




Bruker D8 Discover XRD User's Guide

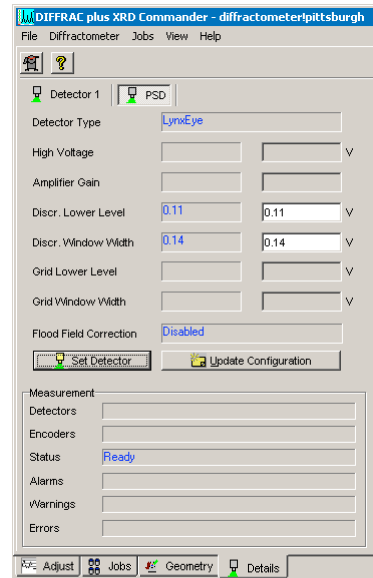
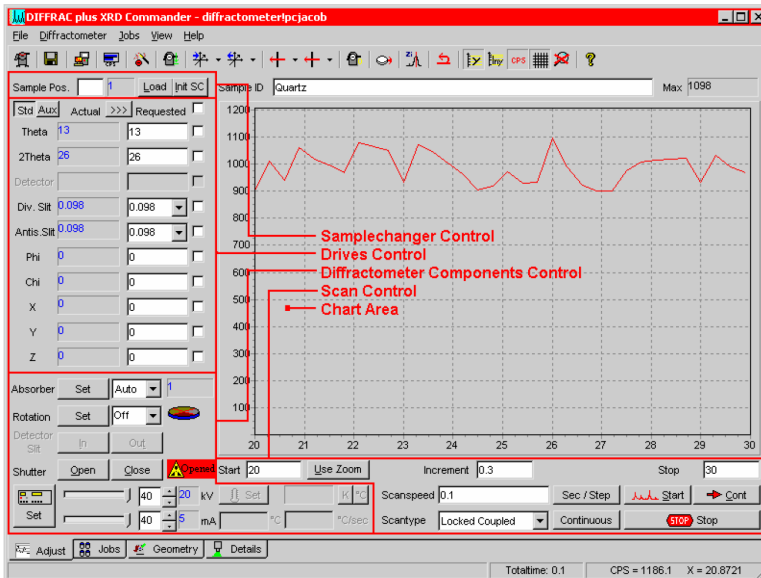
I. Powder Diffraction Measurement using LynxEye Detector






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1. Fill in the Log-book with relevant information before you begin analyze your sample.
2. Check System Control Buttons (located at the front side of the system) and make sure the Ready and On LED lights are on.

	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; padding: 5px;"> <p>Stop Button: Emergency Stop! If hit, it switches off the control electronics and high-voltage generator immediately. The X-ray source is turned off, and all moving drives will stop instantly.</p> </td> <td style="width: 50%; padding: 5px;"> <p>Turn On/Off Illumination This button turns the fluorescent lights located in the D8 enclosure on or off</p> </td> </tr> <tr> <td style="padding: 5px;"> <p>Turn On/Off Fan This button turns the fan on top of the D8 enclosure on or off.</p> </td> <td style="padding: 5px;"> <p>Turn Off Power of the Diffraction System: switches off the control electronics, the high voltage generator and all components connected to the AC outlets.</p> </td> </tr> <tr> <td style="padding: 5px;"> <p>System Instrument – Key Switch: lock/unlock the system. Can be removed in the “locked” position. During normal operation the switch must be “unlocked”.</p> </td> <td style="padding: 5px;"> <p>Turn On Power of the Diffraction System: switches on the control electronics, the high voltage generator and all components connected to the AC outlets.</p> </td> </tr> <tr> <td style="padding: 5px;"> <p>Open Door Button Under normal operating conditions the door handles are locked by a mechanical shutter. To open the front door(s) this button must be pressed. Then the front door(s) can be opened. If the Open Door button is activated while the tube window is open, the X-ray shutter will close automatically. After closing the front doors, the X-ray shutter can be opened again.</p> </td> <td style="padding: 5px;"> <p>High Voltage-Rotary Switch: Turning the switch to the right position and hold it until the orange status display (READY) starts flashing. The switch can be released now.</p> </td> </tr> </table>	<p>Stop Button: Emergency Stop! If hit, it switches off the control electronics and high-voltage generator immediately. The X-ray source is turned off, and all moving drives will stop instantly.</p>	<p>Turn On/Off Illumination This button turns the fluorescent lights located in the D8 enclosure on or off</p>	<p>Turn On/Off Fan This button turns the fan on top of the D8 enclosure on or off.</p>	<p>Turn Off Power of the Diffraction System: switches off the control electronics, the high voltage generator and all components connected to the AC outlets.</p>	<p>System Instrument – Key Switch: lock/unlock the system. Can be removed in the “locked” position. During normal operation the switch must be “unlocked”.</p>	<p>Turn On Power of the Diffraction System: switches on the control electronics, the high voltage generator and all components connected to the AC outlets.</p>	<p>Open Door Button Under normal operating conditions the door handles are locked by a mechanical shutter. To open the front door(s) this button must be pressed. Then the front door(s) can be opened. If the Open Door button is activated while the tube window is open, the X-ray shutter will close automatically. After closing the front doors, the X-ray shutter can be opened again.</p>	<p>High Voltage-Rotary Switch: Turning the switch to the right position and hold it until the orange status display (READY) starts flashing. The switch can be released now.</p>
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3. Before opening the D8 door, check for any abnormalities in and around the instrument. Give particular attention to the **shutter lights on the X-ray tube**. The **green light** indicates that the tube is energized (light should be lit). The two **red lights** indicate the **shutter position** (if they are lit the shutter is open and you must not open the doors on the D8; if the red lights are off, then it is safe to open the doors and mount the sample stage on the goniometer).
4. Mount sample in the appropriate holder and then place the holder in the sample stage on the goniometer.
5. If the **XRD COMMANDER** is not opened, click the icon,  on the desktop to open it.
6. Set up DIFFRACplus XRD Commander



- 1) Click the **Details** tab to activate the **Details** page,
 - a. Select the **PSD detector (LynxEye)**.
 - b. Input **Discriminator Lower Level: 0.12; Discriminator Window Width: 0.14**.
 - c. Click **Set Detector** button.
- 2) Activate the **Adjust** page
 - a. Make sure that for each drive its actual position is shown in **blue**. If any of the drive positions are displayed in dark red, you will not be able to use the D8 — **ask for help**.
 - b. Set the X-ray generator voltage to **40 kV** and the current to **40mA** or other your desired combination.
 - c. Find zero position of the **Z** drive for your sample:
 - i. Open the Video program by clicking the video icon,  **VIDEO**, and start live view.
 - ii. In **XRD Commander** window, click **Direct Mode** icon,  to enter direct laser control mode.
 - iii. In **Direct Command** window, type **RC1** and press **Enter** to activate the direct laser control mode.
 - iv. Type **OC16,1** to turn the laser on.
 - v. Close the **Direct Command** window.
 - vi. Input a specific **Z** position and click the **drive to** icon,  to adjust the **Z** drive to center the laser spot at the crosshair.
 - vii. Open the **Direct Command** window again, and type **OC16,0** to turn off the laser.
 - viii. In Video program, stop Grab the live video.
- 3) Select measurement parameters:
 - a. scan type: **Locked Coupled, continuous** scan.
 - b. start: **>3.5°**, stop: **<100°**, scanspeed: **0.40** Sec/Step, and increment: **0.04°**.
 - c. slit size: **0.2** mm; high voltage: **40** kV, current: **40** mA.
- 4) Click **Start** button,  to begin a measurement.
- 5) Click **Stop** button,  to end the immediate measurement.

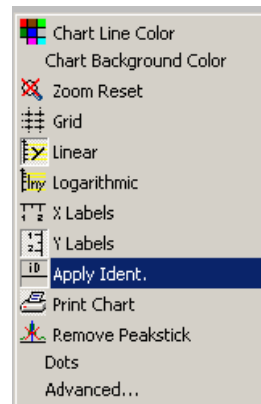
7. After the scan is complete

- 1) Save the measured data as a **RAW** data file to your folder on the hard drive. **XRD Commander** displays the **Save As** dialog box, and you must enter the **RAW** filename. The **RAW** data file can later be examined (e. g., with EVA).


Note: by clicking the *right mouse button* inside the **Chart** area, you will bring a popup menu:

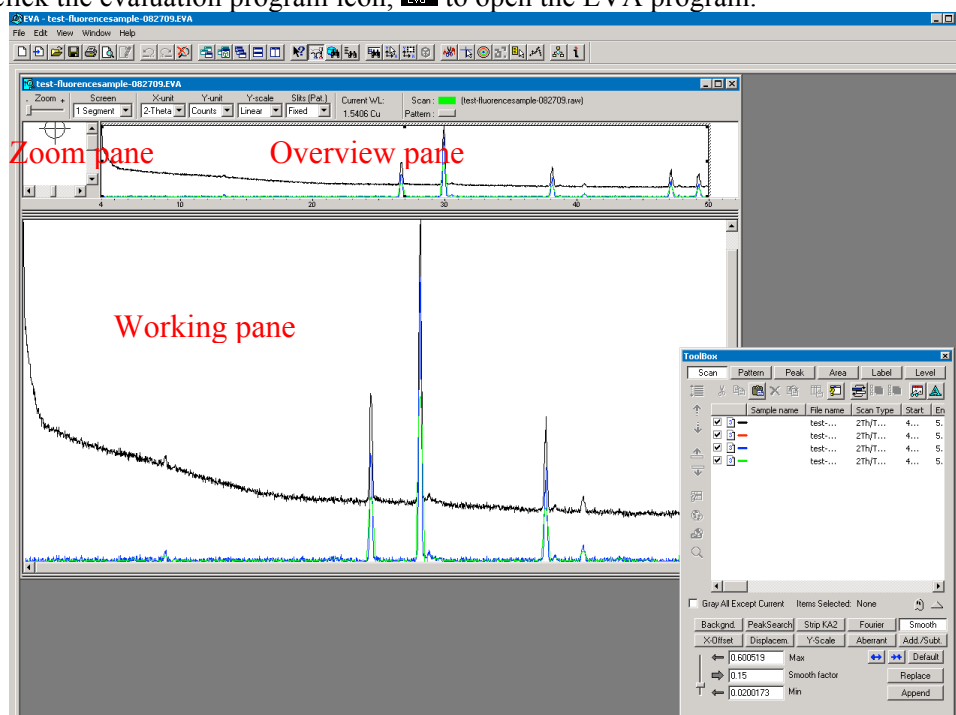
- **Apply Ident** (applies the sample identification on the top axis)
 - Print Chart
 - Remove Peakstick
 - Dots (shows the scan data as little dots instead of connected lines)
 - **Advanced** (shows advanced customized options for the chart area such as **Save the chart data as a Text, XML, HTML, or Excel file**).
- Export*→*Data*→*Excel*→*Save As* to your folder on the hard drive.

- 2) Set the X-ray tube voltage to 20 kV, current to 5mA
- 3) **Minimize XRD Commander. Do not close the program.**






II. Diffraction Evaluation using *DIFFRAC^{plus} BASIC* Evaluation

1. Double click the evaluation program icon,  to open the EVA program.

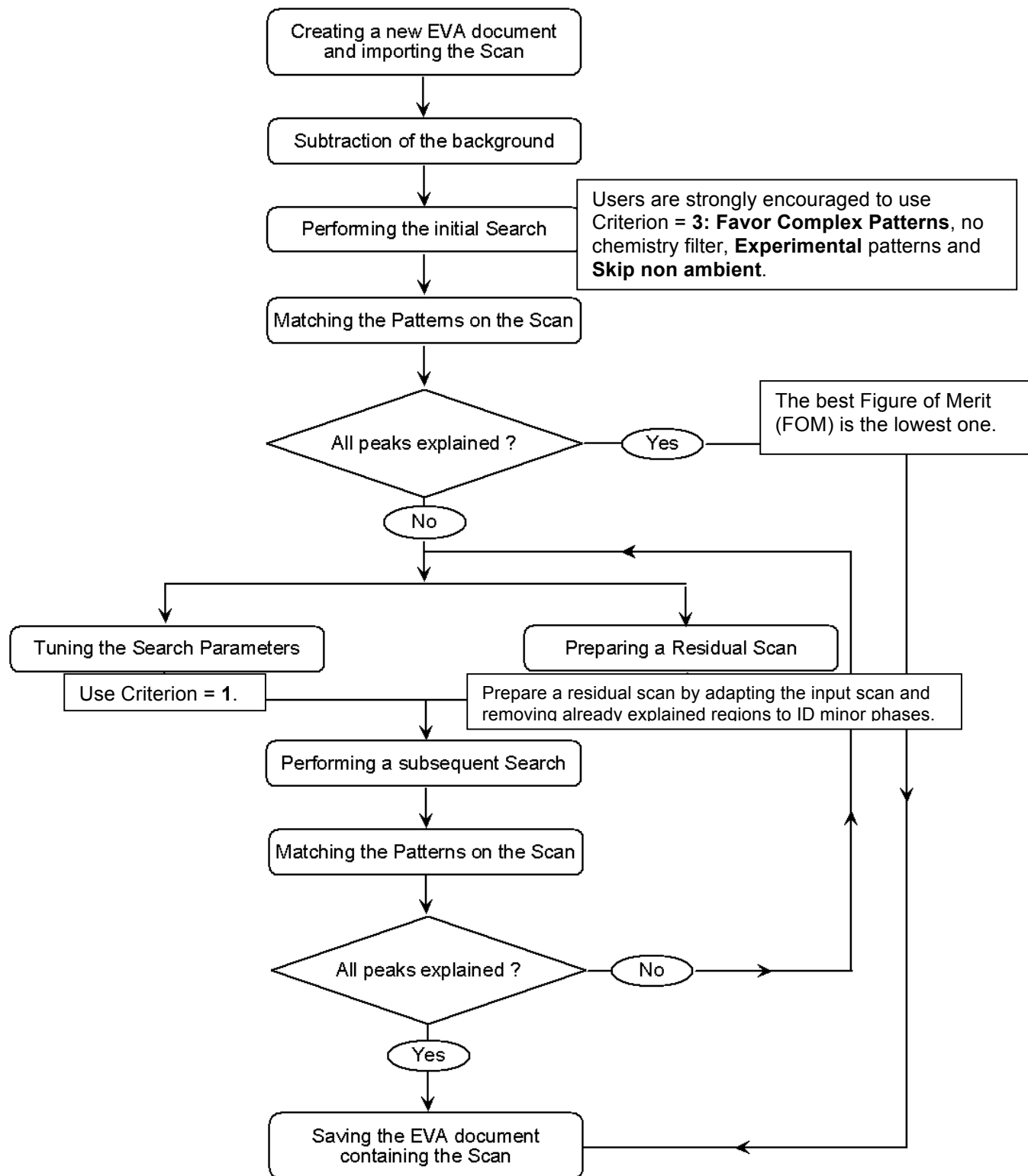


2. Creating EVA Documents
 - a. On the toolbar, click the **Import** button.
 - b. Locate the data **.RAW** file you want to import in the directory of your RAW files.
 - c. Click **Open**.
3. Working with Scans
 - a. Performing a Background Subtraction
 - i. The **DIFFRAC** traditional method: *prepare data for search/match*.
 - 1) Click the **Background** button on the Data Treatment palette
 - 2) Use the slider to adjust the curvature if there are background humps.
 - 3) Select the **Subtract** from Scan check box.
 - 4) Click **Append** to append the background-subtracted scan data to the document
 - ii. An enhanced method: gives a smooth curve.
 - iii. A **Bézier** method: allows you to draw the background exactly the way you want.
 - 1) Draw a calculated background with the method above; get as close as you can to the background you intend to draw.
 - 2) Check the **Bézier** box to transform this background into curves.

- 3) Move the *slider* to adjust the curve to what you intend to draw.
 - 4) Click **Edit** button to display all the control points: the passing points are disks, the tangent points are squares, and the tangent vectors are in dashed lines. You can **move**, **erase**, or **add** a passing point.
 - 5) Check the **Subtract** box and then click on **Append** to create your drawn background.
- b. Performing a Peak Search
 - i. On the Data Treatment palette click the Peak Search button.
 - ii. Move the slider to see ghost peaks in the Overview and Working panes.
 - iii. When no manual editing is required, click **Make DIF** to transfer the found peaks to a DIF pattern.
 - iv. When manual editing is required, click **Append To List**.
 - c. Computing $K_{\alpha 2}$ Stripping
 - i. Click the **Strip $K_{\alpha 2}$** button on the Data Treatment palette.
 - ii. Use the slider to adjust the Intensity Ratio.
 - iii. Click Append to append the $K_{\alpha 2}$ subtracted scan to the document
 - d. Smoothing Scans
 - i. Click the Smooth button on the Data Treatment palette.
 - ii. Use the slider to adjust the Smooth factor.
 - iii. Click Append to append the smoothed scan to the document
 - e. Fourier Smoothing and Expansion
 - i. Click the Fourier button on the Data Treatment palette.
 - ii. In the Fourier treatment control panel, select the expansion (x1, x2, x4, x8, and x16).
 - iii. Use the slider to adjust the cutoff.
 - iv. Click Append to append the smoothed scan to the document.
4. Search/Match:  (** see the next page for a general procedure.)
 5. Saving and Printing in EVA.
 6. Export file as ASCII file.
 - a. **Export** your EVA file as a RAW file;
 - b. Open the **RAW File Exchange** , or **XCH File Exchange** , to convert your RAW and/or processed files to ASCII files.
 7. Exit EVA after you complete data processing.
 8. Sign off the instrument by filling in the log-book.

** The purpose of EVA **Search/Match** is to search the current scan of an unknown material and then identify reference patterns that are likely to explain the unknown scan.

The general procedure for EVA Search/Match is as follows:



Use the **Scroll List** mode to match the scan