Bruker D8-GADDS
User's Manual

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- Diffractometer's mailing list: d8gadds_dif@socrates.berkeley.edu
- Diffractometer's web-page: http://theanvil.cchem.berkeley.edu/~gadds/
- Computer: bragg.cchem.berkeley.edu
  (username: gadds, password: …….)
- X-ray source: CoKα λ=1.79026Å

If you have any doubt, ask Fabio before improvising!

Important

- Each time you touch the diffractometer, be gentle, especially with the microscope, collimator, detector, or beam stop.
- If the shutter is open, never open the diffractometer's windows.
- When you are moving the goniometer (manually or with the computer), beware of collisions.
- Before starting a measurement, always check the alarm lights.
- Do not log off the computer
- The generator operating power is 45kV, 35mA.
- The generator sleeping power is 20kV, 5mA.

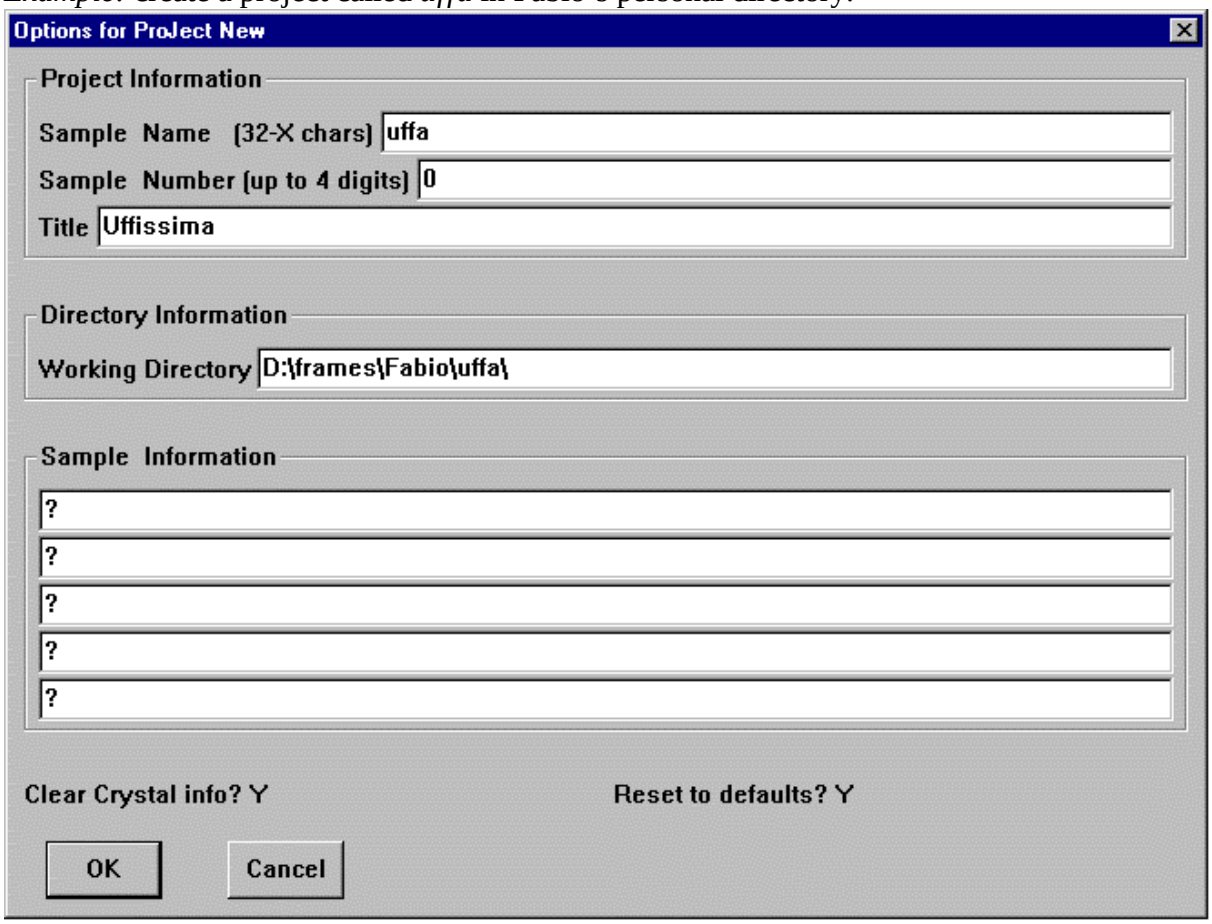
Measuring a Powder X-ray Diagram

- Open the program Gadds (if it is not already open).
- Set the generator's power to the user's setting (45kV, 35mA). Before starting the measurement, always check the generator's power (*Collect:Goniometer:Generator*)

Create a new Project

- Select *New* under the *ProJect* menu.

Example: create a project called *uffa* in Fabio's personal directory.



Note: you must create the new project in your personal directory, by writing D:\frames\username\projectname\ in the field *Working Directory*. The program will create a new subdirectory called *projectname* in the *username* directory. The *projectname* directory should contain a file named *gadds._nc* where project's preferences are stored. You can also use an old project (*ProJect:Load*), or read an old gadds._nc file (*Edit:Configure:Read*).

- Check whether the numbers on the gadds window are correct and whether the program loaded the right detector's distance, flood field correction, and spatial correction.

Moving the Goniometer

- Within the program gadds: Select *Collect:Goniometer:Drive.*
- Using the Manual Control Box: Select *Collect:Goniometer:Manual* and press *Shift+F1* on the Manual Control Box.

Commands on the Manual Control Box:

1:	2θ	5:	x
2:	ω	6:	y
3:	ϕ (not in use)	7:	z
4:	χ (not in use)		
↑↓:	to move	+/-:	drive speed

Emergency stop when driving the goniometer: press any key on the keyboard.

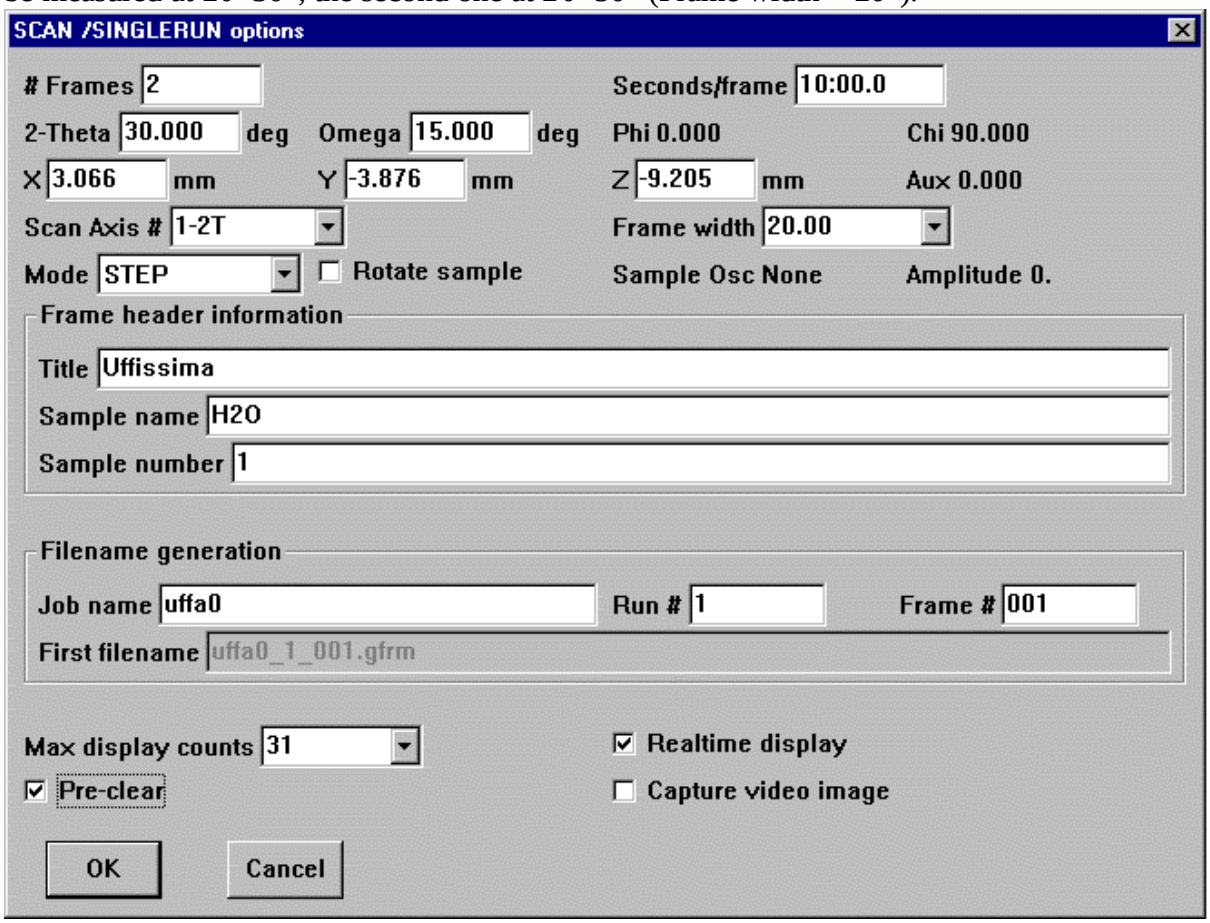
Mounting the Sample

- If you are measuring capillaries, set the collimator in the standard position (with the pin in the hole) and mount the *beam stop*.
- If you are measuring plates, set the collimator at a higher distance (with the pin outside the hole) and do not use the beam stop. Be careful that the direct X-ray beam *does not* hit the detector.
- Mount the sample on the XYZ-stage.
- Move the goniometer angles (2θ and ω) at the position where you want to start the measurement. Beware of *collisions*, especially with the beam stop, sample holder, or collimator.

1. Option: single sample

- Select *Collect:Goniometer:Manual.*
- Center the sample in the microscope using the manual control box.
- Close the diffractometer's window. The alarm light should stop blinking.
- Select *Collect:Scan:SingleRun.*

Example: using the following options, you will measure two frames (uffa0_1_001.gfrm, uffa0_1_002.gfrm) for 10 minutes each, with 2θ as Scan Axis and ω=15°. The first frame will be measured at 2θ=30°, the second one at 2θ=50° (Frame width = 20°).



Note: Use the symbol @ for the x, y, z positions (@ = current value). The usual *Scan Axis* is 1-2T (2θ). The typical ω values are 0° for *capillaries* and 15° for *reflection* measurement. For *transmission* measurements select *Coupled* in the *Scan Axis* field, which stays for a 2θ/θ scan (ω=2θ/2).

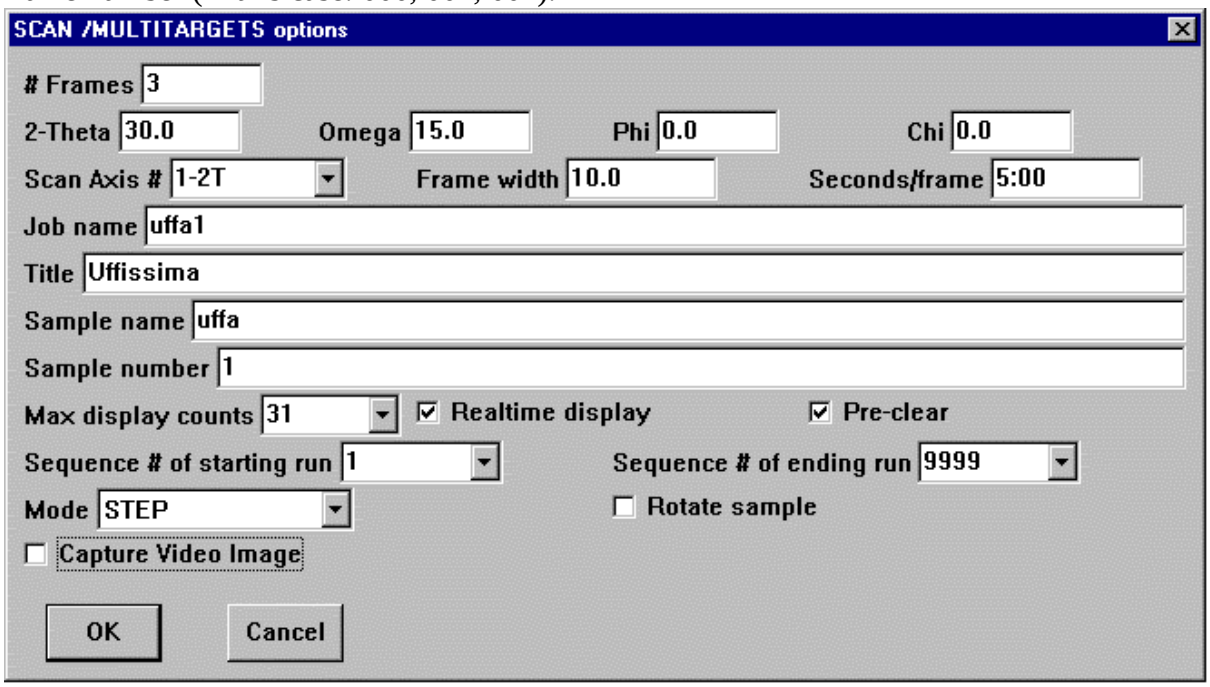
2. Option: multiple samples

- Select *Collect:Scan:PickTargets.* You enter in the manual mode.
- Center the first sample in the microscope using the manual control box.
- Press *Esc* on the computer to exit the manual mode and add the position to the targets list.
- Repeat this procedure until you have centered all your samples.

Beware of collisions!

- With *Collect:Scan:EditTargets*, you can check and edit the targets in the list.
- Select *Collect:Scan:MultiTargets.*

Example: using the following options, you will measure the targets in the list collecting three frames for each target. The frames are collected at 2θ=30°, 40°, and 50° (ω is fixed at 15°). The frames names are uffa1_X_00Y. gfrm with X = target number (Sequence #) and Y = frame number (in this case: 000, 001, 002).



Note: all the targets are measured using the *same* options (angles, time, ….).

It is very useful for running several samples plus a *blank* X-ray diffractogram, which can be used for subtracting the background caused by the air scattering.

3. Other Options

- *EditRuns/MultiRuns*: you can program several runs with different measuring angles, but only *one* single target.
- *CoupledScan*: you can perform a 2θ/θ coupled scan (ω=2θ/2) using the area detector as a point detector.
- *Add*: you can measure one frame at the current, and fixed, angles for a specified collecting time. Note that the frame is not automatically unwarped, therefore you have to select *Process:Spatial:Unwarp* and save the unwarped file as .gfrm.

To interrupt a measurement: press Ctrl+Break.

If a measurement is currently running, you can not use the program Gadds. Open the program Gadds-Offline, instead.

Analyzing the Frames

The frames are already unwarped (except if you use *Add* to collect the frame).

- Select *File:Load* to load a frame.
- Select *Analyze:Cursors:Conic* to check the 2θ angle of a reflection.
- Select *Analyze:Cursors:Pixel* to check the intensity measured on a pixel.
- Select *Analyze:Graph:Write* to save the graphics in the frames (not the frame itself) for further use.
- Select *Analyze:Graph:File* to open a saved graphic on a frame.

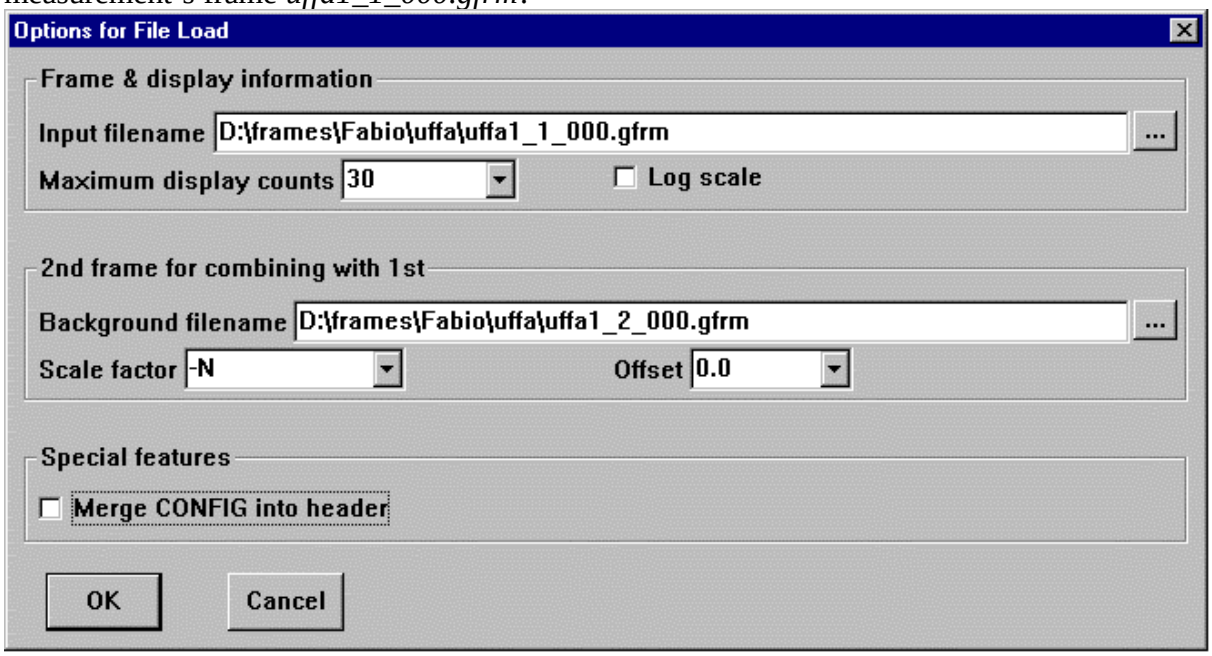
The program Gadds is also able to calculate the percent crystallinity, the stress of a probe, and many other properties. If you want to use these cool features, check with Fabio and read the software manual.

Integrating the Frames

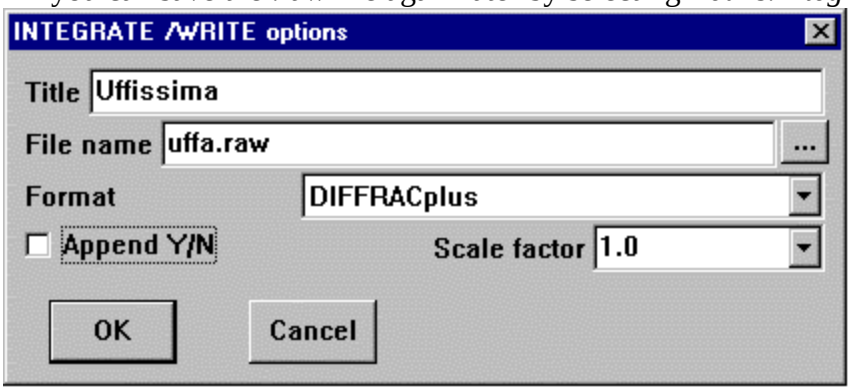
- Select *File:Load* to load the frame.

If you have measured a blank frame, you can subtract it from the measurement's frame in the *Load* window.

Example: the blank frame *uffa1_2_000.gfrm* will be subtracted (*Scale factor* –N) from the measurement's frame *uffa1_1_000.gfrm*.



- Select *Peaks:Integrate:Chi*. Choose *3-Normalized by solid angle* (faster) or *5-Bin normalized* (slower) in the *Normalize intensity* field and the desired *Step size*.
- Using the *1,2,3,4* buttons and the *mouse*, you can adjust the size and position of the frame's sector that you want to integrate. The *Enter* button starts the integration.
- When the integration is complete, save the *.raw* file. Always choose the *DIFFRACPlus* format. The *Write* window comes automatically when the integration is completed, but you can save the .raw file again later by selecting *Peaks:Integrate:Write.*



Note: If your measurement has different ranges (more then one frame), you have to integrate the different frames separately. You should assign to 2θ *Start* the same value you used as 2θ *End* in the previous frame. Save the .raw file using always the same name, but select the option *Append* (except for the first frame).

Merging the different ranges

- Load the .raw file in the program *merge*.
- Select *Do it*. The program creates a merged file named *merge.out*.
- Rename this file with a .raw extension.

You can also do the merging inside the Gadds program by selecting Special:System and writing *merge* in the command field.

Printing a X-ray diffractogram

- Open the .raw file with the program EVA.
- Print the diffractogram

EVA has a lot of features. To learn about it, play with it and read the software manual.