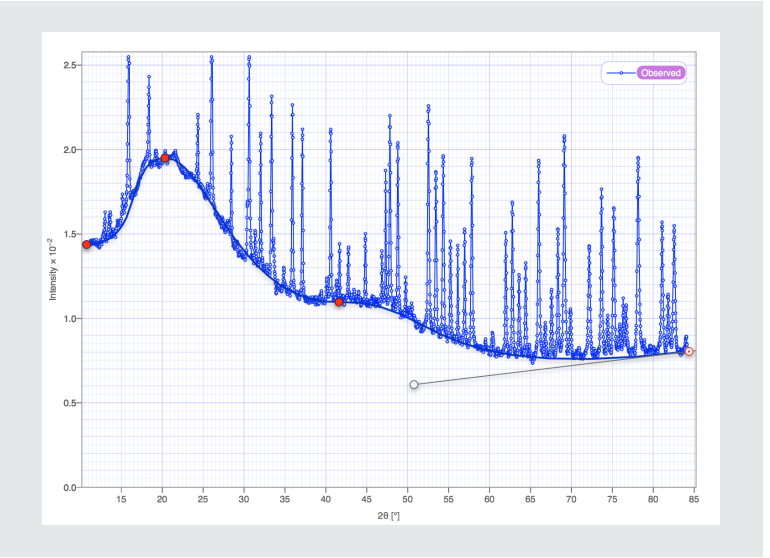
22 Click a control point to select it and reveal the grey tangent handles. Click-and-drag a tangent handle to change the radius-of-curvature:

Using control handles to adjust curvature.



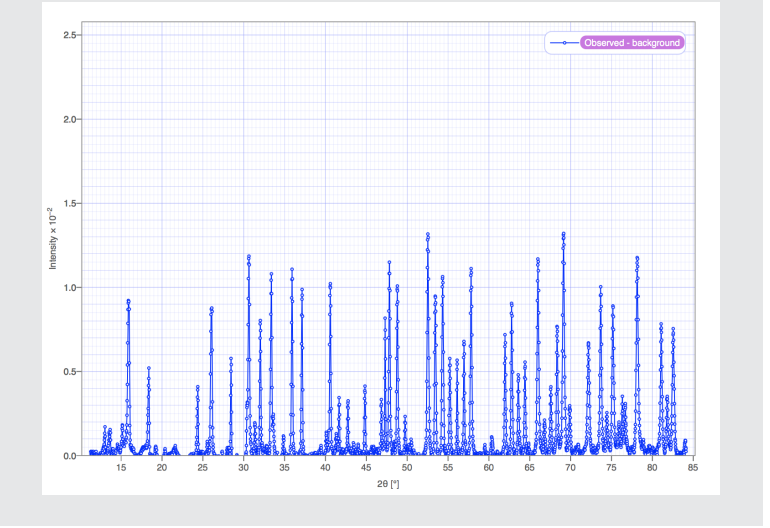
23 Adjust the remaining control points to give a smooth flowing curve:

The final background function.



24 Uncheck the **Show Background** checkbox in the **Background** group. Your data will be replotted, minus the background:

Observed data minus the background function.



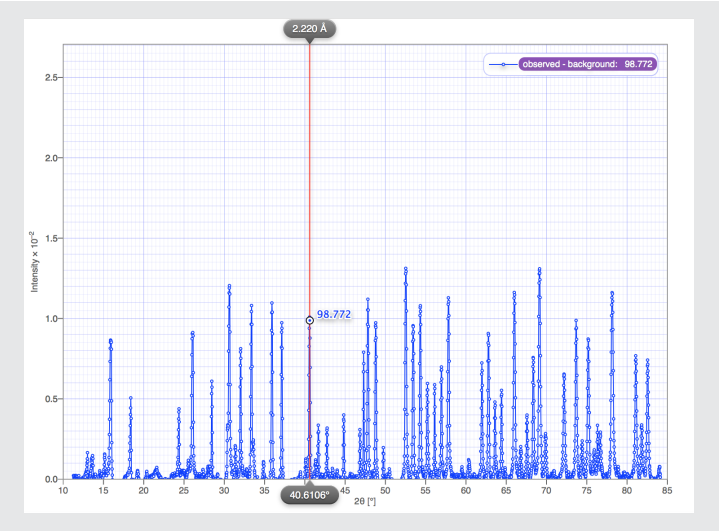
We are now in a good position to start measuring our observed (corrected)data, and then comparing it with the ideal, simulated diffraction patterns.

Measuring Points

CrystalDiffract includes a Cursor tool which you can use to measure horizontal positions (*x*-values) and the corresponding intensity values (*y*-values) for any displayed patterns.

25 Click in the Graphics Pane and release the mouse. A vertical red cursor appears:

Graph with vertical cursor.



Notice that the cursor is labelled: the *d*-spacing is displayed in a box above the graph; the corresponding *x*-axis value (in our case, this in degrees 2θ) is shown in a box below the graph. The *y*-axis value at the cursor is shown in two places: a labelled point on the graph, and repeated in the graph's legend—in the top right-hand corner.

26 Position your mouse pointer over the cursor, so that it changes to a resize icon. Click-and-drag the cursor with the mouse, and notice how the cursor labelling is updated.

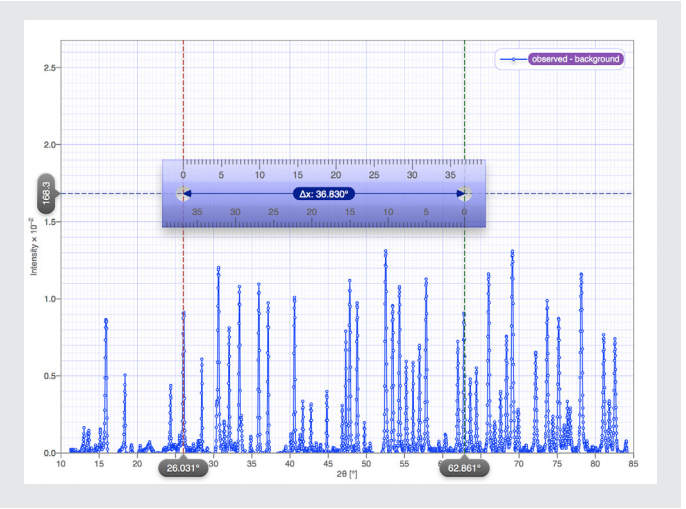
27 Hide the cursor by clicking in the Graphics pane, away from the cursor. (You can also toggle the cursor by clicking the **Cursor** button in the toolbar.)

Measuring Distances

You can use CrystalDiffract's Ruler tool to measure the separation of peaks.

28 Click the **Ruler** button ( ) in the toolbar. The Ruler tool appears:

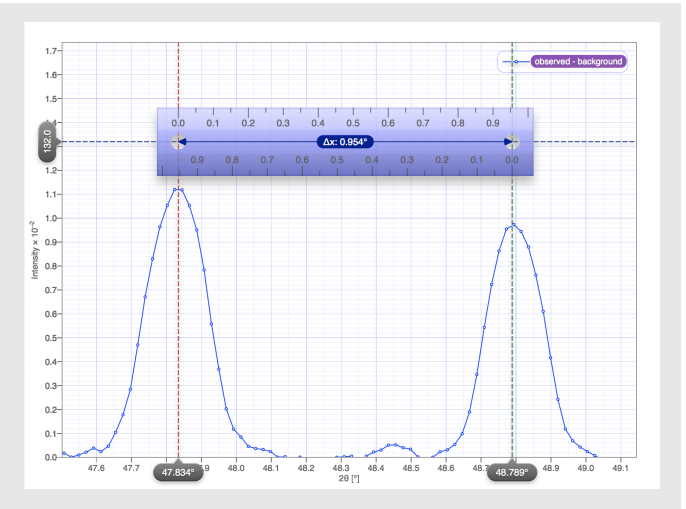
Graph with Ruler tool.



29 Click and drag the ruler with the mouse, to reposition it. Notice that the *y*-position is shown, and two vertical cursors are displayed (and labelled).

30 Position the mouse pointer over the right-hand (green) ruler cursor. The mouse pointer changes to a resize icon. Click and drag to move the ruler cursor, and hence resize the ruler itself. You can repeat this for the left-hand ruler cursor, e.g., to measure the distance between two peaks:

Measuring the horizontal distance between two peaks.



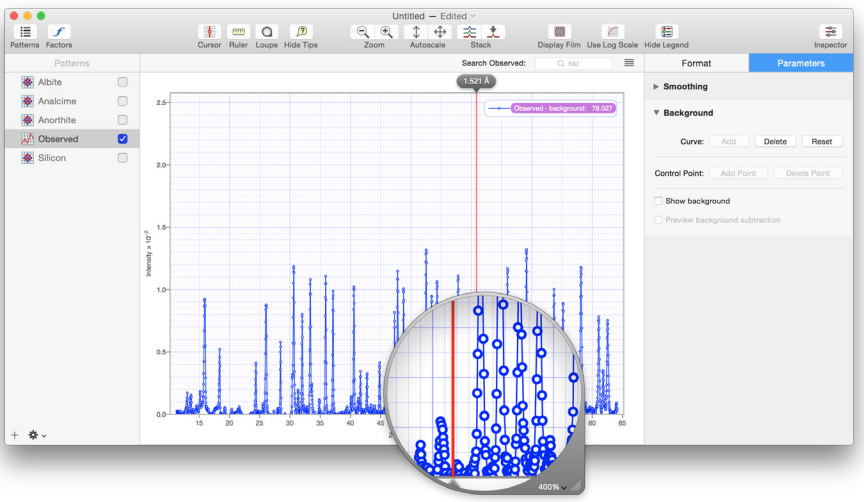
31 Click the **Ruler** button in the window toolbar to hide the ruler.

Seeing Fine Detail with the Loupe

The Loupe tool lets you resolve fine detail, without having to keep zooming in and out.

32 Click the **Autoscale X and Y** button ( )in the window toolbar to automatically scale the Observed pattern.

33 Click the **Loupe** button ( ) in the toolbar. The Loupe tool appears over the Graphics Pane, showing a high-resolution view:



Using the Loupe to magnify part of the Observed diffraction pattern.

34 Click-and-drag the loupe to reposition it.

35 Resize the Loupe: move the mouse pointer over the loupe, then use a multi-touch "pinch" gesture. (If you don't have multi-touch, you can resize the Loupe by clicking-and-dragging its bottom right-hand corner.)

36 Change the Loupe magnification: move the mouse pointer over the loupe, then use a multi-touch two-finger vertical slide gesture (slide two fingers up or down your trackpad). (If you don't have multi-touch, you can change the Loupe magnification by right-clicking on the Loupe and choosing a new magnifcation from the contextual menu.)

37 Hide the Loupe by clicking the **Loupe** button in the toolbar.

Arranging Patterns

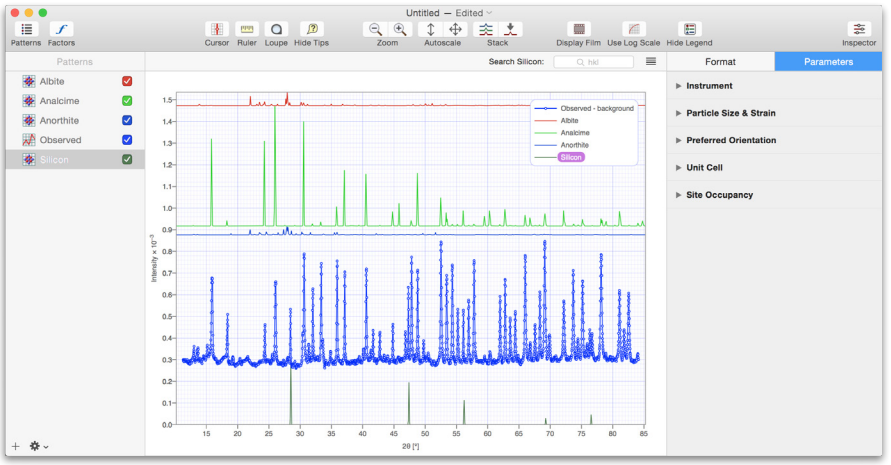
Now that you know how to work with individual patterns, it's time to look at displaying multiple patterns in the same window.

38 Locate the Patterns list, on the left-hand side of the diffraction window. (If the Patterns List isn't visible, choose: **View > Layout > Show Patterns List**.)

39 Turn on the checkboxes for the remaining (unplotted) patterns: **Albite**, **Analcime**, **Anorthite** and **Silicon**. Simulated diffraction patterns for these phases are displayed as coloured profiles in the Graphics pane.

40 Choose **View > Stacked**. The diffraction patterns are now neatly stacked.

Stacked diffraction graphs.

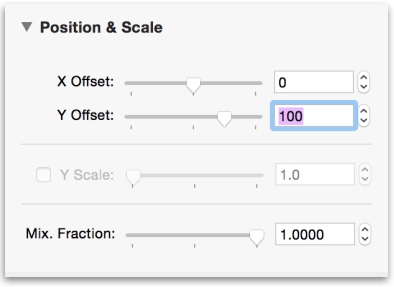


41 Select the **Silicon** diffraction pattern by clicking on its entry in the Patterns List. The Legend is updated, to indicate the pattern selection.

42 Click the Format tab at the top of the Inspector to switch to the Format Inspector.

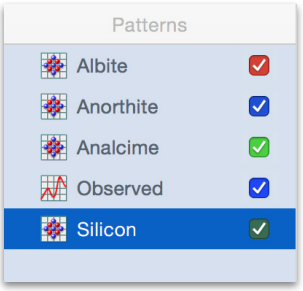
43 Open the **Position & Scale** group in the Format List and locate the **Y Offset** slider and text control. This is currently set to zero, meaning that the selected pattern (Silicon) is plotted as normal, without any offsets. Type 100 into the text field, and press **Return** or **Enter** to replot the pattern.

Changing the y-offset for silicon.



44 We'll now bring the selected (Silicon) pattern to the front, so that it is plotted over the other patterns. To do this, choose: **Pattern > Bring to Front**. Notice how the ordering of the diffraction patterns has changed. This is also reflected in the Patterns List.

The Patterns List, showing the rearranged order of patterns



45 Choose: **View > Collapsed** to "unstack" the plots.

Comparing Patterns

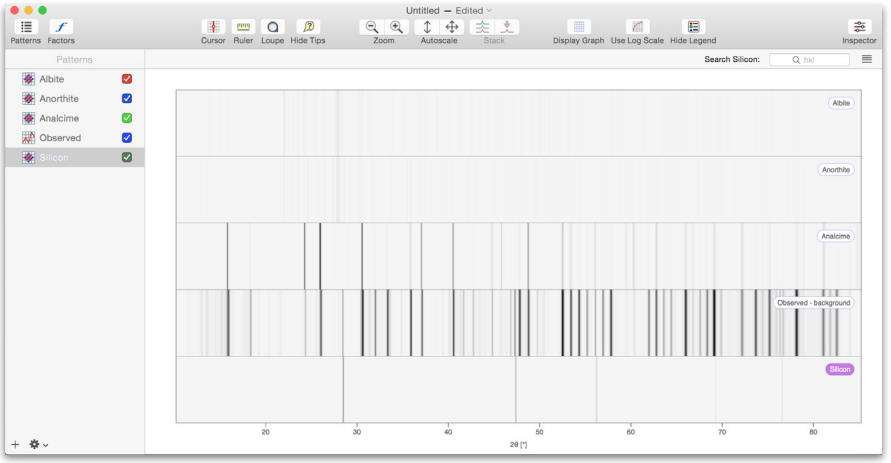
Sometimes the easiest way to compare different patterns is to show them as strips of film: this makes it easier to see which peaks line up, without the complication of different peak heights. (Of course, the *intensities* of the peaks do matter, but we can gauge these from the relative *darkening* of the lines on the film.)

46 Click the toolbar's **Display Film** button ( ). The diffraction patterns are replotted as films, stacked according to the current plotting order (with the "frontmost" film plotted first, at the top of the window).

47 Ensure that the film type resembles a greyscale "negative". If necessary, change the film style by choosing: **View > Film Type > Negative**.

48 Rearrange the position of the patterns: In the Patterns List, select **Analcime** and drag it to just above **Observed**. Similarly, drag **Silicon** to just below the **Observed** pattern.

You should now be in a good position to evaluate which phase(s) are represented in our observed pattern:



Comparing diffraction "films". For clarity, the Inspector has been hidden.

Have you guessed? You may recall that our observed pattern contains Silicon as an internal standard; the "lines" match up nicely. The remaining lines match up quite nicely with… Analcime!

There are some subtle differences in intensity. These are due to the use of photographic *film* to record the observed diffraction pattern, and the fact that the film was slightly overexposed (the more-intense peaks have saturated the film).

The diffraction peaks for Albite and Anorthite don't match up with the Observed pattern—and in particular, the low-angle peaks at around 22° for Albite and Anorthite aren't present in our Observed pattern.

49 In the Patterns List, uncheck the check boxes for **Albite** and **Anorthite**. The Graphics Pane now shows two simulated patterns (Analcime and Silicon) plus our observed pattern (minus background).

Simulating a Mixture

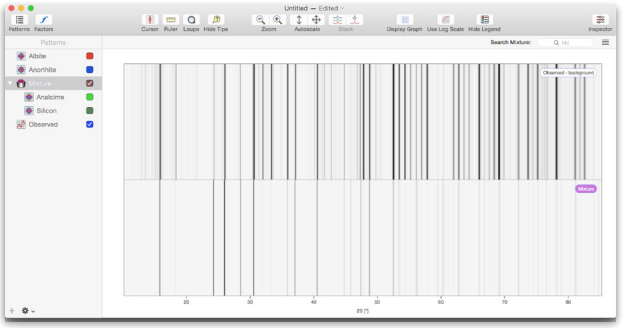
We shall now simulate a two-phase mixture of Analcime and Silicon, in order to replicate the observed pattern.

50 Select the Silicon and Analcime patterns: click on **Analcime** in the Patterns List, then hold down the **command** key (Mac) or **control** key (Windows) and click on **Silicon**.

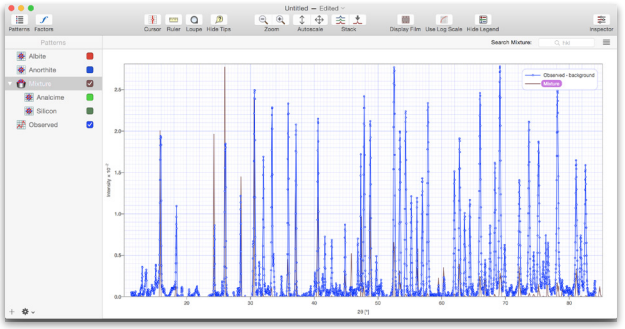
51 Right-click in the Patterns List and choose: **New Mixture with Selection**. A new, "mixture", pattern is added to the plot, representing an equal mix of your two selected components, Silicon, and Analcime.

Icon for mixture pattern

52 Uncheck the checkboxes for Analcime and Silicon, leaving just your simulated mixture and the observed pattern:



53 Click the toolbar's **Display Graph** icon ( ) to replace films with graphs.



Editing the Mixture

Next, we shall edit the mixture's composition, by changing the relative proportions of its three components.

54 Stack the graphs by clicking the **Stack** button ( ) in the toolbar, or by choosing: **View > Stacked**.

55 Select the simulated Mixture by clicking on its entry in the Patterns List.

56 Ensure that the Inspector is visible, and click its **Parameters** tab to display the Parameters List.

57 Click the **Mixture** disclosure button to show its controls. Both simulated phases, are displayed, with equal phase proportions (0.5).

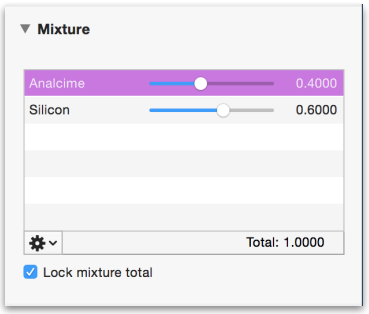
58 Select the **Analcime** item in the Mixture list (notice that selecting a phase in this list is independent of any selection elsewhere in the program).

59 Adjust the slider to change the proportion of Analcime. Notice that the proportion of Silicon also changes, as the **Lock mixture total** checkbox indicates that the overall mixture total should remain constant.

> **Tip:** You can unlock the Lock checkbox, which then allows you to adjust the proportions of mixture components independently. However, there is one major caveat: CrystalDiffract won't allow the overall mixture total to exceed 1.0, and will automatically reset the other components if you enter too high a proportion of one component.

60 For more precise control, click on the number to the right of the slider and then type **0.4** into the edit text box that appears. Press the **return** key on your keyboard to replot the mixture with this composition:

Adjusting the composition of our simulated mixture



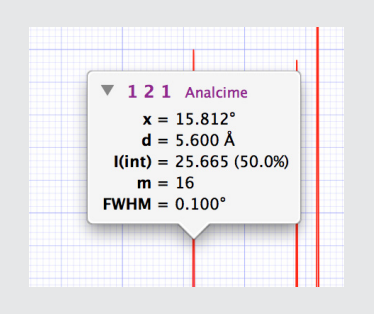
Identifying Peaks

Now that we have a fairly good characterization of our Observed crystals, we shall direct our attention to the simulated patterns, and how to measure these peaks.

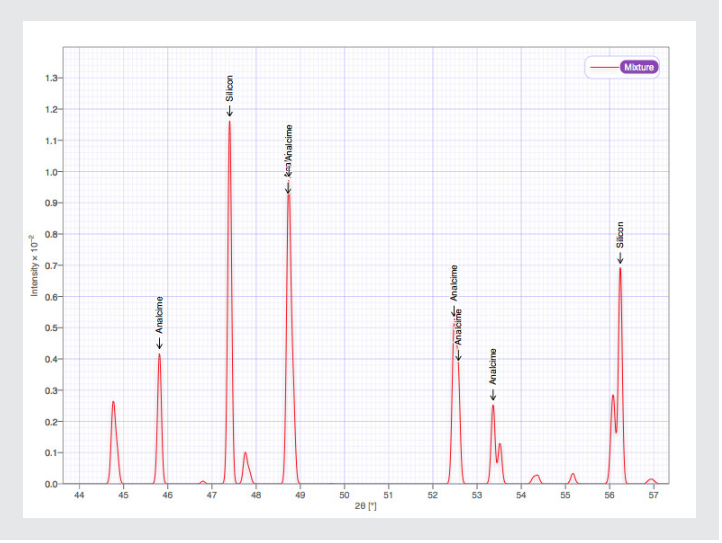
61 "Unstack" the diffraction patterns by pressing the **Collapse** button ( ) in the toolbar, or by choosing: **View > Collapsed**.

62 Hide the observed pattern by unchecking its checkbox in the Patterns List.

63 Move the mouse pointer over various peaks in the Graphics pane. "Peak Tips" will appear, identifying each peak. Click inside the Peak Tip to expand it and reveal more information about the peak:



64 Ensure that the Mixture pattern is selected, then open the **Labels** group in the Format Inspector. Turn on the "**Show Labels**" checkbox. Your simulated mixture now has peak labels:



Notice that, for clarity, the weaker peaks remain unlabelled.

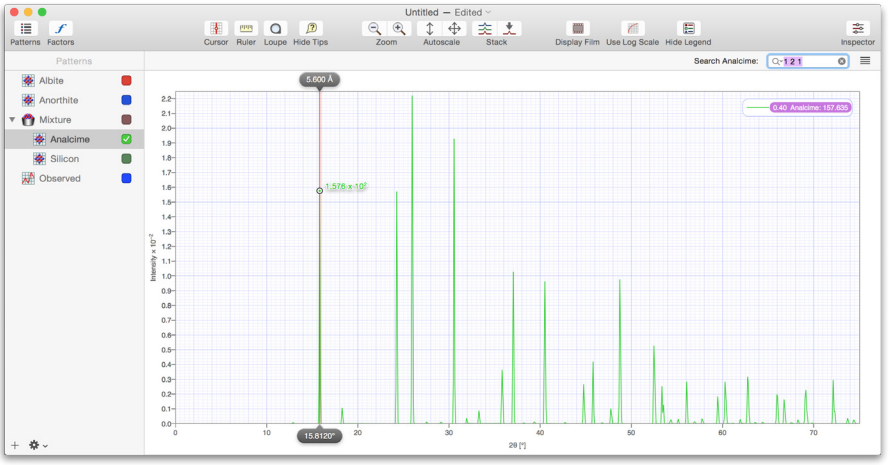
Searching for Peaks

You may have noticed that the Graphics Pane has a search bar. We shall now use this to find a reflexion.

65 Using the Patterns List checkboxes, hide the Mixture pattern and show Analcime.

66 Search for the (121) peak in Analcime: type: `1 2 1` (note the gaps between indices) into the **Search** field, and press the **return** or **enter** keys on your keyboard. The cursor appears, showing the location of the peak:

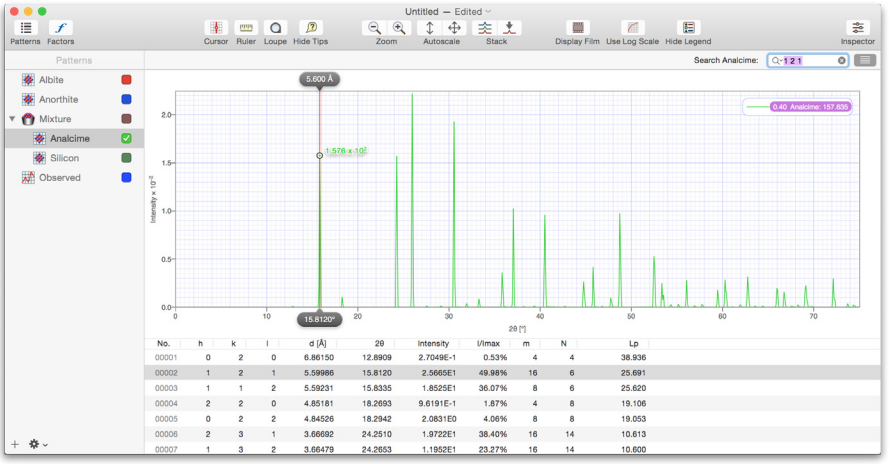
Using the Search field to locate a displayed peak



Tip: Double-click a peak in the Graphics Pane to go directly to its corresponding entry in the Reflexions List. (This is the reverse of double-clicking on a line in the Reflexions List!)

67 Click the **Reflexions List** button ( ) on the right of the **Search** field. The Reflexions List appears, with the current reflexion (121) highlighted:

Locating a reflexion in the Reflexions List.



68 Locate the 040 reflexion line in the list (you may have to scroll the list down a few lines), then double-click the line. The position of the 040 reflexion is shown in the Graphics Pane.

69 Hide the Reflexions List by clicking on its button on the right of the **Search** field (or choose: **View > Layout > Hide Reflexions List**).

Parameter Control

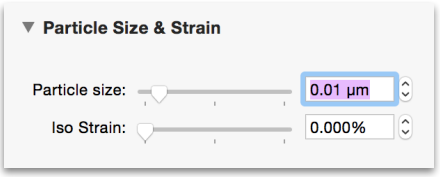
We shall now demonstrate that it is possible to apply different parameters to different simulated patterns.

70 Verify that the Analcime pattern is still selected, and that the **Parameters** tab is selected in the Inspector.

71 Open the **Particle Size & Strain** group in the Parameters Inspector. Two items are shown: **Particle Size** and **Iso Strain**.

72 Change the **Particle Size** value from 1.0 to 0.01 µm: drag the slider to the left (or, for more precise control, type `0.01` into the text box and press the **return** key.) Notice that the Analcime peaks become very broad and their heights are reduced.

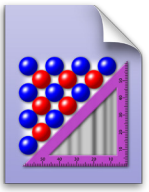
Simulating the effect of particle size.



Saving a Diffraction Experiment

Finally, we'll save our work in a single, self-contained CrystalDiffract document. This can be used to reconstruct everything in our window, without needing any of the original files.

CrystalDiffract document icon.



73 Choose the **File > Save** command, then specify a filename and location for your session file.

74 Quit CrystalDiffract: on the Mac, use the **CrystalDiffract > Quit** command; on Windows, use the **File > Exit** command, or click the diffraction window's Close box.

75 Relaunch the program and open your previously-saved session file. You should have the same window size, in the same position, with the same display as before.

> **Tip:** Use the **File > Open Recent** submenu to open a recently-saved document.

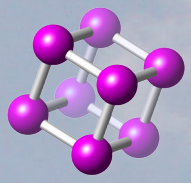
Summary

You've now reached the end of the CrystalDiffract tutorial. Well done! You've seen how to load data and structure files, measure a pattern, compare it with other patterns, simulate a mixture, identify simulated peaks, and edit sample properties.

Experimental Workbench

There's much more to CrystalDiffract than just displaying diffraction patterns. The program also lets you create new structures from scratch, edit structural data, visualize and edit atomic scattering factors, export tables of data, save individual diffraction profiles—and of course, copy, save or print high-quality vector graphics.

CrystalDiffract is designed to work with CrystalMaker: you can view a crystal structure in CrystalMaker then, with a single menu command, simulate its diffraction properties. You might wish to edit your structure in CrystalMaker, and get immediate feedback on how this affects the diffraction properties. In fact, the ability to view multiple diffraction patterns in the same CrystalDiffract window is a great way to analyse structure/diffraction relationships.



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