Batch conversion
from 2D to 1D powder diffraction data

using

Fit2D[1] V12.043

A short tutorial

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1. Introduction

In situ powder diffraction data are often recorded using 2-dimensional detectors like image plates (like the widespread Mar345 detector) or CCD-cameras. Depending on the nature of the experiment and the readout times of the detector, hundreds or thousands of 2D-powder patterns are generated during such an experiment. In order to process the powder diffraction data with software like Powder3D (Hinrichsen, Dinnebier & Jansen, 2004), the series of two dimensional powder diffraction data must be converted into series of one dimensional powder patterns (2θ versus intensity). A suitable software for this purpose is the well known program Fit2D from Andy Hammerley (Hammersley, A.P.; Svensson, S.O.; Hanfland, M.; Fitch, A.N.; Hausermann, D. *High Pressure Research* **1996**, *14*, 235-248). The software is available free of charge at http://www.esrf.fr/computing/scientific/FIT2D/. FIT2D is a platform-independent multi-purpose data reduction, visualisation, and analysis program. Among other things, it allows for detector calibration and integration of powder diffraction data from 2-D detectors to 1-D 2θ scans.

The aim of this simple tutorial is to guide the unexperienced user through this first step of data reduction step-by-step.

2. Detector calibration

Before the start of any new experiment, it is good practice to measure a standard material like lanthanum hexaboride for calibration of wavelength, sample-to-detector distance and tilt angle.

2.1 Data input

After starting the Fit2D program, the screen to the right appears. The conditions of use should be accepted by pressing the <I ACCEPT> button
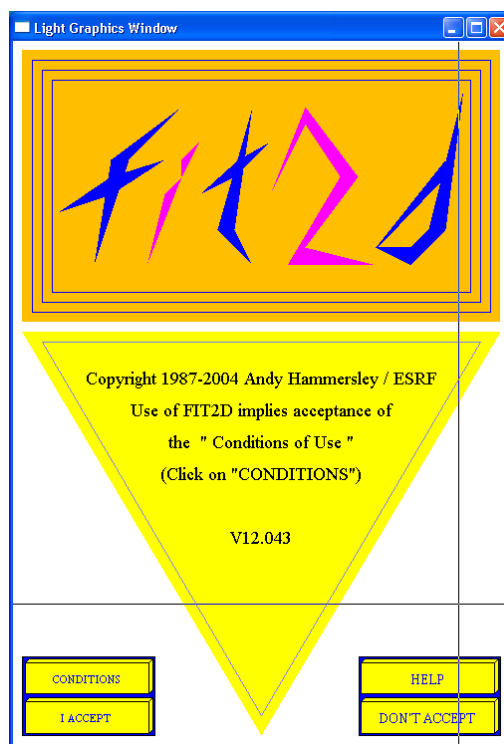
Now, the dimensions of the pixel arrays must be set. For the Mar345, the X- and Y-dimension are either 2300 or 3450 pixels. The extension of the filename often contains this information. Confirm the information by clicking <O.K.>.

Light Graphics Window

DIMENSIONS OF PROGRAM ARRAYS

(need to be big enough to store

and work on data)

| O.K. | CANCEL | ? | HELP | INFO |

DESCRIPTIONS	VALUES	CHANGE
FIRST DIMENSION OF ARRAYS	2300	X-DIMENSION
SECOND DIMENSION OF ARRAYS	2300	Y-DIMENSION
CREATE MEMORY ARRAYS	YES	MEMORY
CREATE VARIANCE ARRAYS	NO	VARIANCES

Click on variable to change, or 'O.K.'

The main menu appears in which POWDER DIFFRACTION (2-D) should be selected.

FIT2D GRAPHICS WINDOW

WELCOME TO FIT2D GUI

SELECT SCIENTIFIC INTERFACE

FROM GIVEN CHOICES

(OR CLICK ON "HELP" FOR INFORMATION)

FIT2D: SCIENTIFIC INTERFACES

| ? |
| HELP |
| 2-D FITTING |
| FILE SERIES |
| IMAGE PROCESSING (GENERAL) |
| KEYBOARD INTERFACE |
| MACROS / LOG FILE |
| MFIT (MULTIPLE 1-D FITTING) |
| ON-LINE CRYSTALLOGRAPHY |
| POWDER DIFFRACTION (2-D) |
| SAXS / GISAXS |
| SET-UP |
| TEST |
| EXIT FIT2D |

In the powder diffraction submenu, the 2D-powder pattern of the measured standard material must be read in using the <INPUT> button.
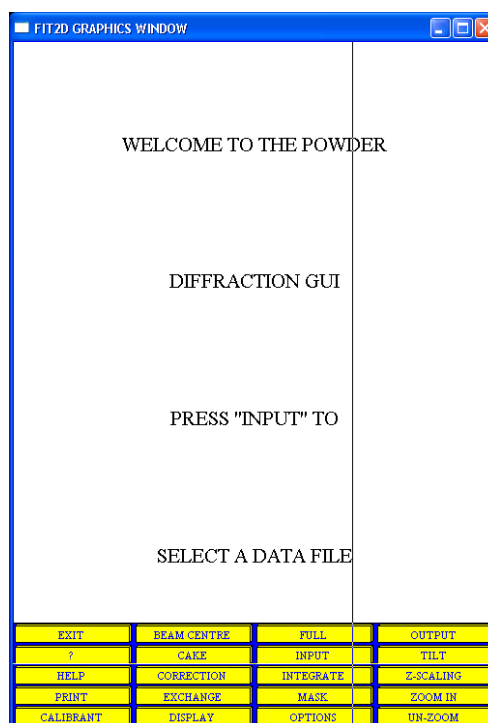
The following menu allows for selection of the file containing the 2D-powder pattern of the measured standard material. In our case, the file name is lab6_170_922_001.mar2300 containing the measurement of a lanthanum hexaboride capillary with a Mar345 detector of 2300x2300 pixels resolution.

Soon after reading the file, a menu with control of the detector distortion corrections occurs. In case of modern self-calibrating detectors like the Mar345, no external dark current, flat field or spatial distortion corrections must be applied. The menu is confirmed by pressing the <O.K.> button.

Now, the 2D-powder pattern of the reference material is displayed. It usually makes sense to select <inverse scaling> and <scaling to weak peaks> as display options.
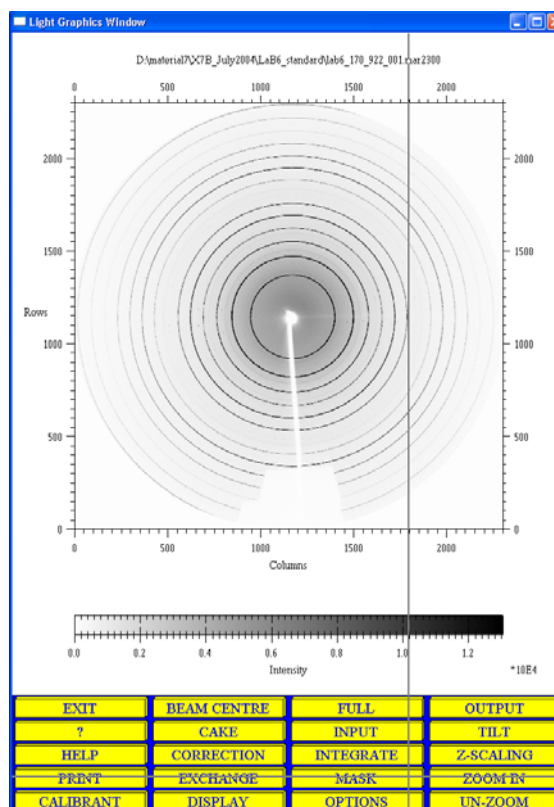
To change the color and scaling, click <OPTIONS>, followed by <COLOURS>.
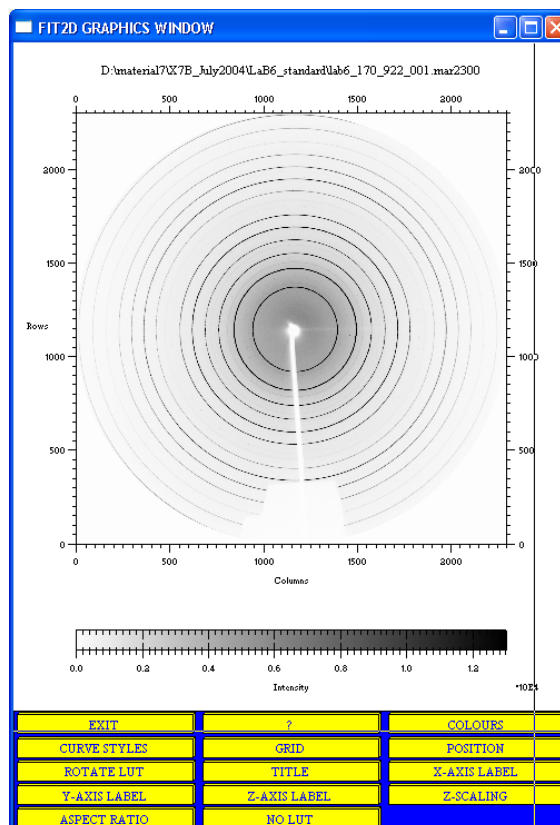
Now, press <INVERSE-GREY-SCALE> and then <EXIT>.
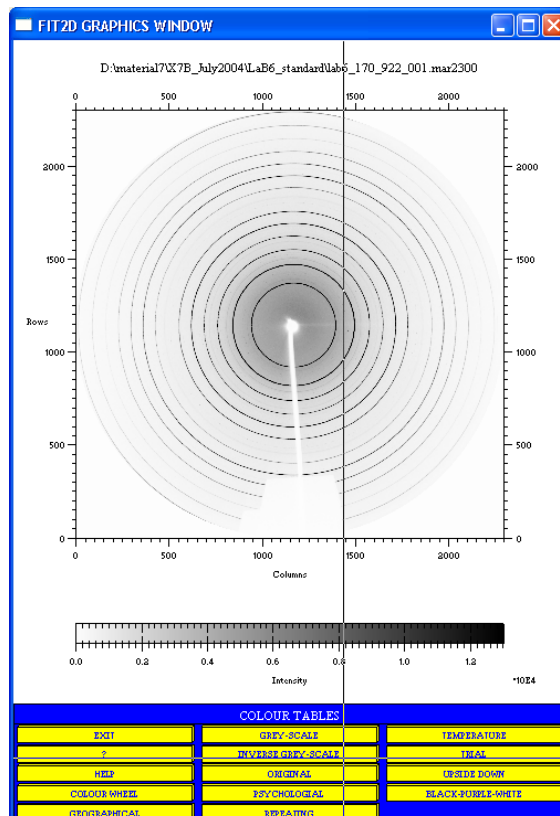
After pressing the <Z-SCALING> button, the intensity scaling menu appears in which the <WEAK PEAKS> button is pressed. Depending on the intensity of the background and the overall intensity of the pattern, other scaling options might be appropriate. To leave the menu, press the <EXIT> button.
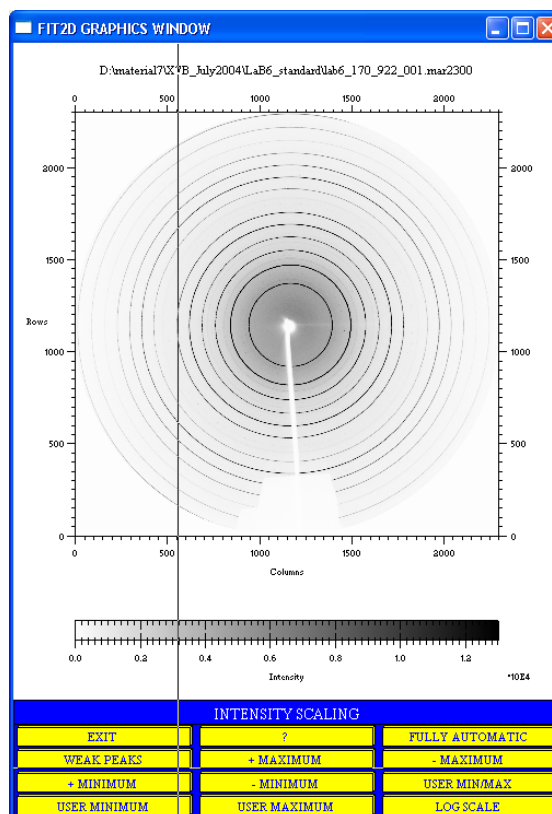
The next (important) step deals with the masking of unwanted scattering information which should not be used for the integration of the powder pattern like Kossel-lines, diffraction by single grains, beam stop etc.

To mask out the shadow of the primary beam stop, the region of interest must first be zoomed in using the <ZOOM IN> button.
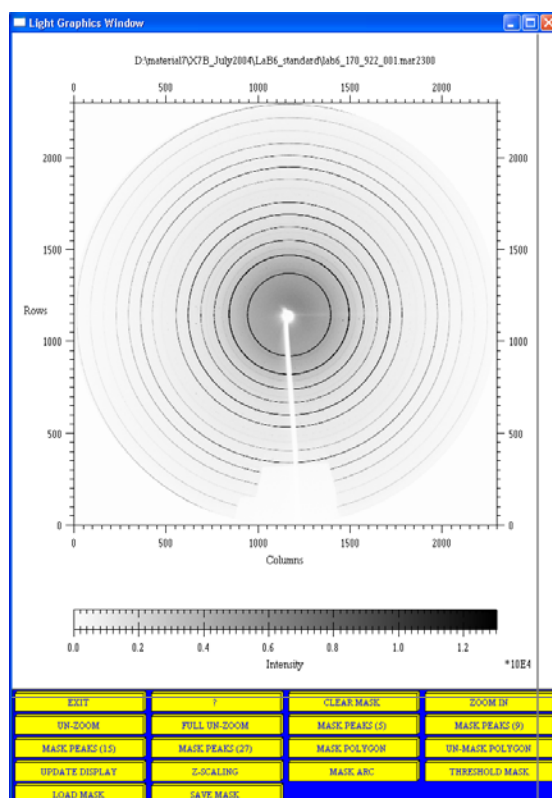
Now, after sufficient details are visible, the <MASK POLYGON> should be pressed.
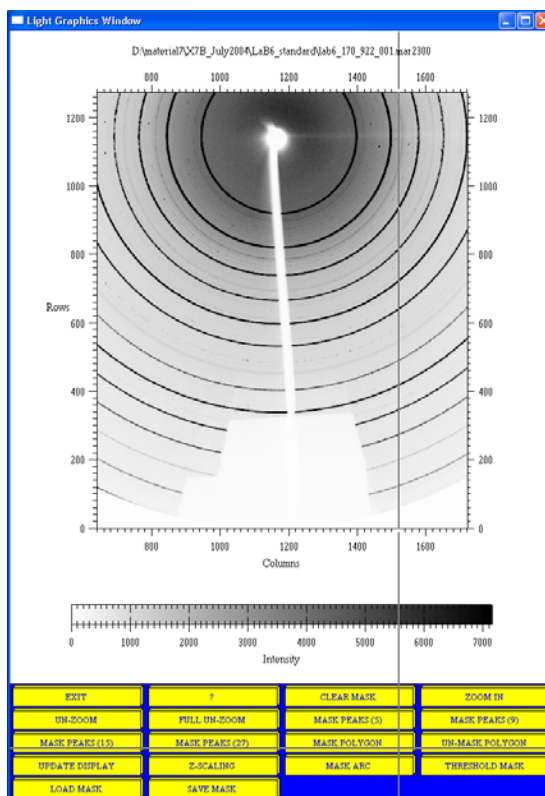
If ONE CLICK mode is active (check bottom right button), the polygon points can be set directly, in TWO CLICK mode, an additional confirmation and/or correction in a spy glass window is necessary. The two different modi should be selected according to required accuracy.
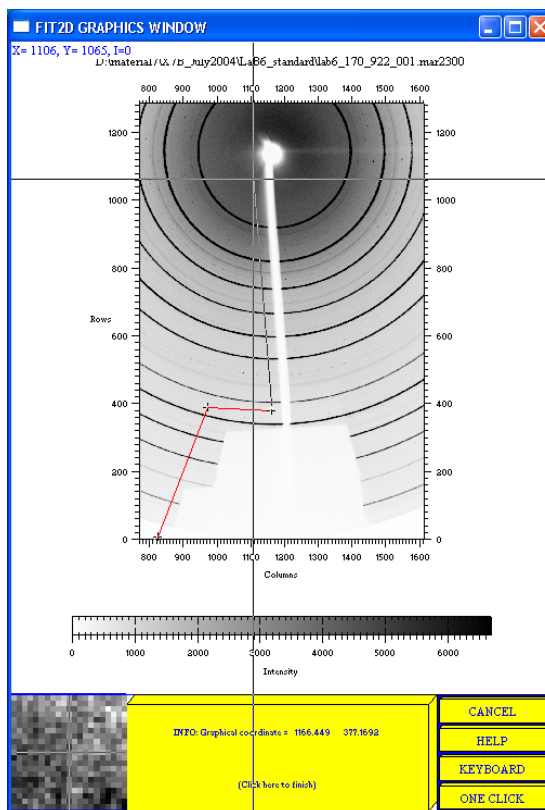
The final result of a masked primary beam stop should look more or less like the picture to the left. It might be necessary to mask out additional parts of the 2D powder pattern using other menu options.

Before leaving the masking menu by pressing the <EXIT> button, make sure that the <FULL-UN-ZOOM> button has been pressed, otherwise, the integration will only be performed on the 2D-data visible in the actual window.
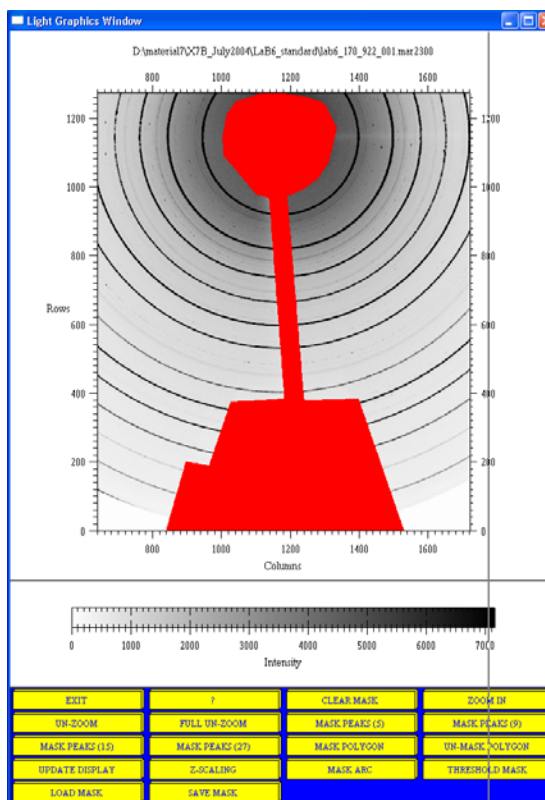
If you see the full 2D-pattern with the selected mask, press the <CALIBRANT> button. In the calibration menu select the reference material used (LANTHANUM HEXABORIDE in our case).
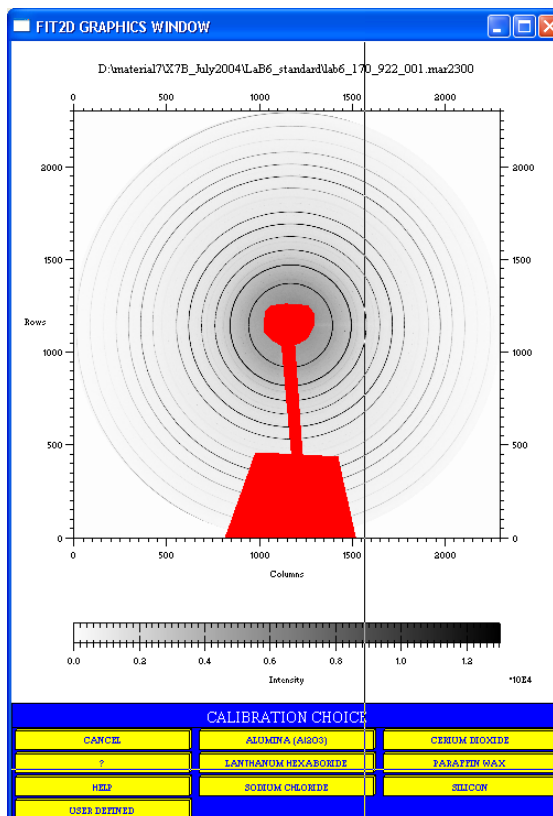
Fill in the approximate values of the wavelength and the sample –to-detector distance. The refinement flags for the sample-to-detector distance, the wavelength, the non-orthogonality, and the X/Y-beam centre are usually switched on. After pressing the <OK> button, the program asks for at least three coordinates of the inner ring of the reference material.

To define the inner ring, TWO CLICK mode should be active and some 10 points equally distributed over the inner ring should be selected to ensure proper statistics.
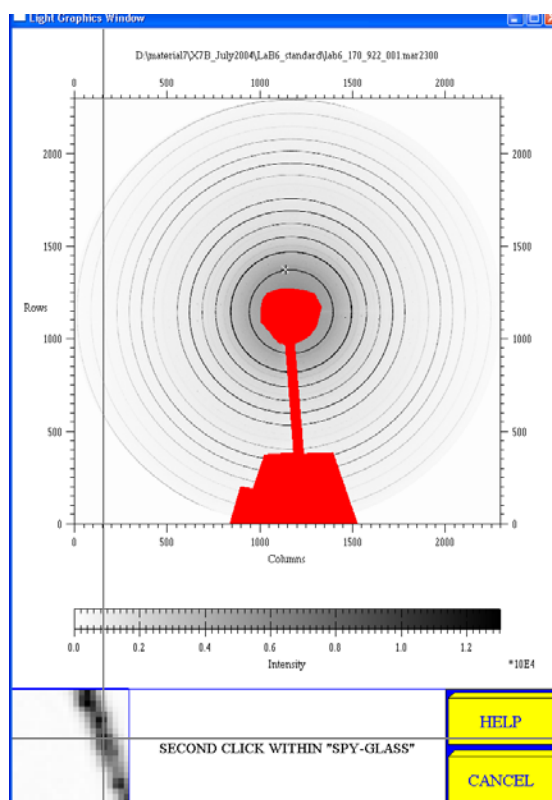
After confirmation of the last point, the program automatically searches for all rings of the standard material.

Now, it is time to perform the integration of the 2D-powder data of the reference material to check the correctness of the previous actions. Press the <INTEGRATE> button.
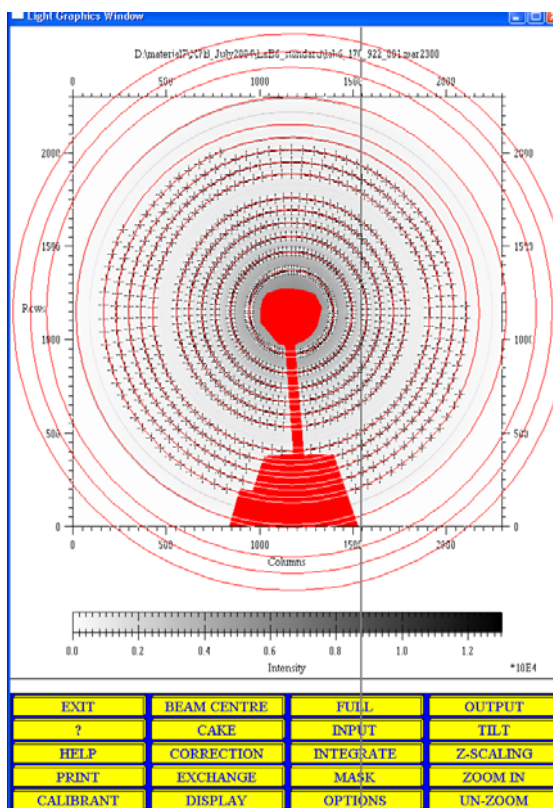
The first of two parameter windows contains all important parameters necessary for this and the following integrations. This is a good moment to check for unrealistic parameters.

If the Fit2D program is exited regularly, it will keep the values but is always a wise idea to note them down manually on an extra sheet of paper.

Press the <O.K.> button to continue to the second parameter window.

EXPERIMENTAL GEOMETRY

CONTROL FORM

DESCRIPTIONS	VALUES	CHANGE
SIZE OF HORIZONTAL PIXELS (MICRONS)	150.0000	X-PIXEL SIZE
SIZE OF VERTICAL PIXELS (MICRONS)	150.0000	Y-PIXEL SIZE
SAMPLE TO DETECTOR DISTANCE (MM)	149.9791	DISTANCE
WAVELENGTH (ANGSTROMS)	0.922064	WAVELENGTH
X-PIXEL COORDINATE OF DIRECT BEAM	1169.947	X-BEAM CENTRE
Y-PIXEL COORDINATE OF DIRECT BEAM	1146.340	Y-BEAM CENTRE
ROTATION ANGLE OF TILTING PLANE (DEGREES)	67.73329	TILT ROTATION
ANGLE OF DETECTOR TILT IN PLANE (DEGREES)	-0.191528	ANGLE OF TILT

Click on variable to change, or 'O.K.'

The second parameter window contains the re-binning parameters. It makes sense to set the number of bins in the output scan to the resolution in pixel of the image plate (2300 or 3450 in case of Mar345). Further, make sure that the polarization factor is set correctly and that the geometrical correction to the intensities is applied to the data. Confirm the parameters by pressing the <O.K.> button.

Now Fit2D performs the integration of the 2D-pattern of the standard material. Depending on the computing power, this might take a few seconds. The final result should look like this. To save the result for reference and further checks (like Rietveld refinement), press the <OUTPUT> button.
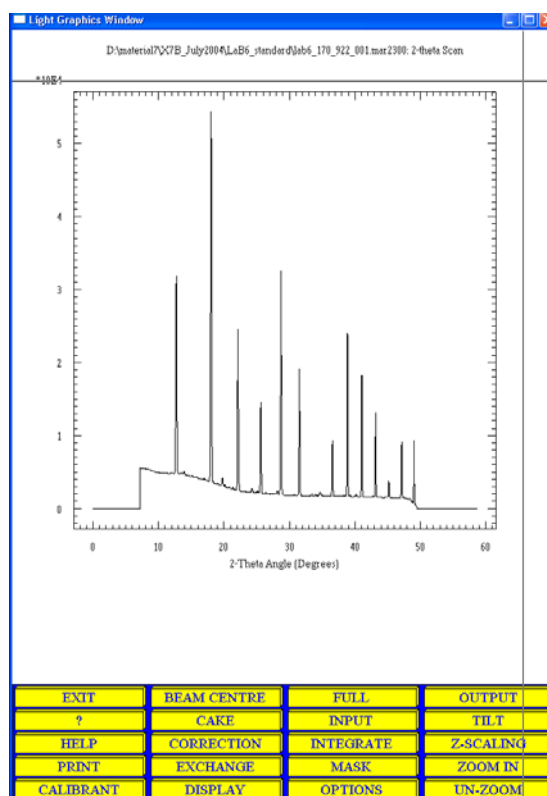
A common format is the plain ASCII CHIPLOT format
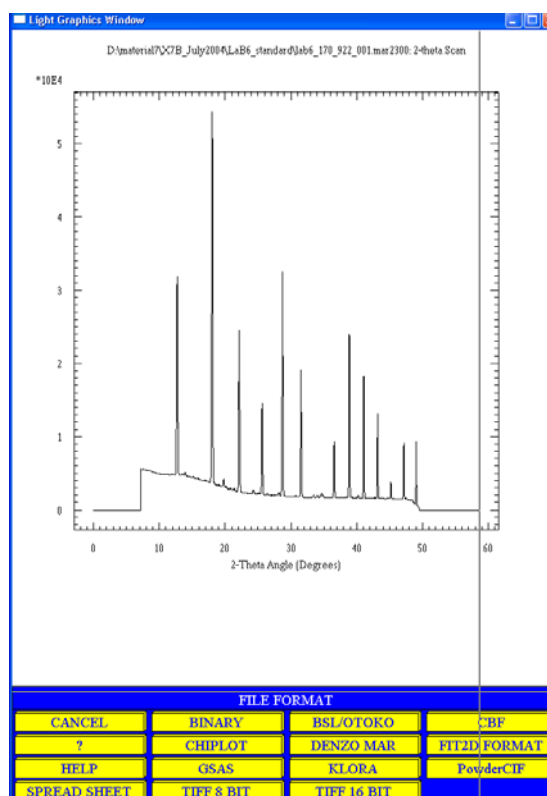
Several parameters allow for compatibility with your favourite data reduction/ viewing program.

3. File series

Once the detector is calibrated, all 2D powder patterns of your *in situ* experiment can be integrated at once. This option is only useful, if the masks necessary to mask out the primary beam stop, single crystal reflections (e.g. from sapphire capillary or diamond), Kossel lines etc. do not change significantly within the series of 2D powder recordings.

After starting the Fit2D program, the button <FILE SERIES> should be pressed.
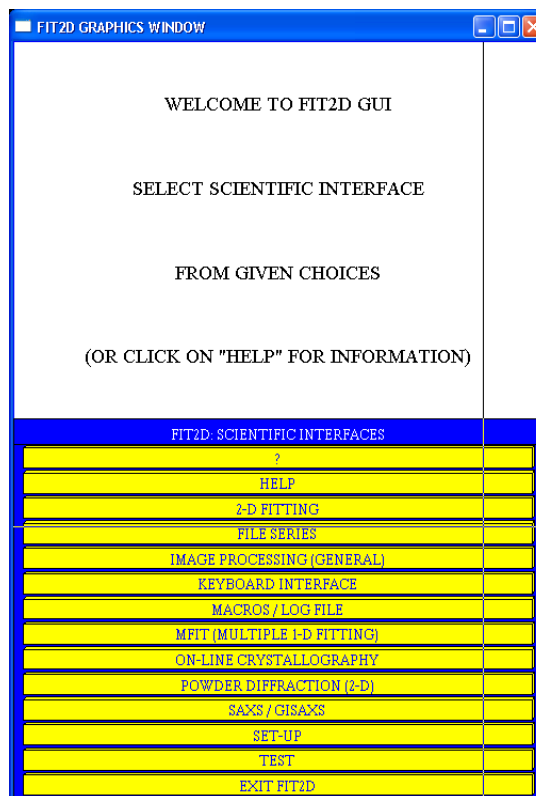
Within the file series menu, the button <INTEGRATE> should be pressed.
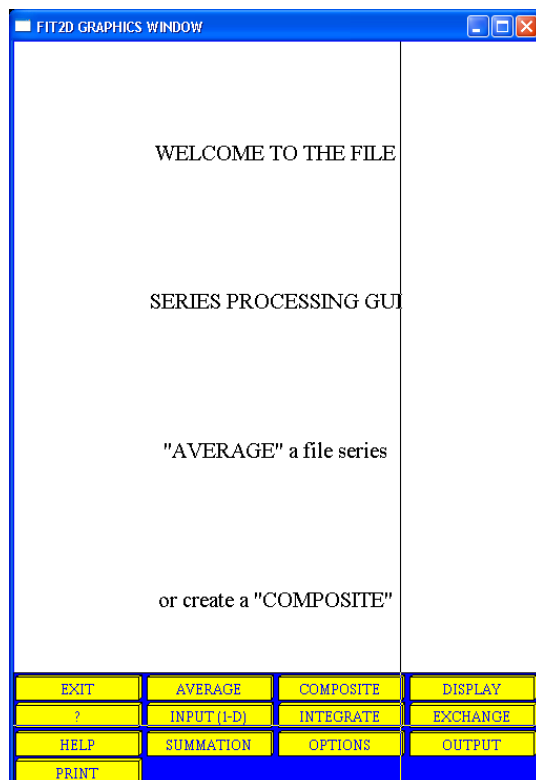
The program now asks for location and name of the first raw file in the series which in our case is IJ65h1h_INDEX_001.mar2300. It is the first set of our data measured at high temperature in a sapphire tube.
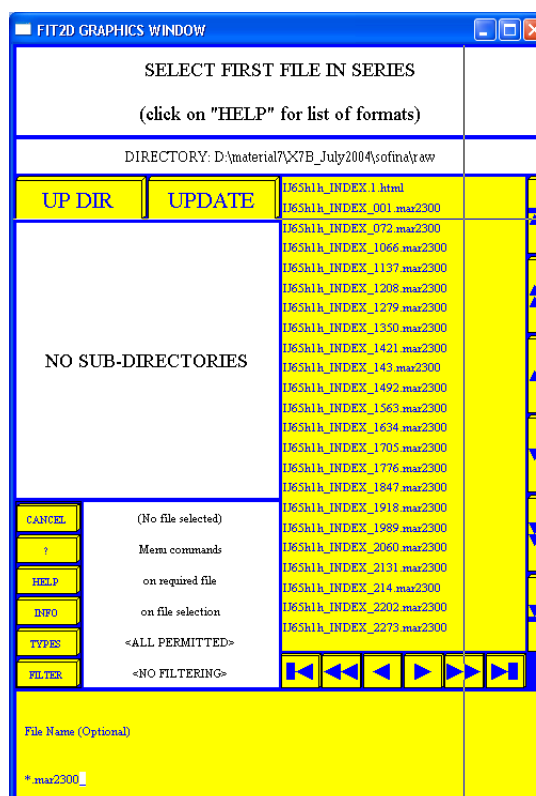
After confirmation of the filename, the 2D powder pattern is displayed. The Bragg reflections of the sapphire single crystal in reflection condition are clearly visible. If this is the correct file, confirm it by pressing the <YES> button.
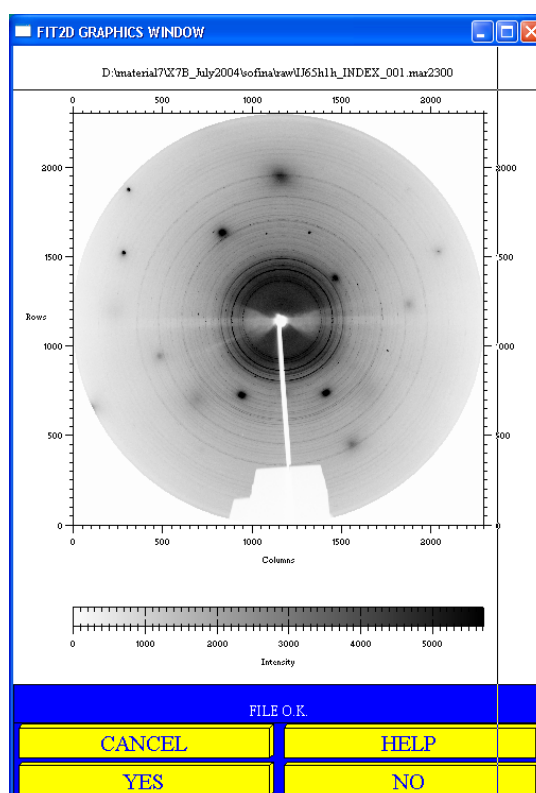
Secondly, the last file of the 2D pattern series is requested. In our case it is IJ65h1h_INDEX_4261.mar2300.
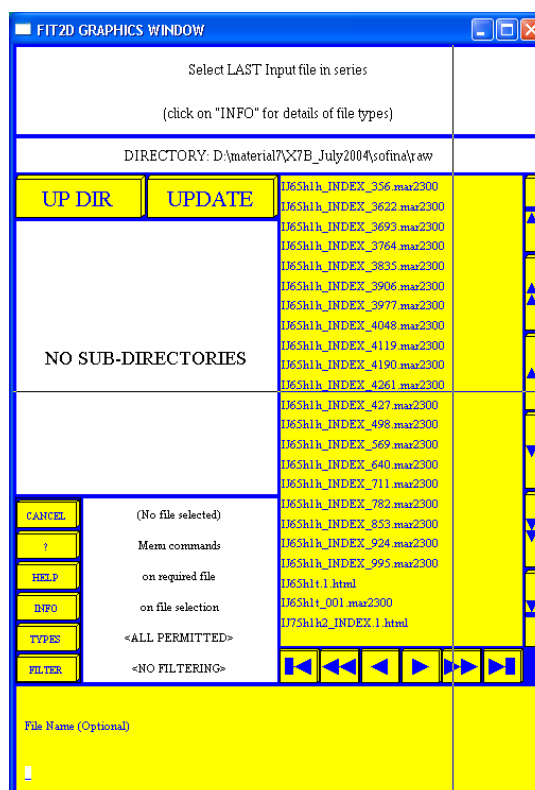
The program finally asks for the increment between the file identifiers, assuming that the last part of the filename contains an ordered number. In our case, the increment between two consecutive filenames is 71.
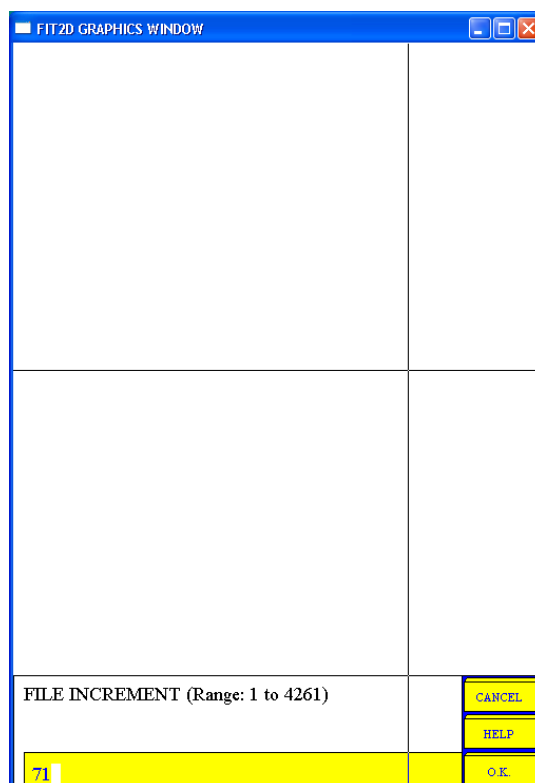
After checking the file names, a menu with control of the detector distortion corrections appears. In case of modern self-calibrating detectors like the Mar345, no external dark current, flat filed and spatial distortion corrections must be applied. The menu is confirmed by pressing the <O.K.> button.

After a short message window, telling the user that the masking window is about to show, the masking window displaying the first data set of the series appears.
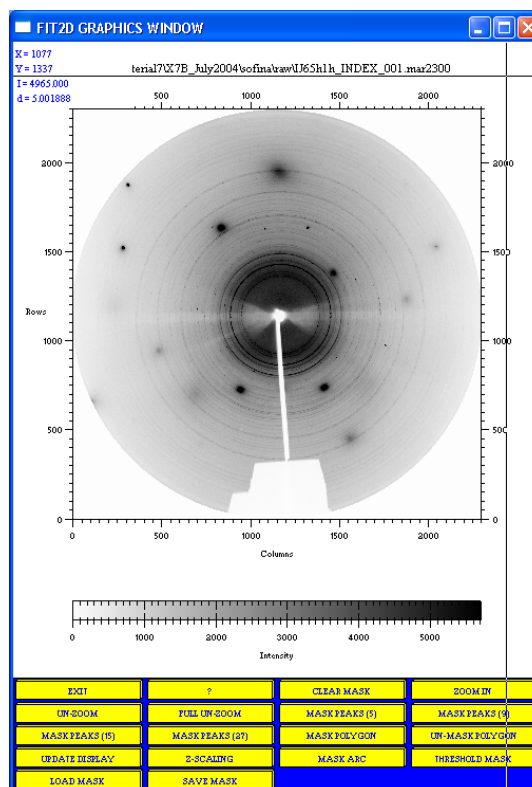
It is recommended to set the masking area with some tolerance to allow for shifts of the aberrations at non-ambient conditions. If the entire frame is subject to integration (which is usually the case), the <FULL UN-ZOOM> button should be pressed before exiting the masking menu by pressing the <EXIT> button.
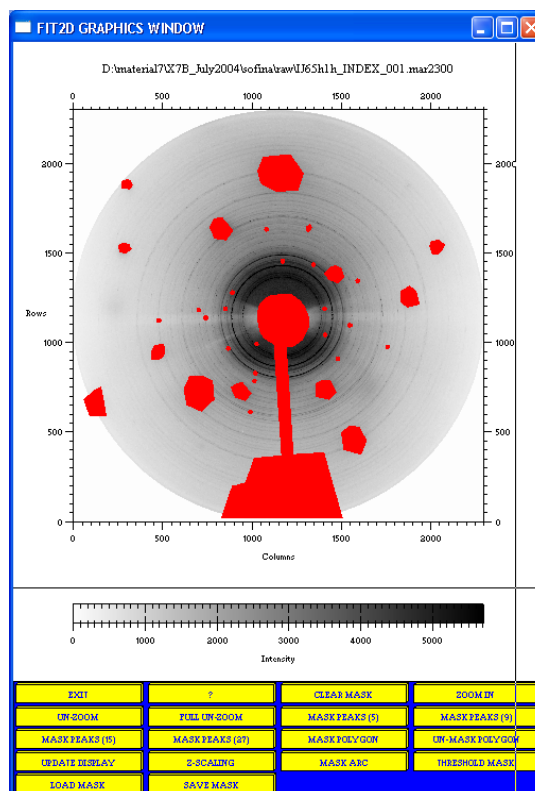
All parameters in the experimental geometry control form should correspond to the appropriate calibration (see above) and should be carefully checked/corrected. Press the <O.K.> button to continue.

The re-binning parameters must be set to the appropriate values of the experiment (see chapter on calibration). It is recommended to "over-bin" according to the image-plate. Press the <O.K.> button to continue.

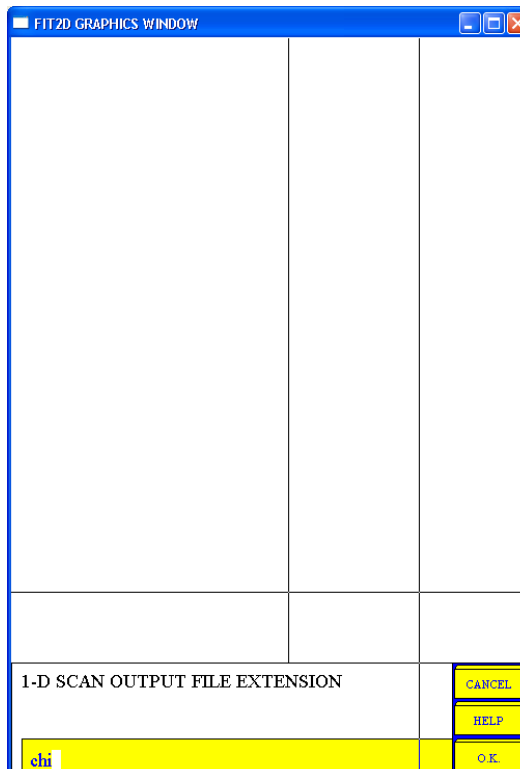Press the <YES> button to automatically save all integrated scans to data files.

Select the file format you want (usually the CHIPLOT format).
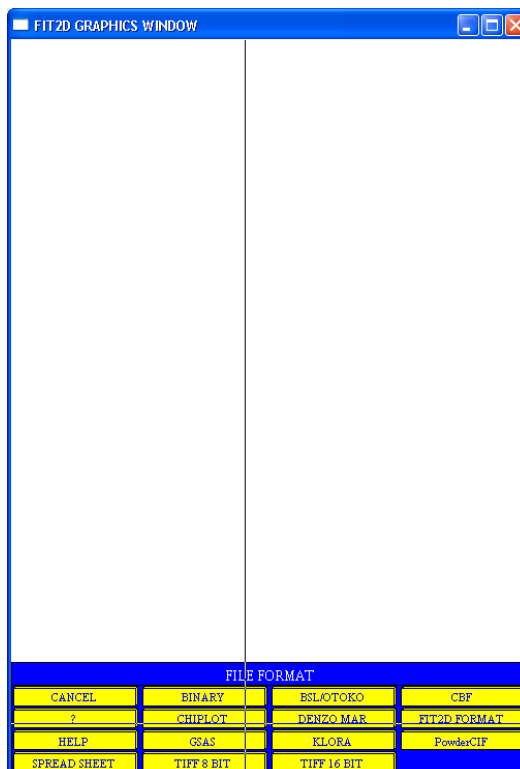
Type in an appropriate extension (usually XY or CHI) for the integrated pattern files. The main part of the name will be taken from the 2D raw files. Then press the <O.K.> button.

Now, all selected 2D powder patterns will be converted to 1D powder patterns. Depending on the resolution of the recording device, the computing power and the number of files, this can take several minutes or more.
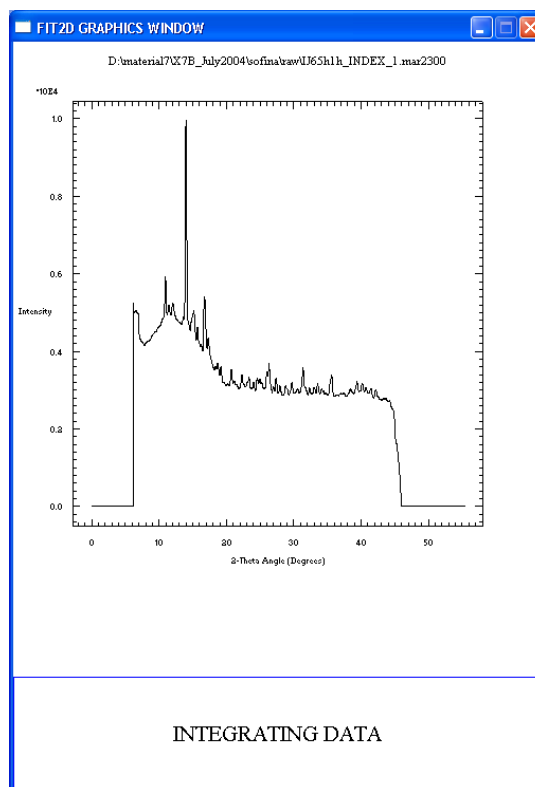
written by R. E. Dinnebier (last change October 8, 2004)