

The CCD detector: how to average radially

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Data retrieval and structure determination from
synchrotron powder diffraction data
Powder XRD Workshop - April 2013

Outline

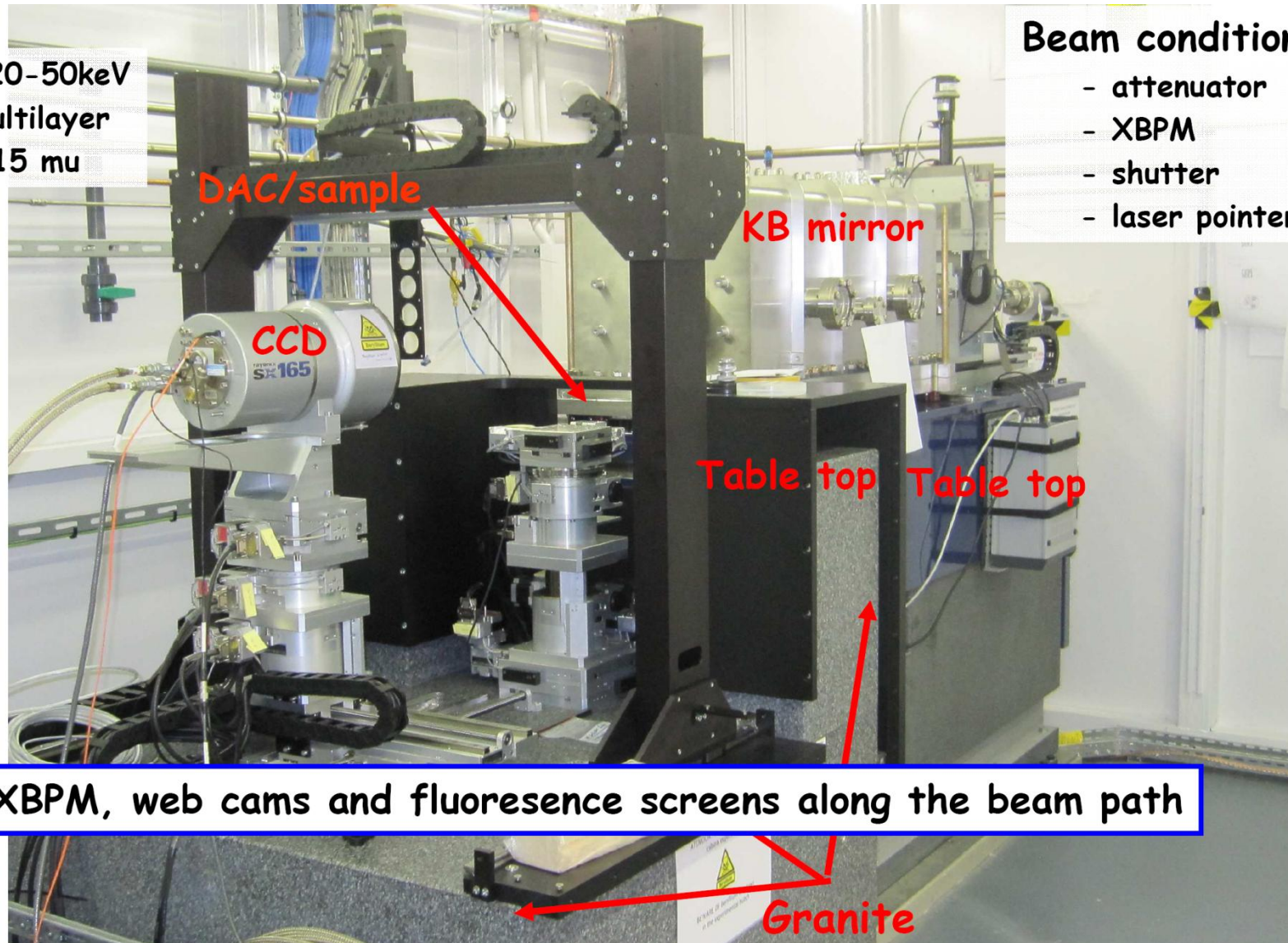
- **Introduction in High Pressure technique**
- **CCD detector**
- **Data Integration of 2D diffraction data**
- **Example-Tutorial**

HP station

Energy 20-50keV
W/Si Multilayer
Spot 5x15 μm

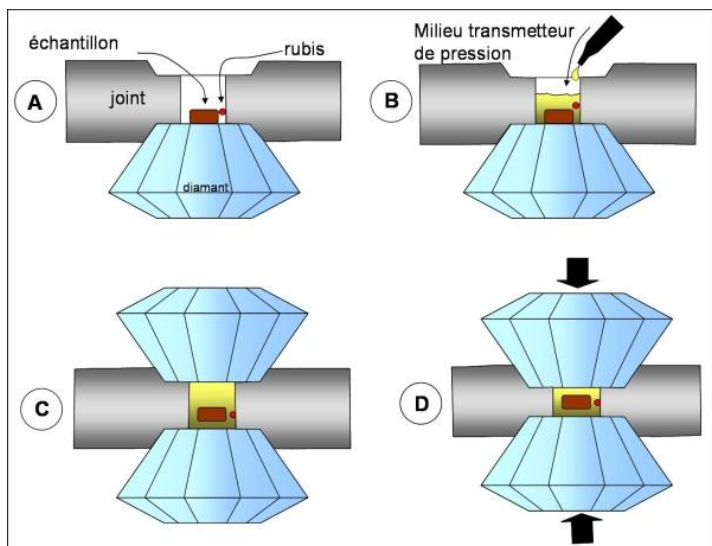
Beam conditioning

- attenuator
- XBPM
- shutter
- laser pointer

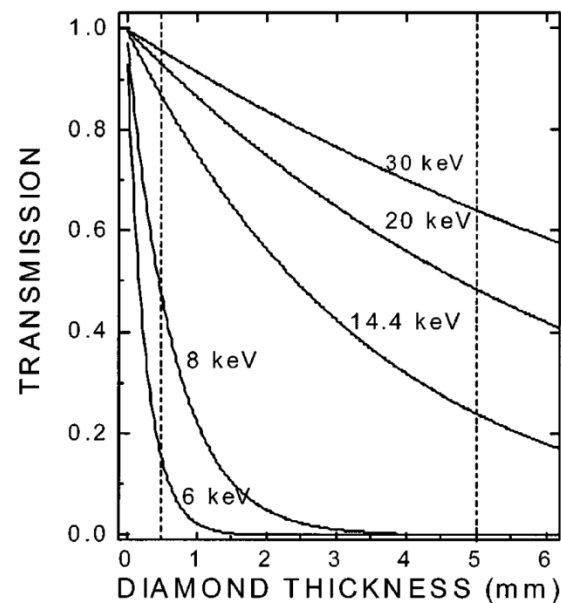


XBPM, web cams and fluorescence screens along the beam path

Diamond anvil cell



<http://www.ens-lyon.fr/LST/HP>



Dadashev et al. Rev.Sci. Instrum. 2001

high compressions $P=F/S$

in situ access for XRD

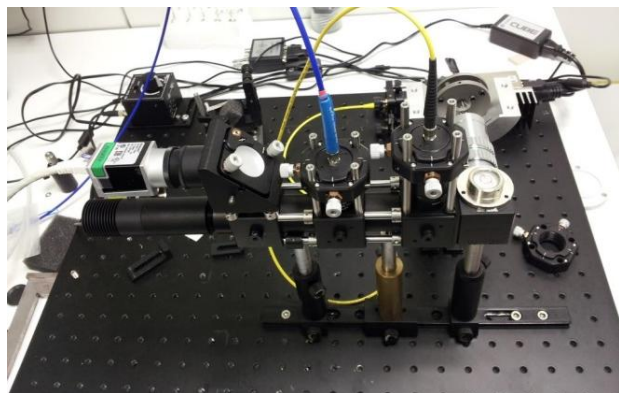
good window above 12 KeV

1 GPa= 10 kbar

1bar = $10^5 \text{ N/m}^2 \text{ (Pa)}$ = 0.9869 atm



HP station

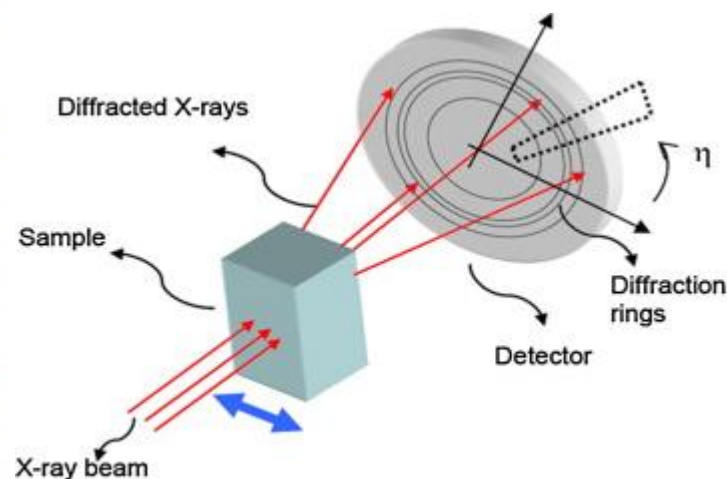
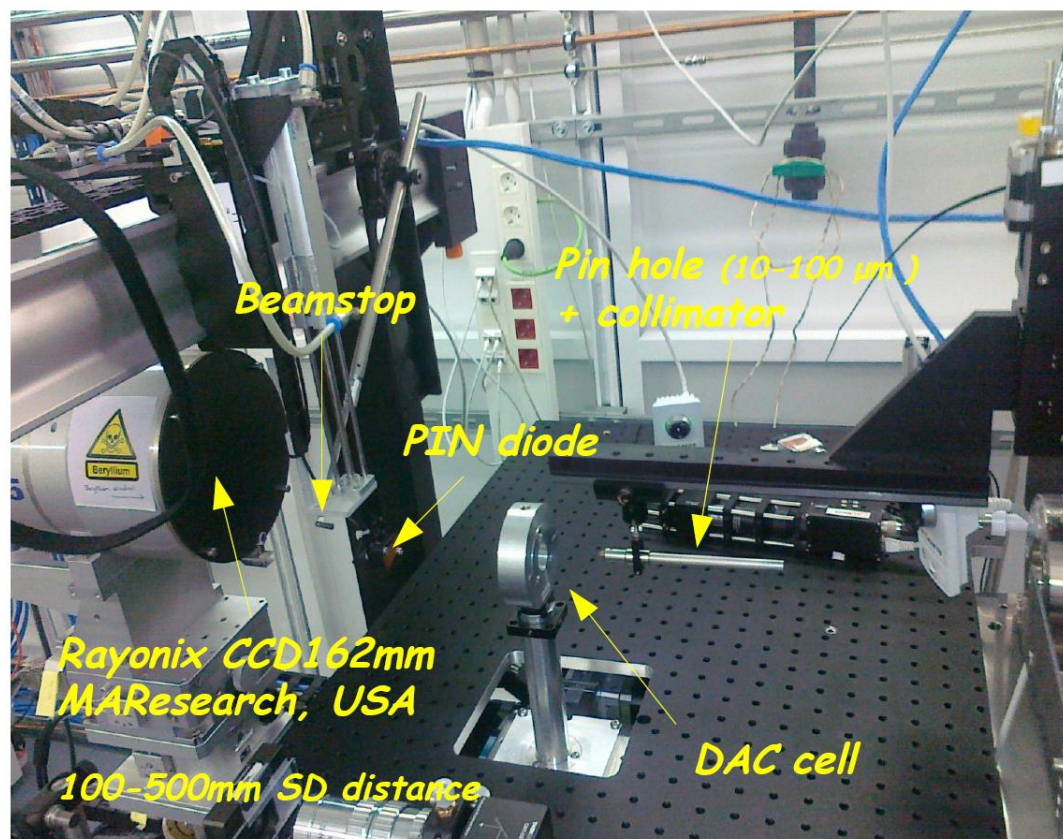


Rayonix CCD SX 165

active area 162 mm, pixel size 79 μm

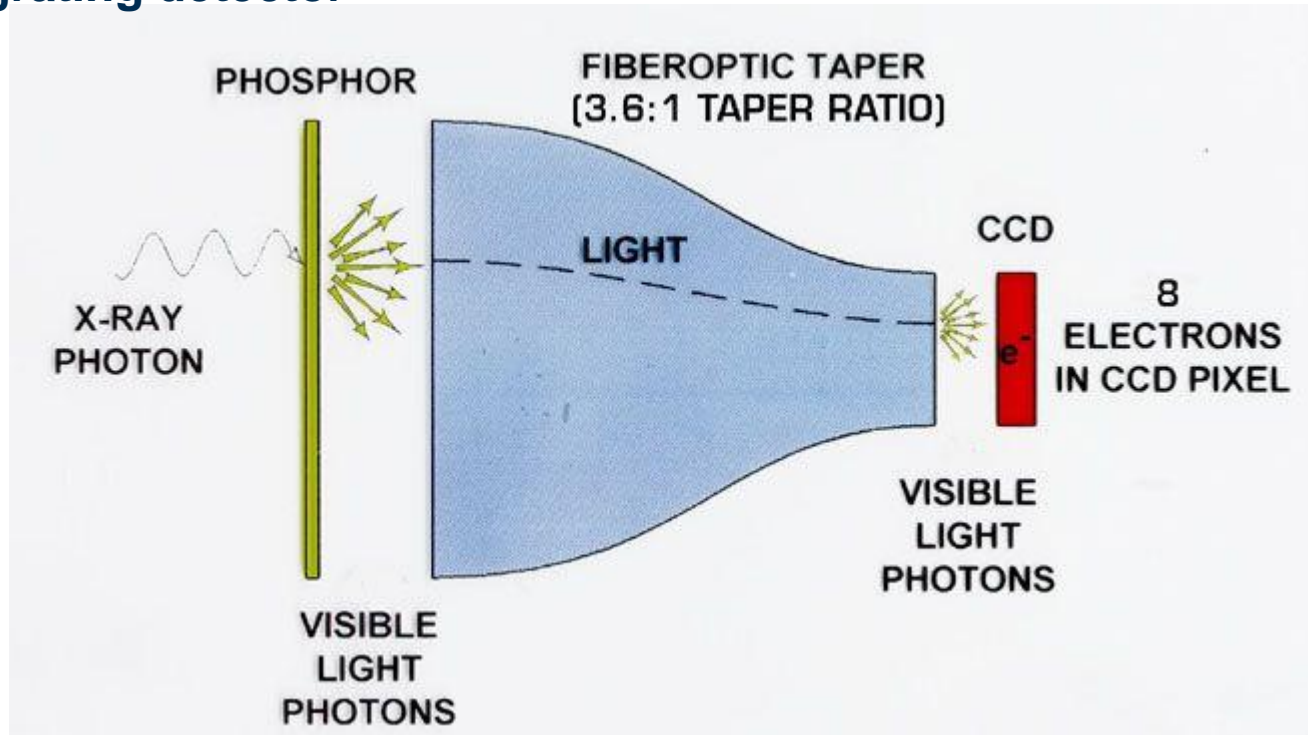
readout 2.5 sec, frame size 2048*2048

In user operation on MSPD since 06.2012



CCD X-ray detector: principles of operation

- X-ray conversion to light by phosphor screen
- Light demagnification onto CCD (CCD chips 61*61mm²)
- CCD sensors (Si/SiO₂/polySi)
- Integrating detector



Software

- **FIT2D: quasi – standard**

<http://www.esrf.eu/computing/scientific/FIT2D>

- **Two2One, Powder 3D IP, MAUD, DataSqueeze**

<http://www.ing.unitn.it/~maud>

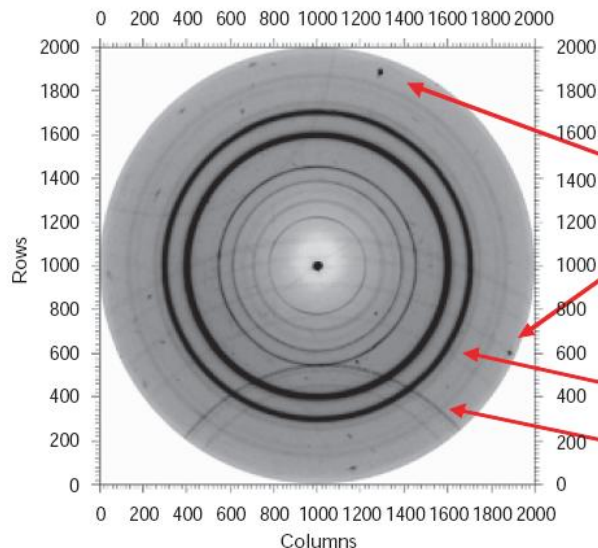
<http://www.datasqueezesoftware.com>

- **XRD2D Scan and GSAS2**

<http://www.ugr.es/~anava/xrd2dscan.htm>

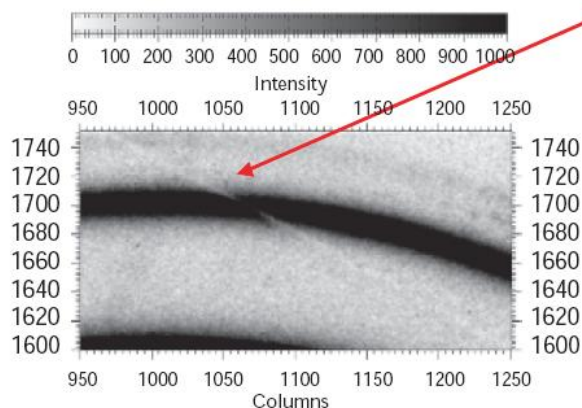
<http://subversion.xor.aps.anl.gov/trac/pyGSAS>

Contamination



2D data may be contaminated
(especially DAC data sets):

- single crystal spots from rubies
used for pressure calibration) or
diamond (used as anvils)
- powder rings from gasket material
- secondary excitations
- Kossel lines



FIT 2D



- platform-independent multi purpose data reduction, visualization and analysis program.
- detector calibration (standard material LaB_6) > sample-to-detector distance and tilt angle.
- integration of powder diffraction data from 2D detectors to 1-D scans.

Data input

Light Graphics Window

DIMENSIONS OF PROGRAM ARRAYS
(need to be big enough to store
and work on data)

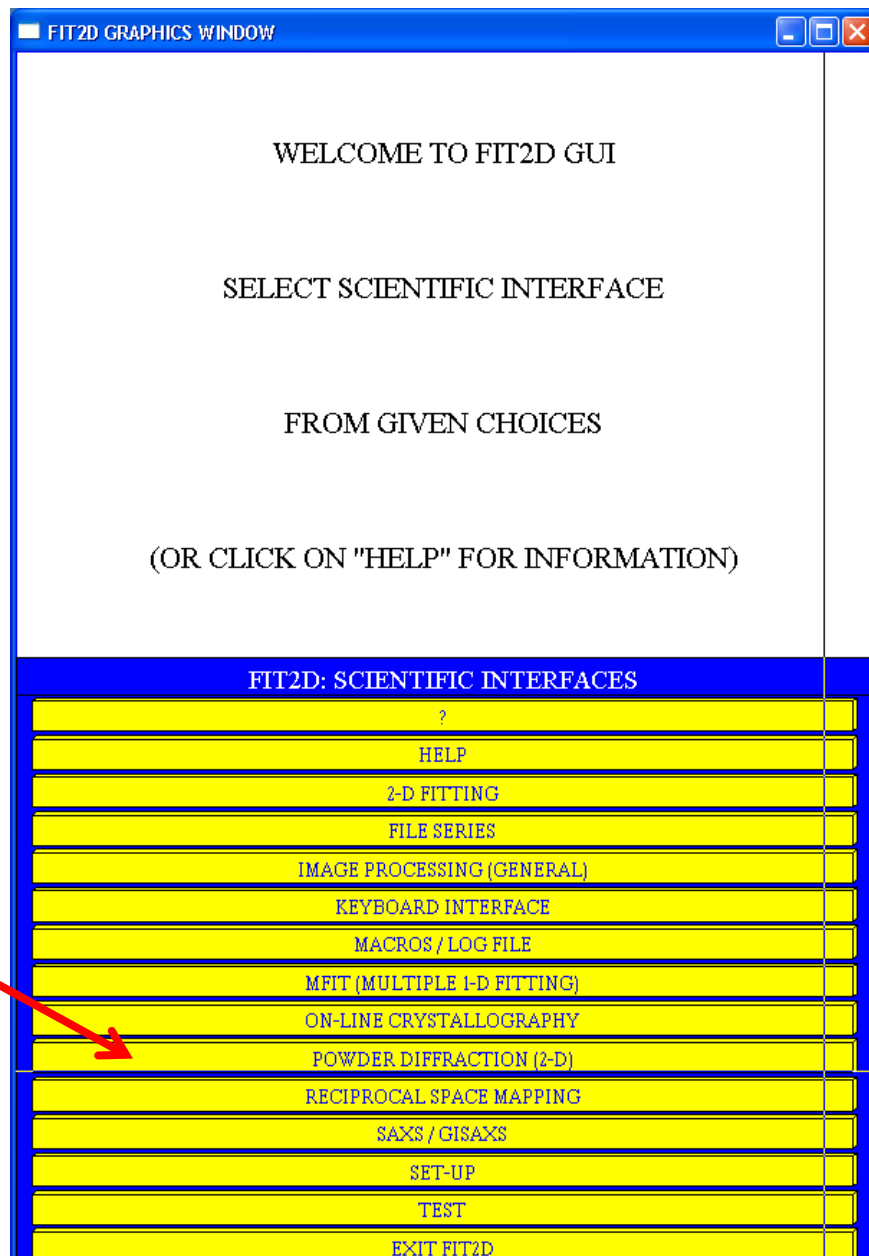
O.K. CANCEL ? HELP INFO

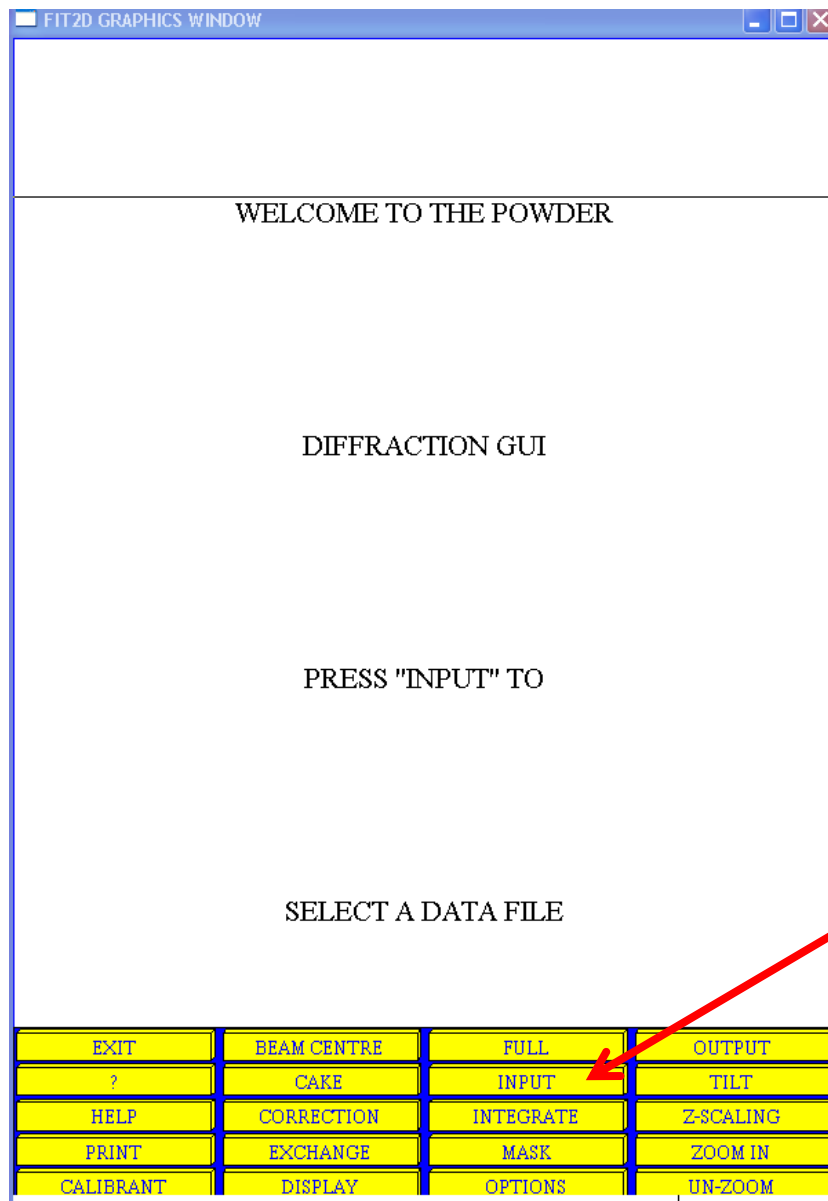
DESCRIPTIONS	VALUES	CHANGE
FIRST DIMENSION OF ARRAYS	2048	X-DIMENSION
SECOND DIMENSION OF ARRAYS	2048	Y-DIMENSION
CREATE MEMORY ARRAYS	YES	MEMORY
CREATE VARIANCE ARRAYS	NO	VARIANCES

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

• dimensions of pixel arrays:

X- and Y- dimension: 2048*2048 (Rayonix CCD)





FIT2D GRAPHICS WINDOW

CONTROL OF DETECTOR

DISTORTION CORRECTIONS

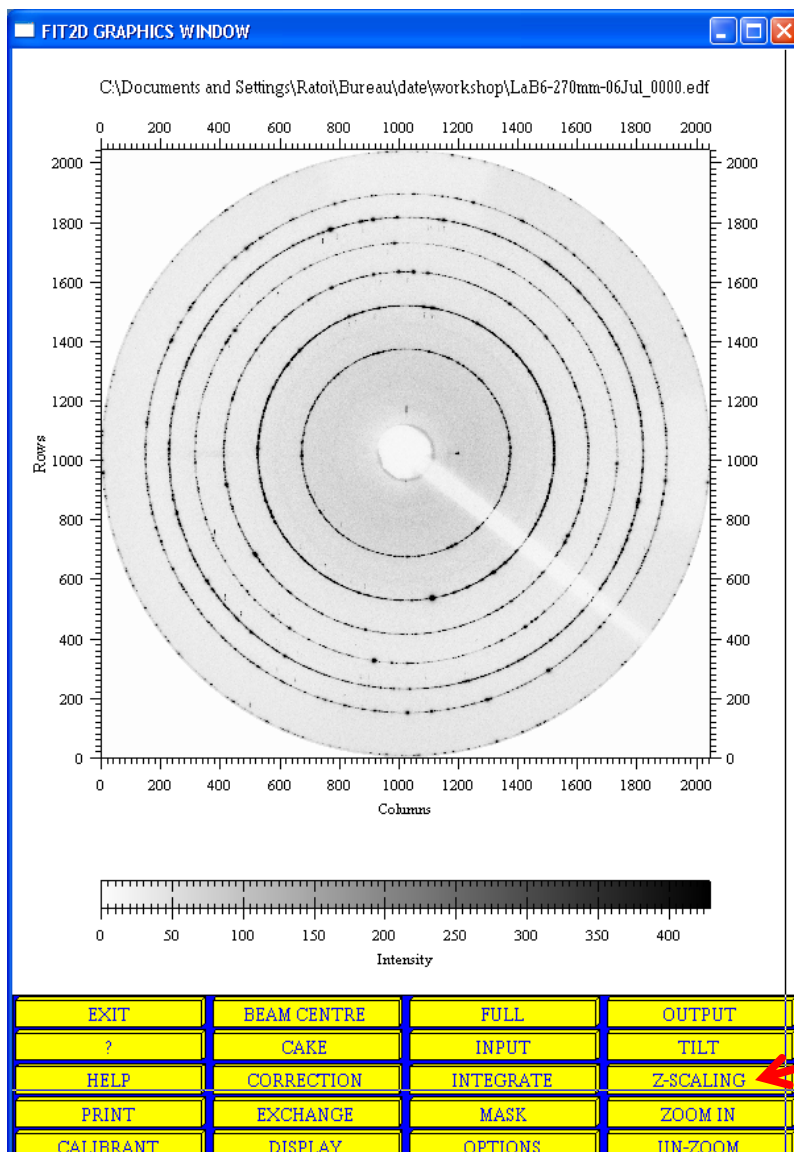
O.K. CANCEL ? HELP INFO

DESCRIPTIONS	VALUES	CHANGE
SUBTRACT DARK CURRENT IMAGE	NO	DARK CURRENT
NAME OF DARK CURRENT FILE	dark_current.bin	DC FILE
APPLY FLAT FIELD CORRECTION	NO	FLAT FIELD
NAME OF FLAT-FIELD FILE	flat_field.bin	FF FILE
APPLY SCALING AFTER FLAT FIELD CORRECTION	NO	FF SCALE
FLAT FIELD MULTIPLIER TO APPLY	1000.000	FF MULTIPLIER
APPLY SPATIAL DISTORTION CORRECTION	NO	SPATIAL DIS.
NAME OF SPATIAL DISTORTION FILE	spatial.spline	SD FILE

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

self calibrating detector

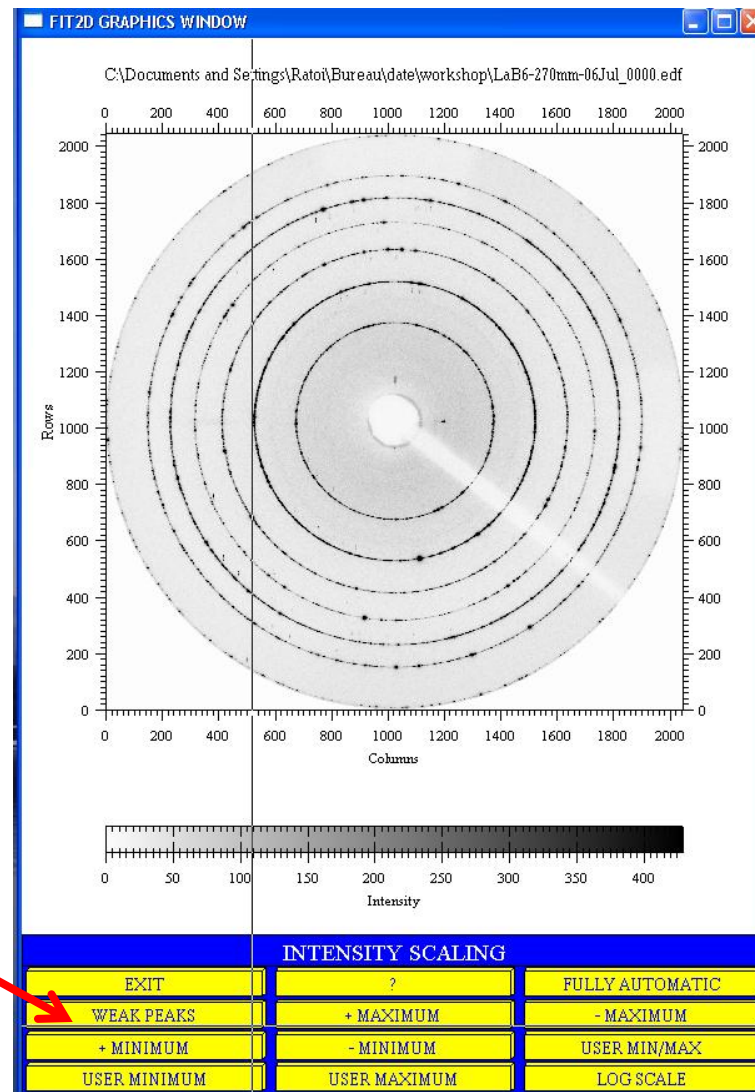
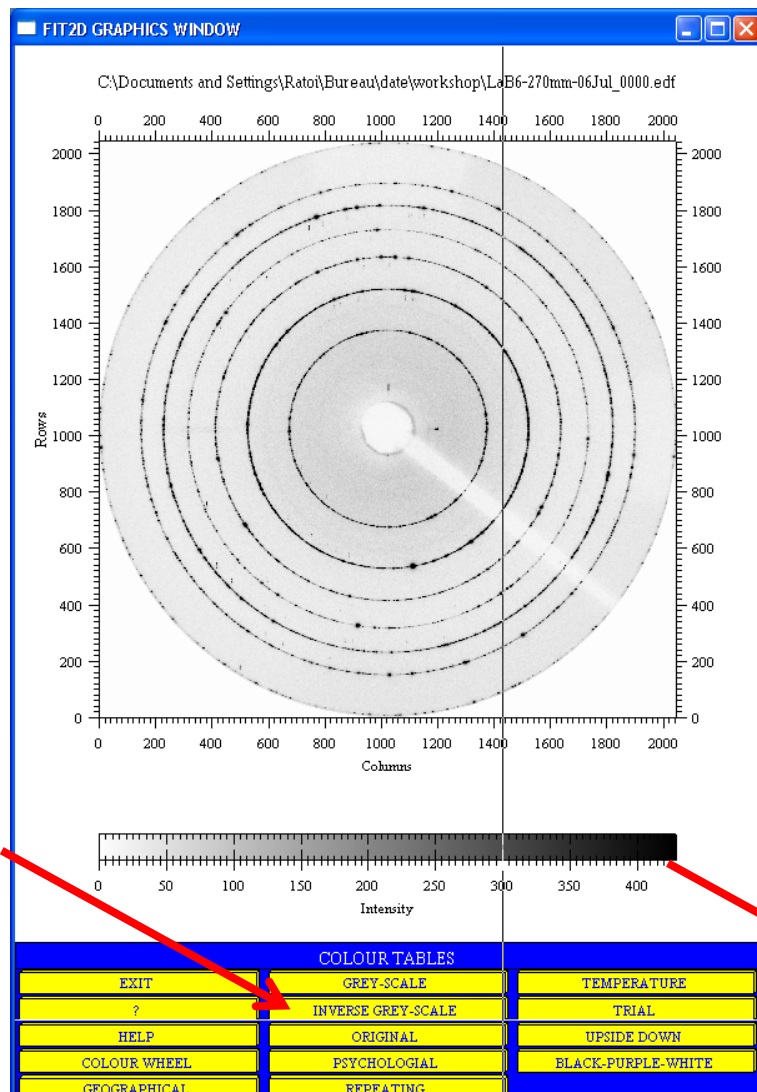
Scaling



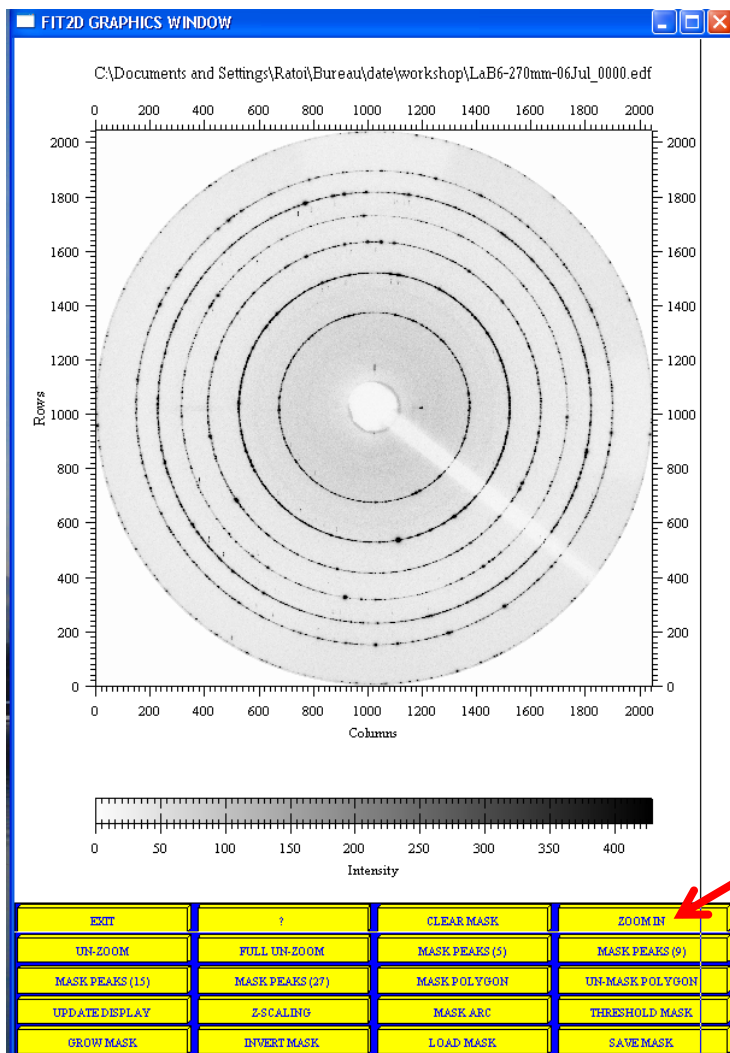
<inverse scaling>

<scaling to weak peaks>

<OPTIONS> → <COLORS> → <INVERSE GREY-SCALE>



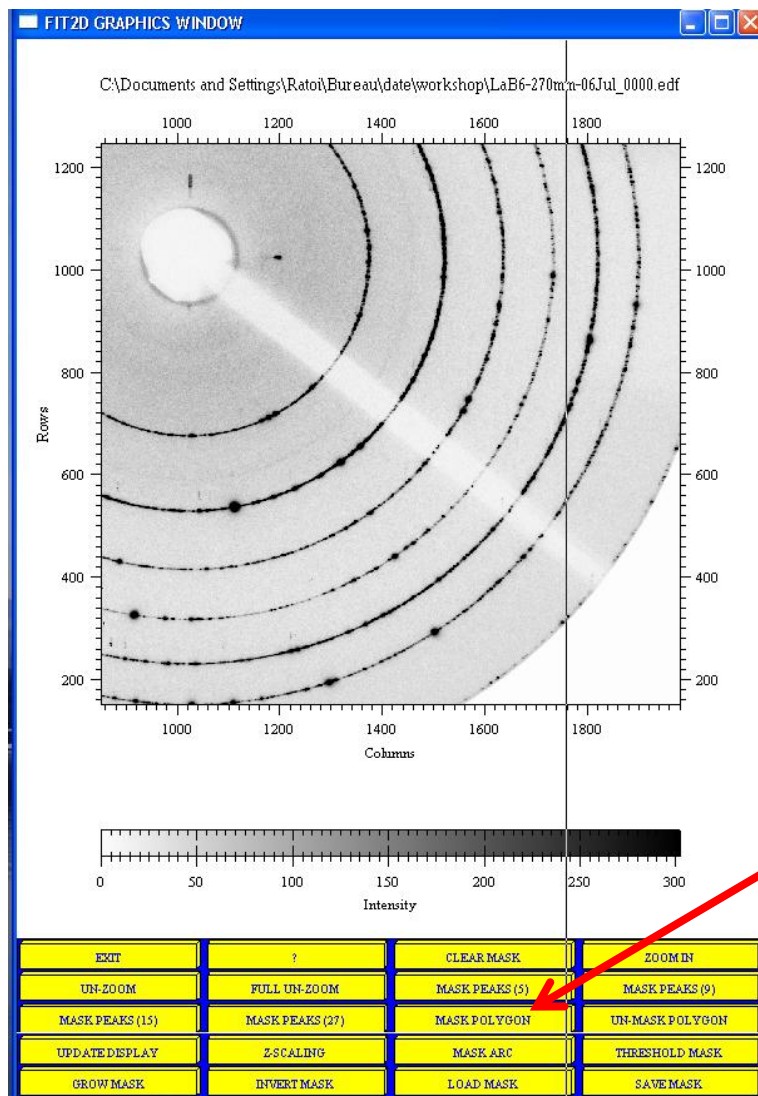
Contamination-Mask



mask unwanted scattering

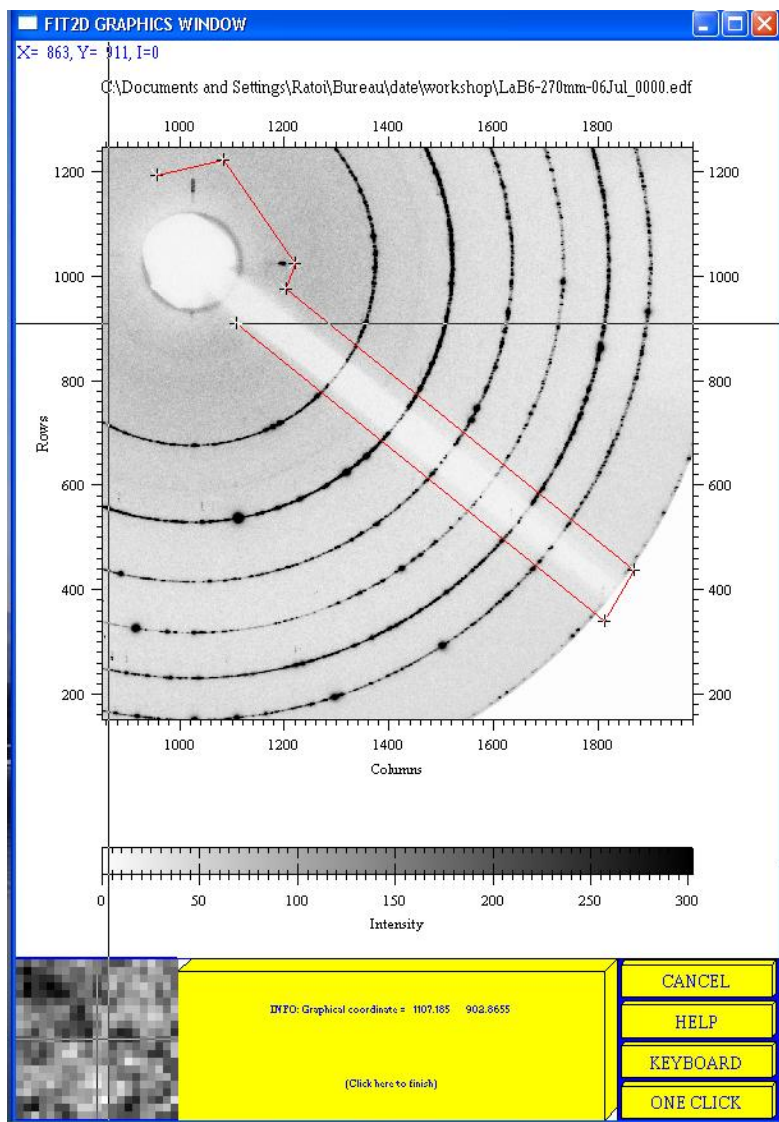
<ZOOM IN>

Mask



<MASK POLYGON>

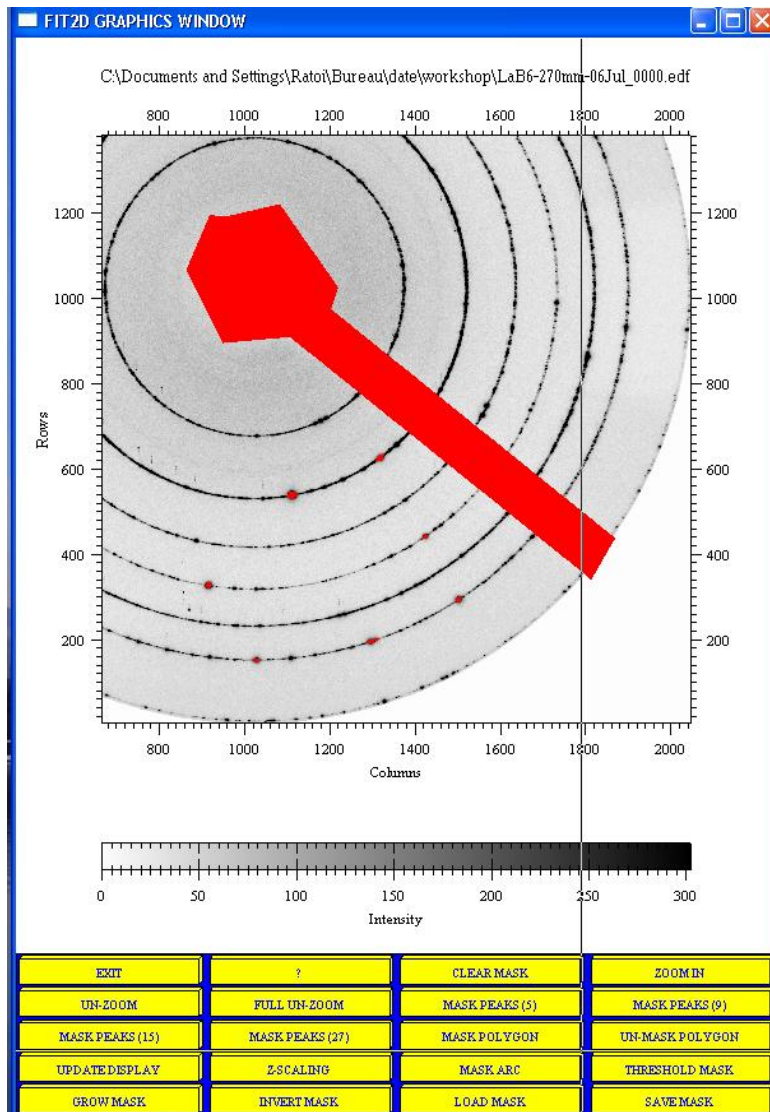
Mask



<ONE CLICK>

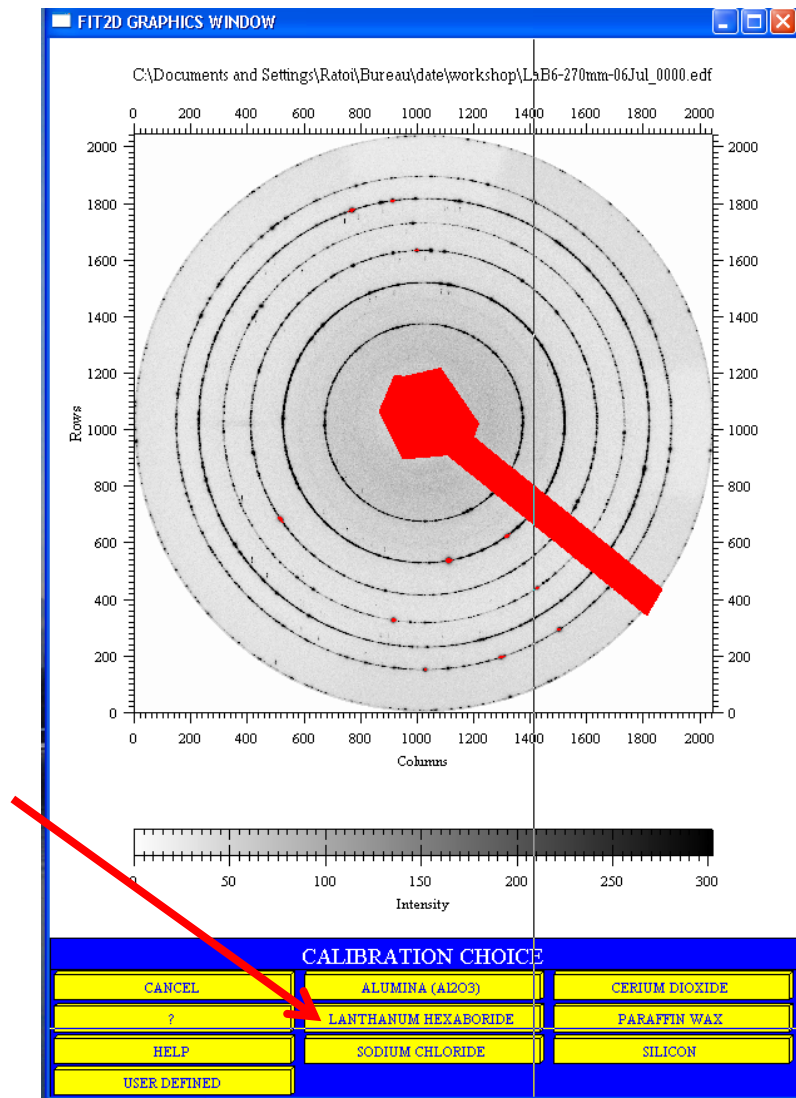
<TWO CLICK> spy glass

Mask



<FULL UN-ZOOM>

Calibration



<CALIBRANT>

Calibration

FIT2D GRAPHICS WINDOW

CALIBRANT PATTERN REFINEMENT

OF DISTANCE WAVELENGTH ETC.

O.K. CANCEL ? HELP INFO

DESCRIPTIONS	VALUES	CHANGE
SAMPLE TO DETECTOR DISTANCE(MM)(STARTING)	270.0000	DISTANCE
WAVELENGTH(ANGSTROMS) (STARTING)	0.424600	WAVELENGTH
SIZE OF HORIZONTAL PIXELS (MICRONS)	79.00000	X-PIXEL SIZE
SIZE OF VERTICAL PIXELS (MICRONS)	79.00000	Y-PIXEL SIZE
NUMBER OF AZIMUTHAL SECTIONS	90	ANGULAR SECTIONS
REJECT OUT-LYING POSITIONS AND RE-REFINE	YES	REJECT OUTLIERS
REJECT LIMIT FROM IDEAL (STANDARD DEVIATIONS)	4.000000	REJECT LIMIT
OUTPUT FULL INFORMATION	YES	FULL INFO
REFINE X/Y BEAM CENTRE	YES	REFINE BEAM X/Y
REFINE SAMPLE TO DETECTOR DISTANCE	YES	REFINE DISTANCE
REFINE X-RAY WAVELENGTH	NO	REFINE WAVELENGTH
REFINE DETECTOR NON-ORTHOGONALITY	YES	REFINE TILT
FIT INTERMEDIATE NUMBER OF RINGS	NO	EXTRA ITERATIONS

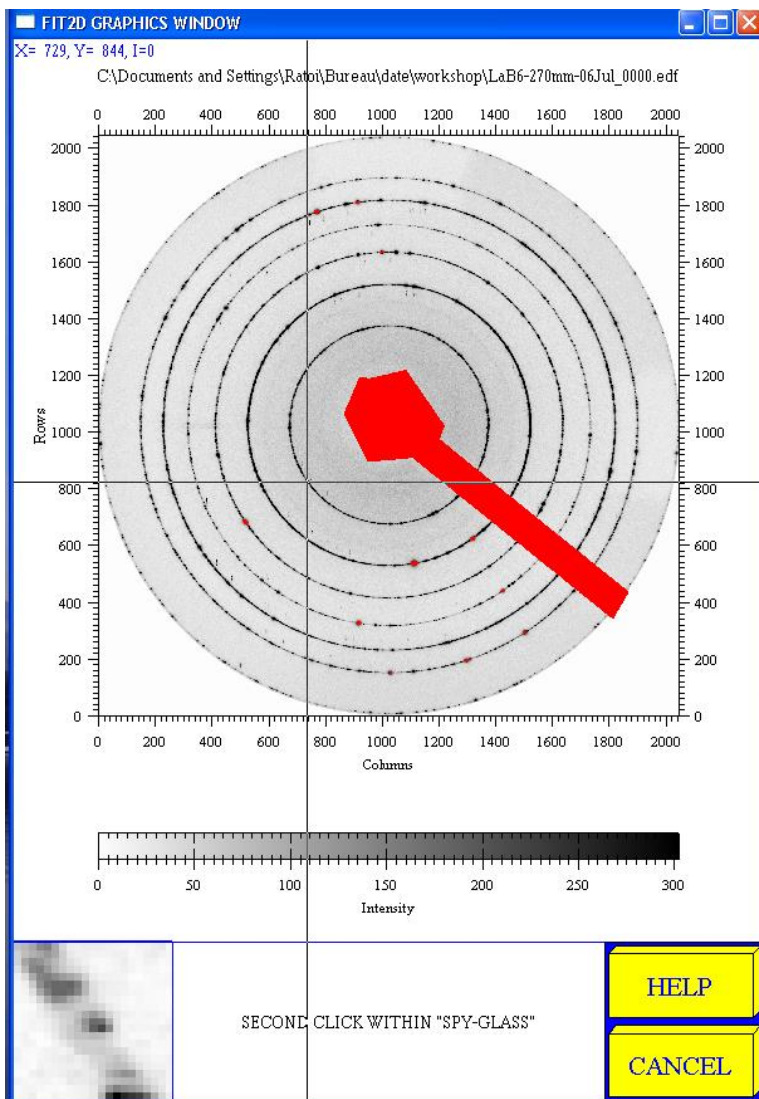
Click on variable to change, or 'O.K.'

sample to detector distance

wavelength (no refinement)

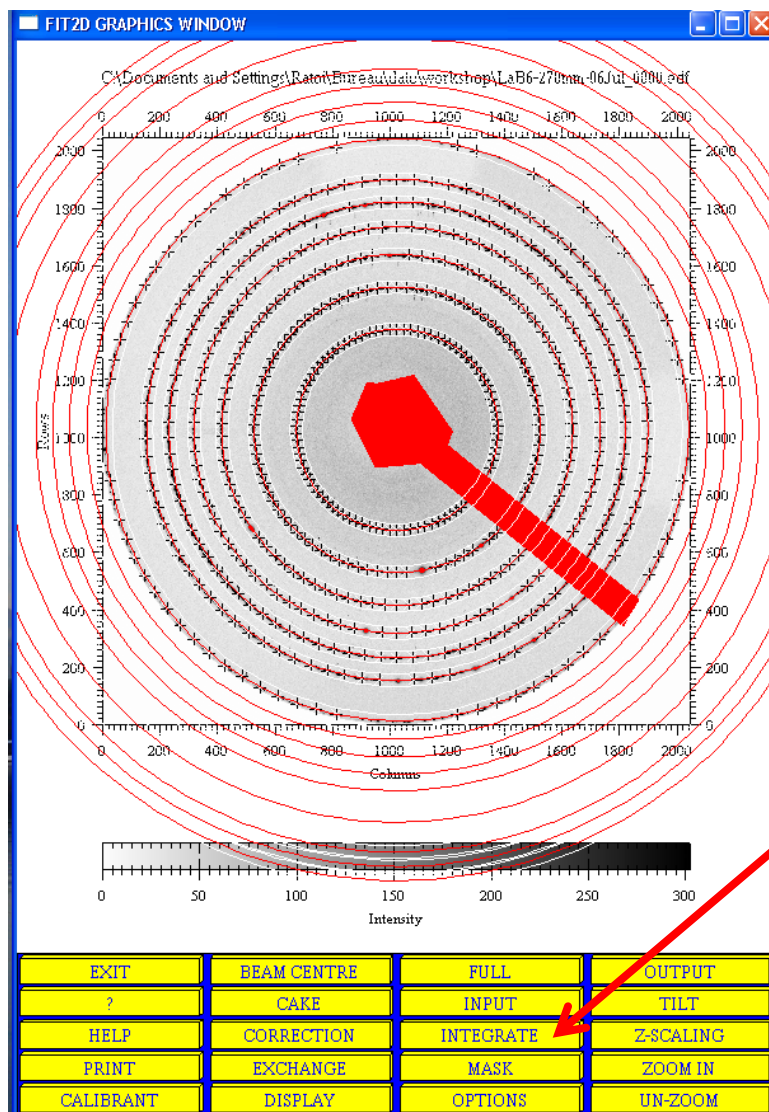
size of pixels

Calibration



<TWO CLICK>

Integration



<INTEGRATE>

Integration

FIT2D GRAPHICS WINDOW

EXPERIMENTAL GEOMETRY

CONTROL FORM

O.K. CANCEL ? HELP INFO

DESCRIPTIONS	VALUES	CHANGE
SIZE OF HORIZONTAL PIXELS (MICRONS)	79.00000	X-PIXEL SIZE
SIZE OF VERTICAL PIXELS (MICRONS)	79.00000	Y-PIXEL SIZE
SAMPLE TO DETECTOR DISTANCE (MM)	269.3411	DISTANCE
WAVELENGTH (ANGSTROMS)	0.424600	WAVELENGTH
X-PIXEL COORDINATE OF DIRECT BEAM	1025.381	X-BEAM CENTRE
Y-PIXEL COORDINATE OF DIRECT BEAM	1027.480	Y-BEAM CENTRE
ROTATION ANGLE OF TILTING PLANE (DEGREES)	-103.7733	TILT ROTATION
ANGLE OF DETECTOR TILT IN PLANE (DEGREES)	0.605970	ANGLE OF TILT

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

Integration

FIT2D GRAPHICS WINDOW

CONTROL OF RADIAL, 2-THETA, OR Q

SCAN RE-BINNING PARAMETERS

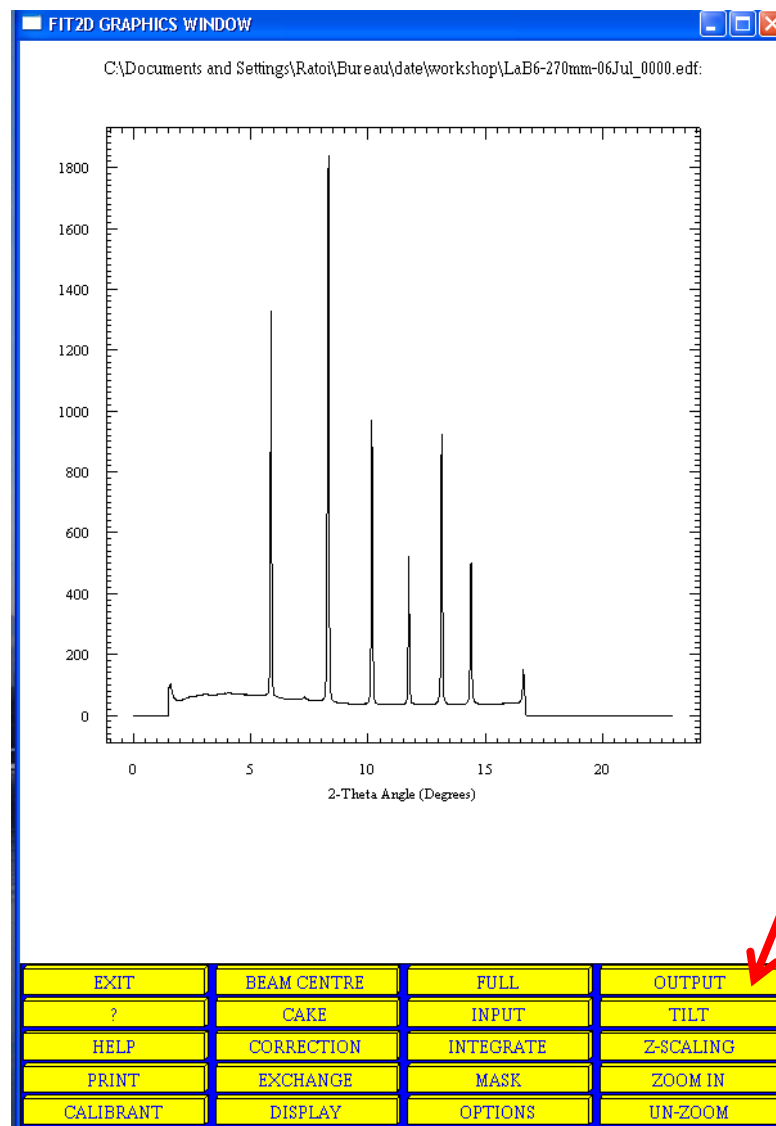
O.K. CANCEL ? HELP INFO

DESCRIPTIONS	VALUES	CHANGE
SCAN TYPE (D, RADIAL, 2-THETA, Q-SPACE)	2-THETA	SCAN TYPE
INTENSITY CONSERVATION	NO	CONSERVE INT.
APPLY POLARISATION CORRECTION	YES	POLARISATION
POLARISATION FACTOR	0.990000	FACTOR
GEOMETRICAL CORRECTION TO INTENSITIES	YES	GEOMETRY COR
MAXIMUM 2-THETA ANGLE OF SCAN (DEGREES)	23.04246	MAX. ANGLE
NUMBER OF BINS IN OUTPUT SCAN	1450	SCAN BINS
MAXIMUM FOR D-SPACINGS SCANS (ANGSTROMS)	20.00000	MAX. D-SPACING

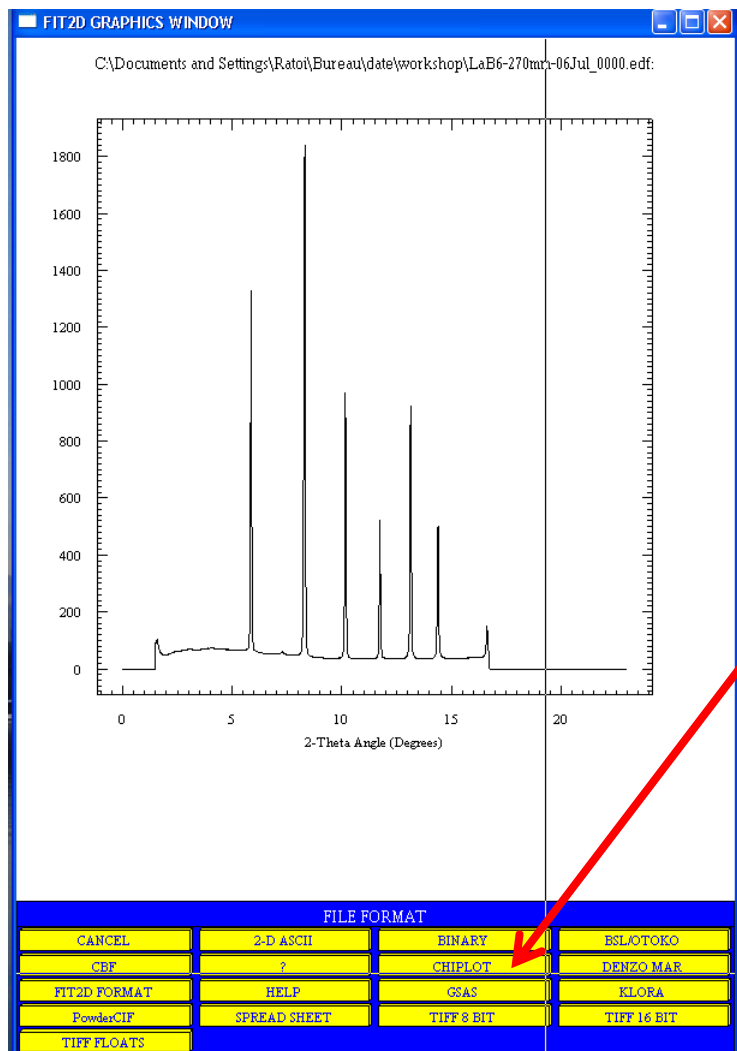
Click on variable to change, or 'O.K.'

**SET TO THE RESOLUTION
OF THE DETECTOR (2048)**

Output



Output



Light Graphics Window

OUTPUT FILE NAME AND
ROW OR COLUMN TO OUTPUT

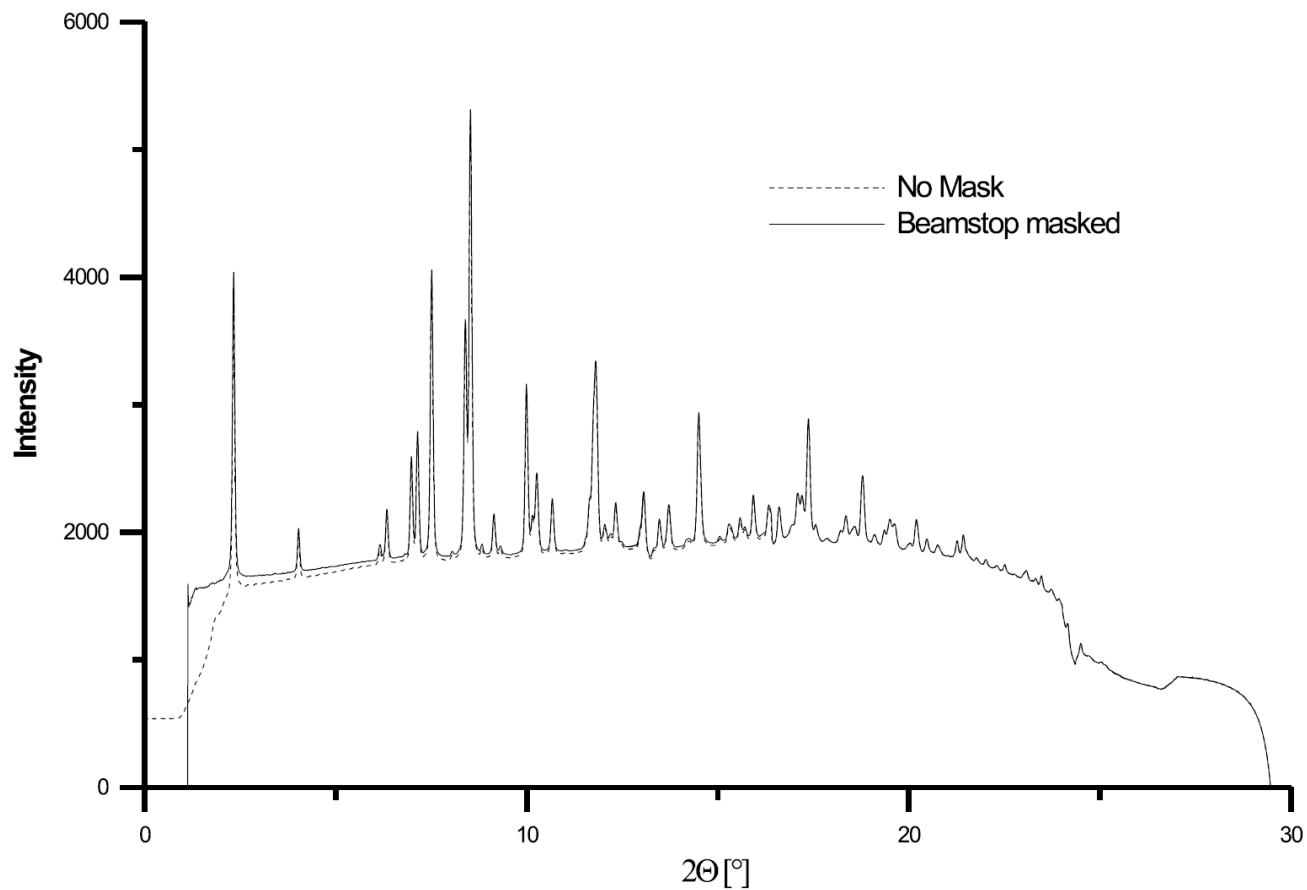
O.K. CANCEL ? HELP INFO

DESCRIPTIONS	VALUES	CHANGE
SELECT FILE TO CONTAIN DATA	ureau\datework shop\LaB6-270mm -06Jul_0000.chi	FILE NAME
OUTPUT ROWS (YES) OR COLUMNS (NO)	YES	OUTPUT ROWS
NUMBER OF ROW TO OUTPUT	1	ROW NUMBER
NUMBER OF COLUMN TO OUTPUT	1	COLUMN NUMBER

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

Mask

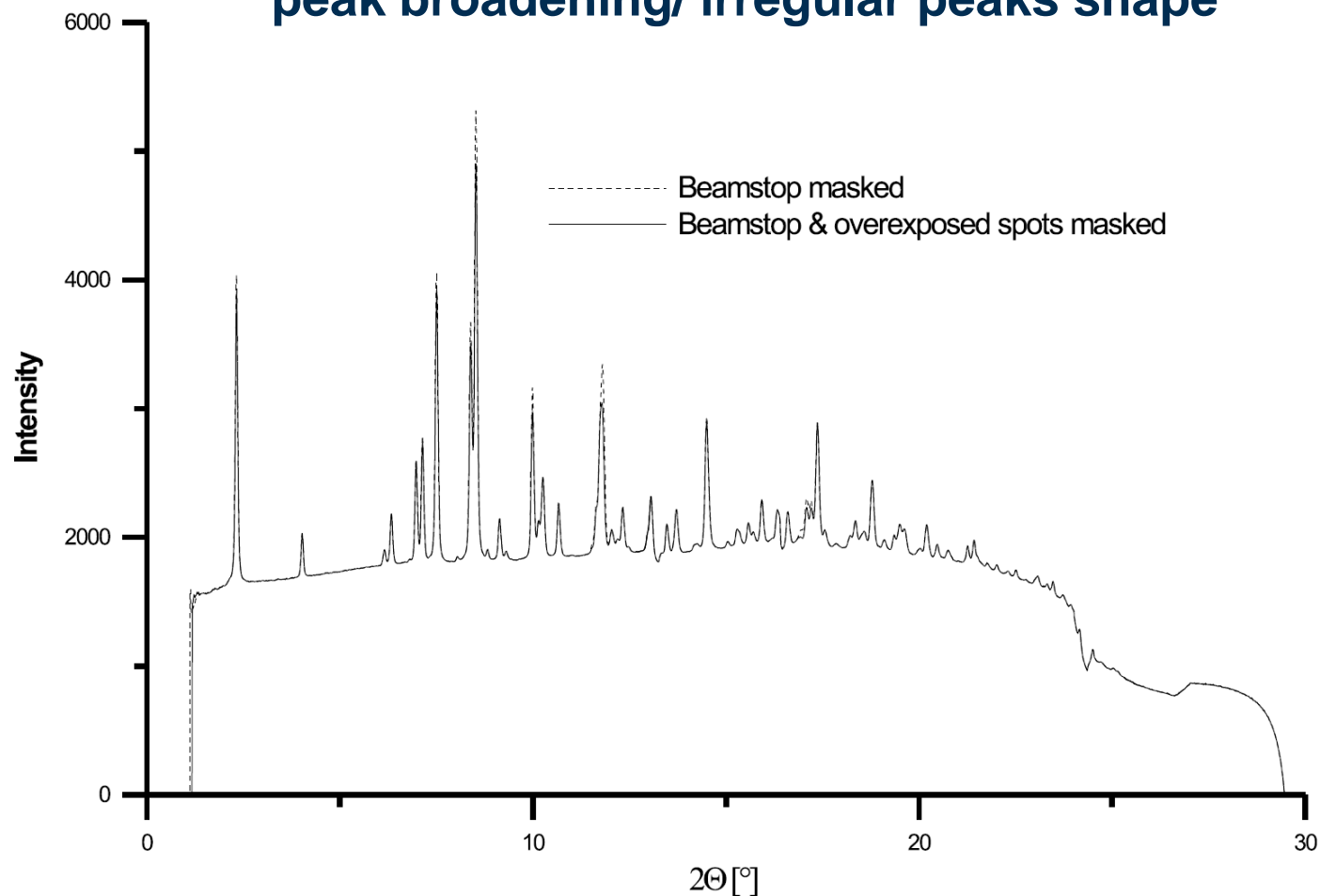
profile fitting → straight bkg



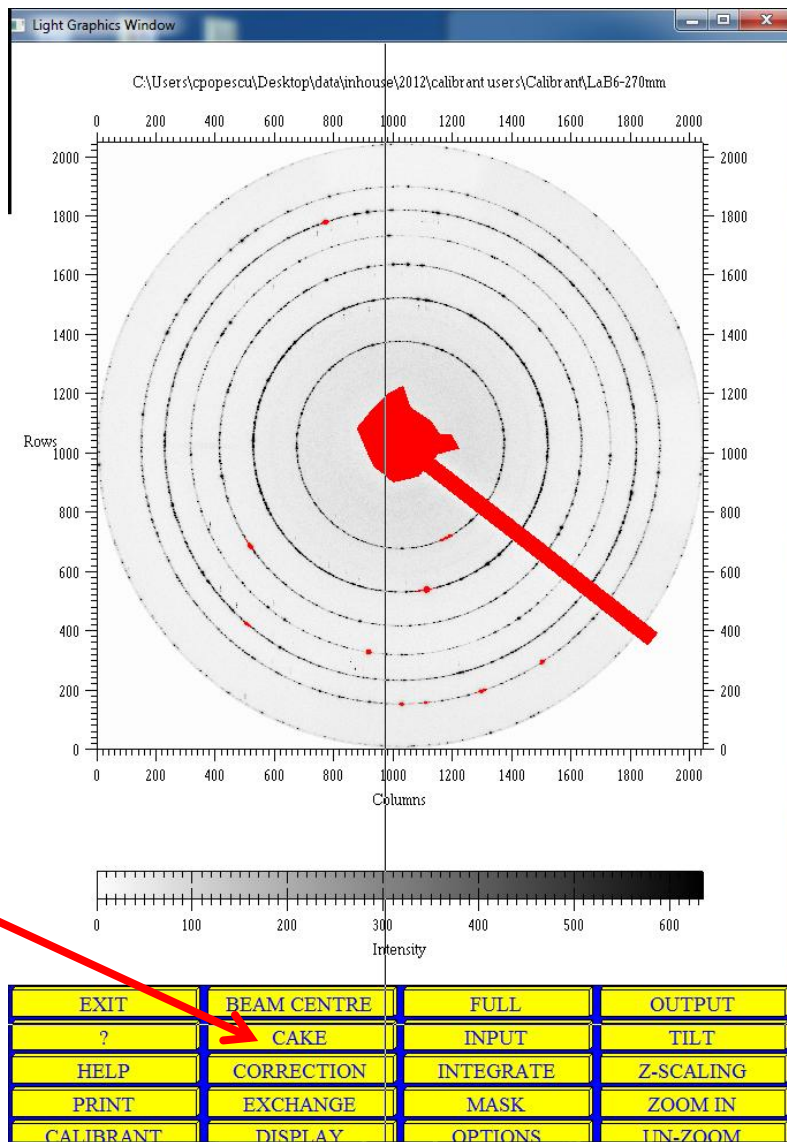
Mask

source of systematic errors

peak broadening/ irregular peaks shape



Calibration check: cake



Start Azimuth: 0

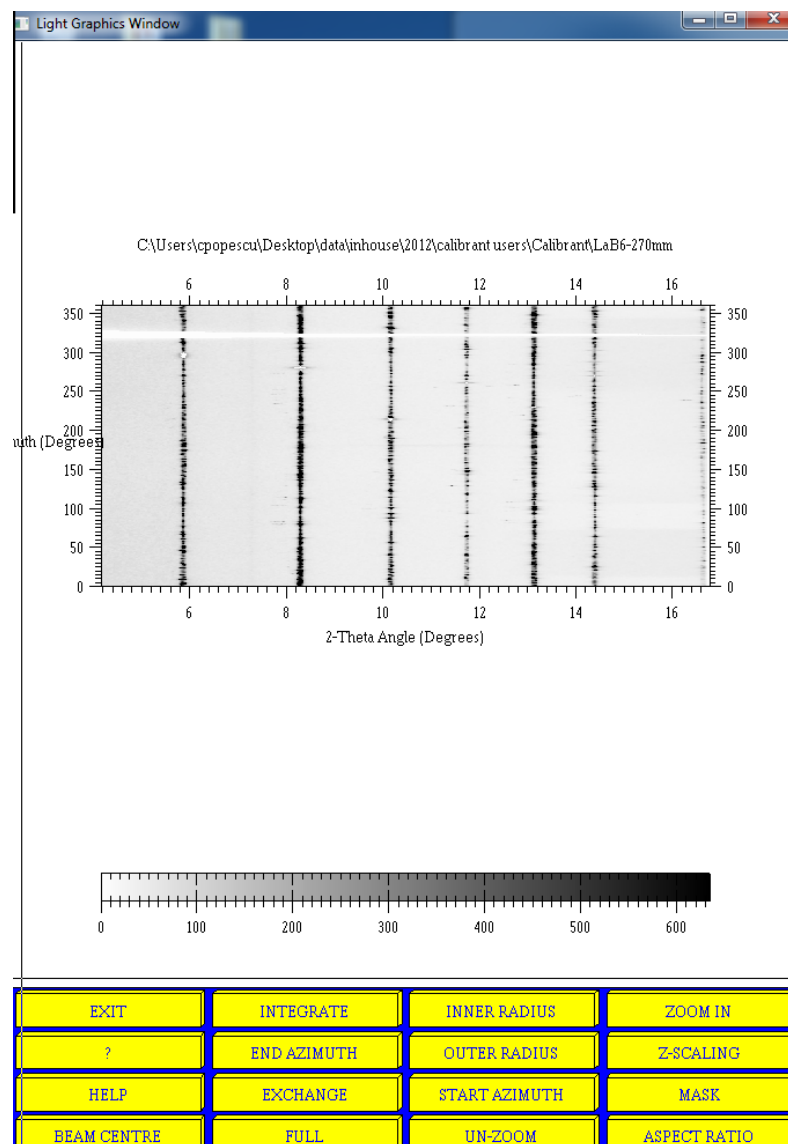
End Azimuth: +360

Inner Limit: beam center

Outer Limit: after the rings

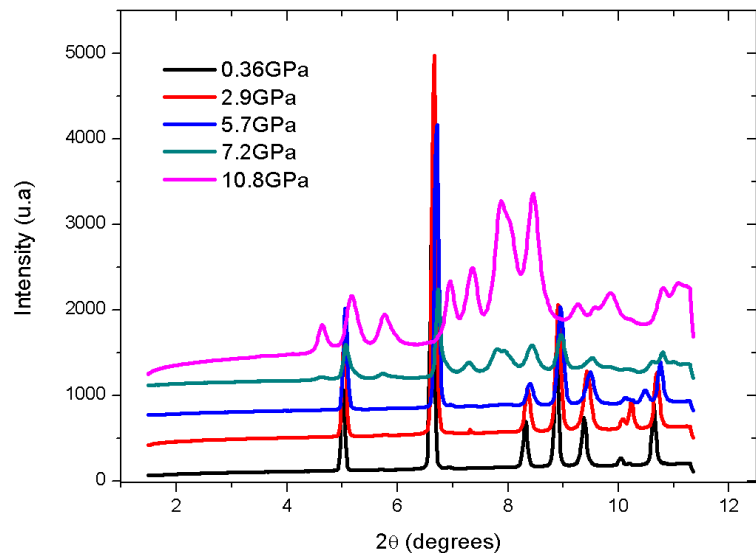
<INTEGRATE>

Calibration check: cake

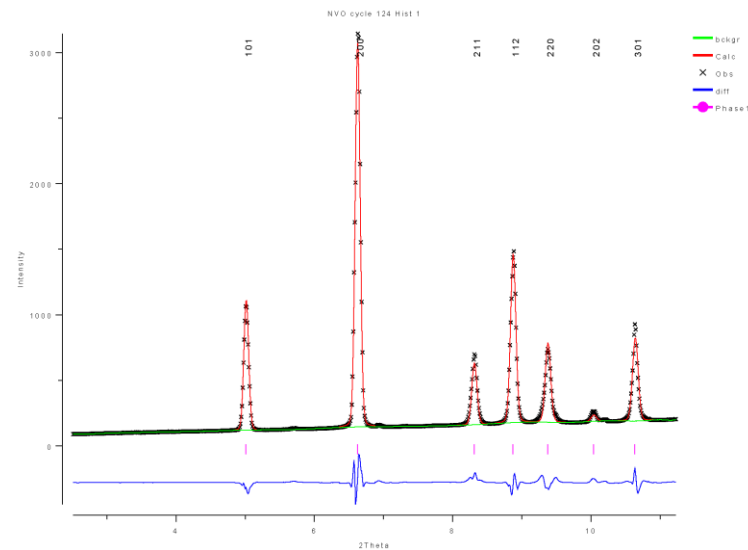


Inhouse NdVO₄

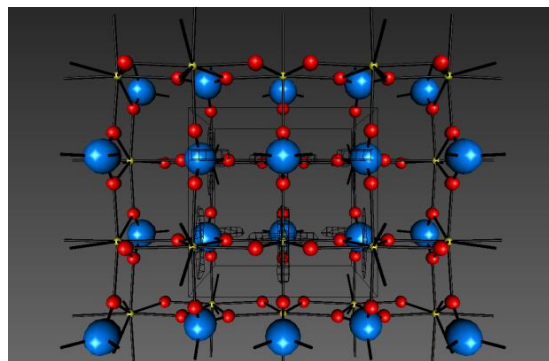
Zircon (I4₁/amd, Z=4) -> Monazite (P2₁/n, Z=4)



Evolution of the XRD patterns of NdVO₄ as a function of pressure



Observed and calculated XRD diffraction patterns for the zircon phase of NdVO₄ at 1 GPa



3D difference Fourier plot (GSAS)

