Calibration of the CPS120

- Put Y2O3 standard in the sample holder
  - Compress and level using a glass slide
  - Put sample holder into sample spinner. Make sure it slides all the way in
  - Close door

- To take the Pattern
  - In Acquistion
  - Clear the screen using the "clear" button
  - DO NOT pre-set the time (set time to 0sec) just watch the live time
  - Take pattern for 5mins: After 5mins (live time)
    - Open the door and remove the whole sample spinner (not just the sample holder)
    - Close door
    - Open Shutter
  - Take direct beam measurement for 45sec

Need to have a direct beam peak in same file as the pattern for calibration

- Save file as a DAT file (.dat)
  - Example:
    - 2007-16-05 caliby2o3xtalmirror.dat
      - Must save file with date (2007-16-05), type of file (calib), calibrant powder (y2o3) and what is in the path of the beam (xtal, mirror, or xtalmirror)

  - Open the .dat file in Data Treatment (Imad)
  - Find Peaks
    - In the "Fitting" tab
      - Peak Search
        - Use CF mode Put numbers 25 (far left box) and 6 (far right box) in the boxes to the left of the CF mode box
        - make sure fit channel and bck sub boxes are NOT checked
        - Set the range of 2theta to be from 0 to 120 in the boxes in the bottom right corner.
      - Click "Peak Search"
        - You need to make sure that all peaks are marked with a little upside down yellow triangle from 0-120°
          - To add peaks hold down the A (left click)
          - To remove peaks, hold down the Z (left click)
      - Under "View" click:
        - X-scale
        - channel

- ▪ Fit Peaks
  - o Still in the "Fitting"tab
    - ▪ Choose Gaussien to fit
    - ▪ Make sure the boxes to the right of the Gaussien tab has the numbers 35 and 200 in them
    - ▪ Ensure that the following boxes are CHECKED
      - • Fit Channel
      - • Bck sub

  - o Click on "Peak Fit"

Imad will do the peak fit and when done you will see dashed lines.

- ▪ Overlay of dashed lines (peak fitting positions) to solid lines (diffraction pattern)
  - o Go to "VIEW" click
    - ▪ X-axis
    - ▪ Channel
  - o Please note and remember the channel number of the direct beam, you will need it during calibration The value will be somewhere around the 320's

- ▪ Calibration
  - o Go to "Calibration" click
    - ▪ Cubic Spline

A new window will open: This is where you will create the calibration file

  - o In that new window select
    - ▪ YOBE/CU-D from the pull down menu on the bottom left
    - ▪ Set the channel value to the value of the direct beam that you noted earlier
    - ▪ Set "at angle (°)" to 0
    - ▪ Click the avg step carrots (<or>) to align the red arrows in the pattern with the blue arrows
      - • You will never get all of the arrows to align up, so focus on aligning the arrows in the lower 2theta values (20-60°)
    - ▪ Watch the R-value (top right of window) You want/need a value below 0.1.
    - ▪ In the table, check the Standard and 2Tcalc columns to make sure the values are similar
    - ▪ Confirm the wavelength is correct in the bottom left hand corner of the screen. This value will be used in the subsequent generation of the calibration file:
      - • Cu $K_{\alpha 1}$ (if with monochromator) = 1.540598 Å
      - • Cu $K_{\alpha,avg}$ (without monochromator) = 1.54184 Å

- o When you are satisfied
  - ▪ Click on the "Create Cal" button

This creates a .cal file with the same name as the .dat file

- ▪ Double-Checking
  - o Close the files that you are working with in Imad
    - ▪ File
    - ▪ Close
  - o In Imad Open the file that you just closed

You should see red dashes on it now

- o In the "standard" tab
  - ▪ Under the "Standard" heading
    - • Select the calibrant powder (YOBE/CU-D)

It will overlay, and you will see blue dashes that will not line up with your peaks

- o Go into "Edit"
  - ▪ Change Cal
  - ▪ Select the .cal file that you just made
    - • Remember it will have the same name as the .dat file

Now the peaks in the diffraction pattern will line up with the blue dashed lines

- ▪ To Save
  - o "Save File"
  - o Chose DAT file
  - o Overwrite the .dat file that you made earlier

You do this so the .dat file is calibrated.

- ▪ In Acquisition
  - o Click on the "Change Calibration" button in the top left corner
  - o Select the .cal file that was just made
  - o Close Acquisition
  - o Open Acquisition
    - ▪ Double check that the calibration file that is displayed is correct
- ▪ Put the sample spinner back in place
- ▪ **BOLDLY** write the name of the calibration file on the record sheet