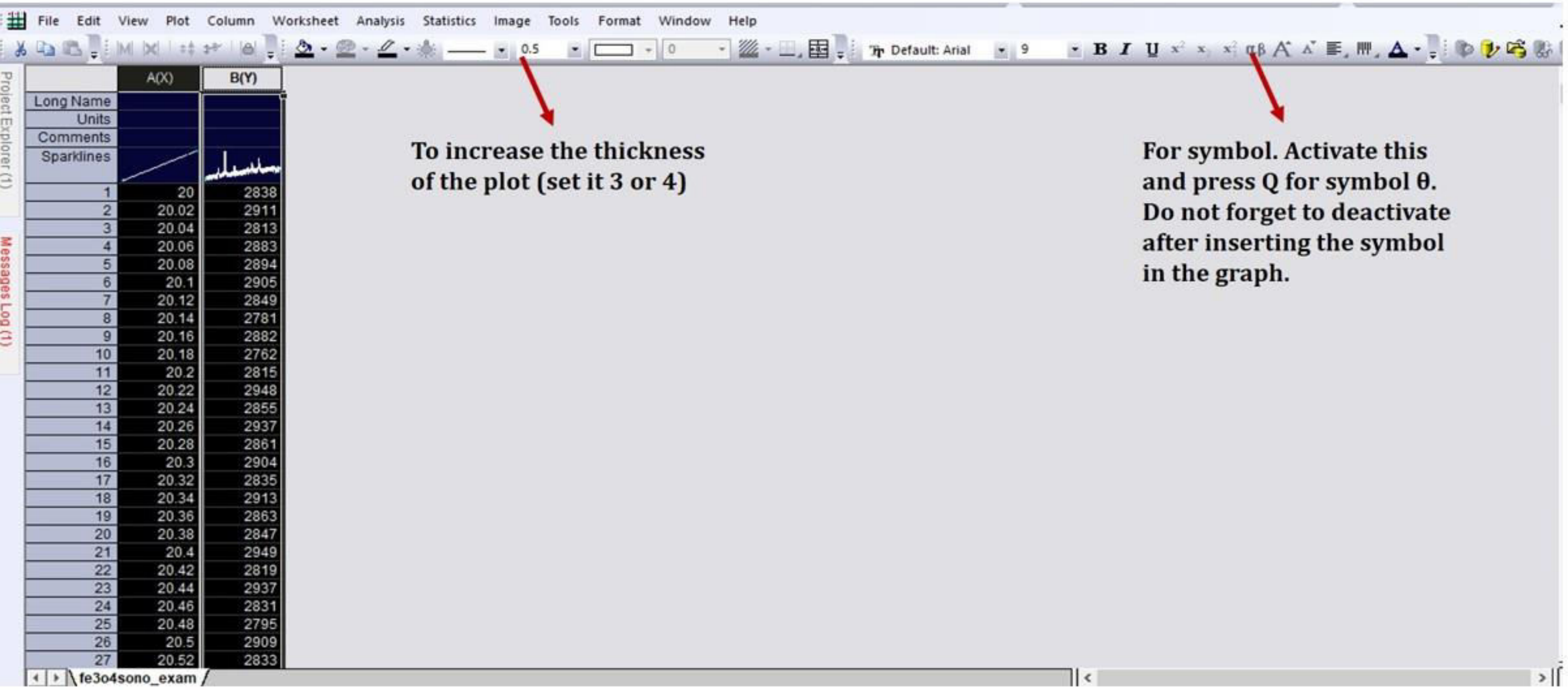
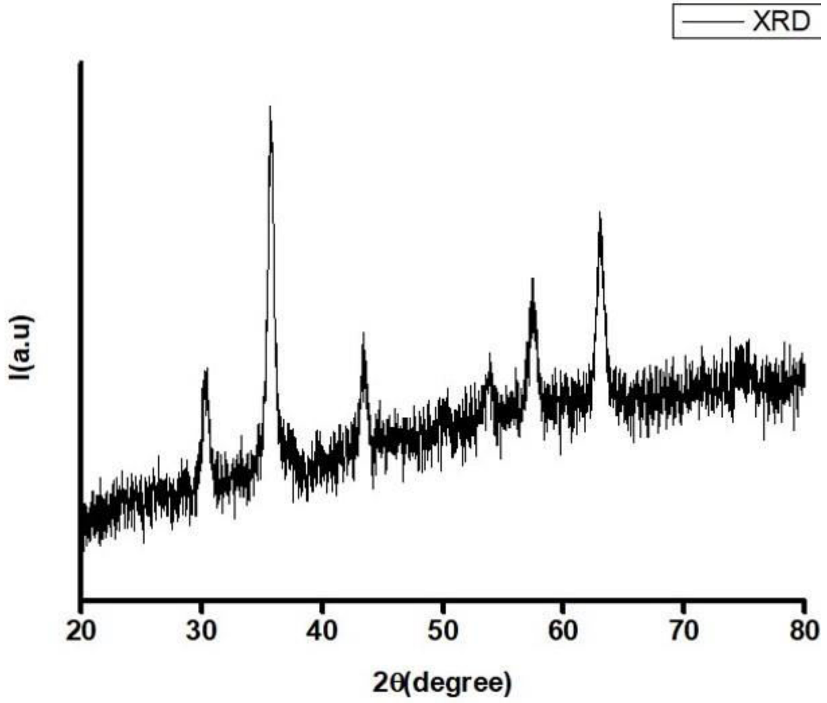
Estimation of crystallite size of an unknown sample

Finding instrumental error from XRD pattern of the known sample:

1. Go to File ⟶ Import ⟶ Single ASCII

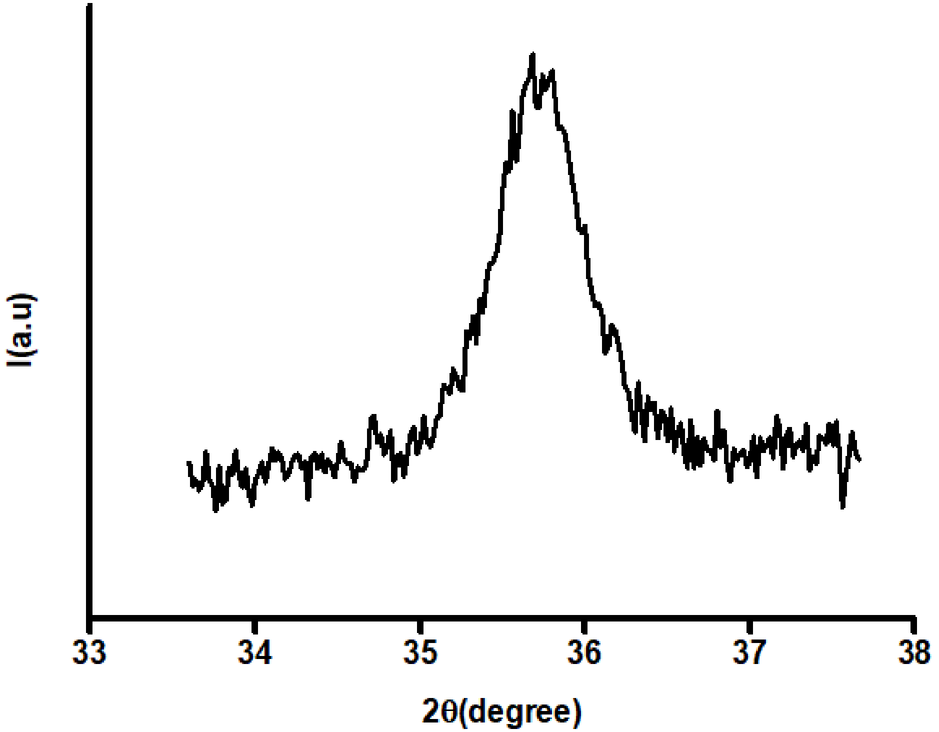
2. Select both the columns and plot the graph. Label X-axis as 2θ (degree) and Y-axis as I(a.u).





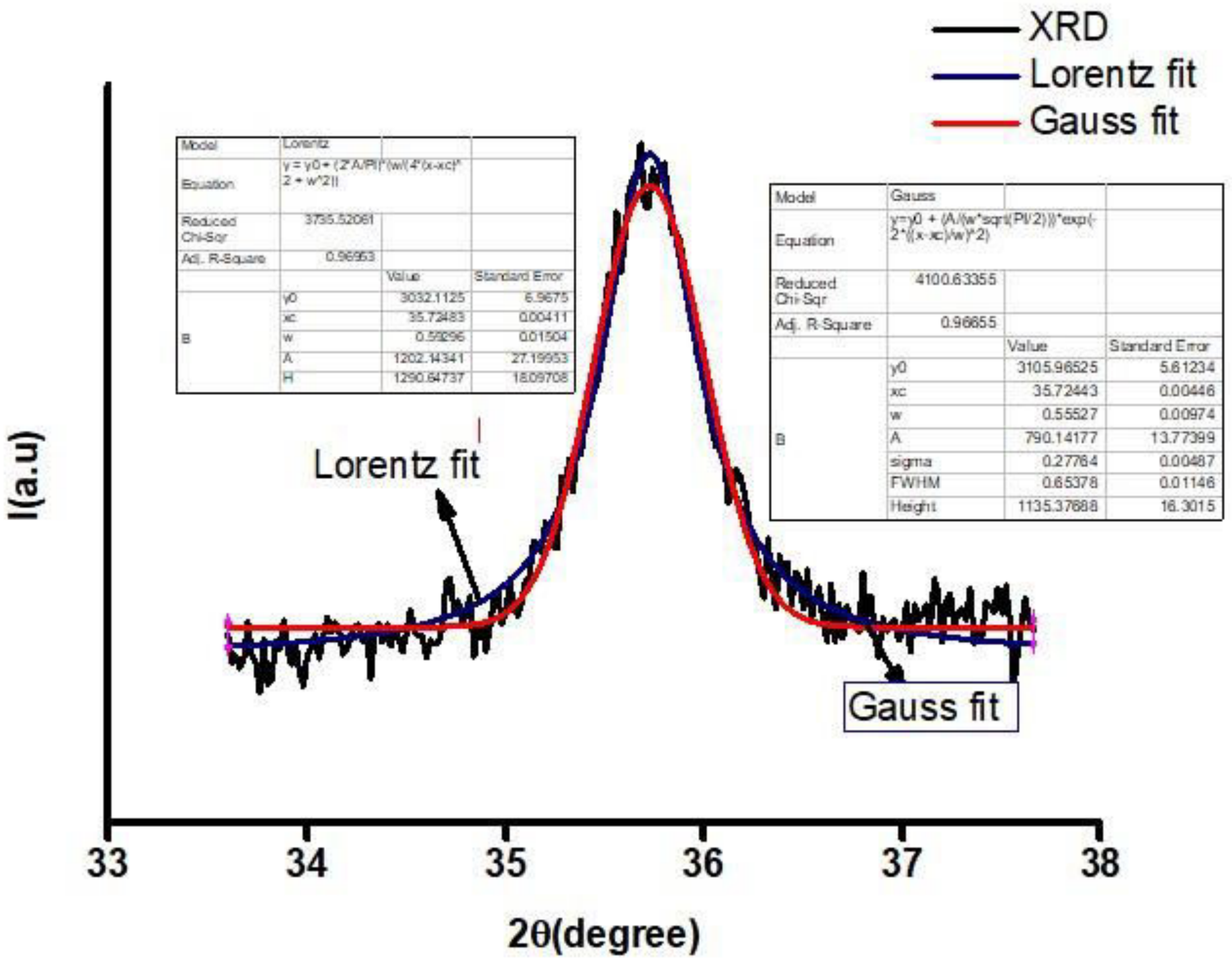
Finding instrumental error from known sample XRD pattern:

3. Now plot only the high intense peak of the XRD pattern and label X-axis and Y-axis as labelled for the complete XRD pattern.



4. Go to Analysis → Fitting → Nonlinear curve fit → Select function type Lorentz/Gauss

5. Fit the experimental graph with both functions and use the best fitted values for the calculation.



Note: For Lorentz fit ‘w’ and for gauss fit ‘FWHM’ is the value of FWHM to be used in calculation of instrumental error and crystal size.

While calculation FWHM value should be converted to radian by multiplying with π/180.

For Gauss Fit, the equation as follows:

$$\beta_{Tot}^2 = \beta_{Cryst}^2 + \beta_{inst}^2 + \beta_{strain}^2$$

For Lorentz Fit, the equation as follows:

$$\beta_{Tot} = \beta_{cryst} + \beta_{inst} + \beta_{strain}$$

For calculation, we ignore the β_{strain} term.

- $\beta_{cryst} = \frac{0.9\lambda}{D\cos\theta}$, obtained from Scherrer's equation. λ is wavelength of X-ray source (in this case, it is Cu-kα source with wavelength 1.5406Å, D is known crystallite size, θ is half of the bragg angle(2θ). Do not convert it to radian, it should be in degree only.
- β_{Tot} is the FWHM of the XRD peak. The FWHM value is obtained from the fitted table: 'w' for Lorentz fit and 'FWHM' for Gauss fit. Convert it to radian while calculation.
- Then β_{inst} is calculated from the above equations.

Calculation of crystallite size of unknown sample:

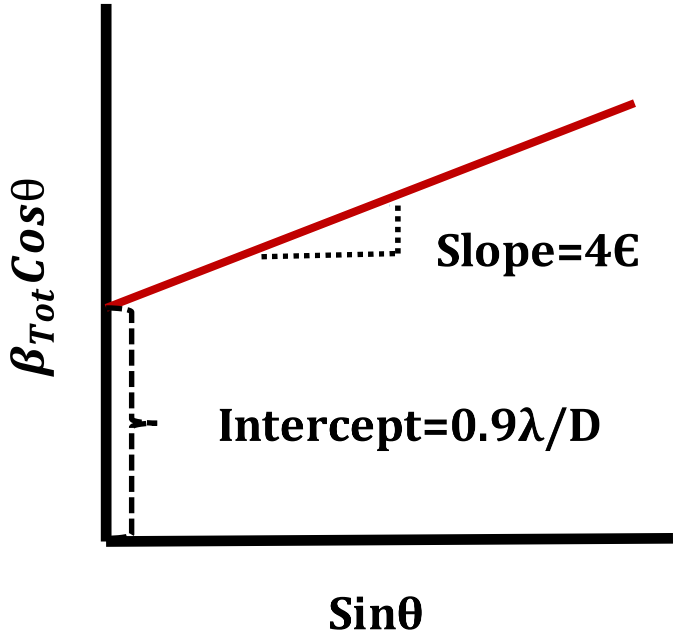
1. Follow steps 1)-5) for unknown sample.
2. Repeat the steps 1) -5) for at least five peaks of the unknown sample.
3. Follow the same equations to calculate the crystal size:

$$\beta_{Tot}^2 = \beta_{Cryst}^2 + \beta_{inst}^2 + \beta_{strain}^2, \text{ for Gauss Fit.}$$

$$\beta_{Tot} = \beta_{cryst} + \beta_{inst} + \beta_{strain}, \text{for Lorentz fit.}$$

4. For unknown sample, β_{Tot} is obtained from fitting of the peaks similar to the known sample. β_{inst} is already known (as calculated from the known sample). Then, crystallite size is calculated for each of the five peaks. Further the mean size is calculated taking average of the five crystallite sizes.

Williamson-Hall plot to calculate strain and crystallite size:



$$\beta_{Tot} = \beta_{cryst} + \beta_{strain}$$

$$\Rightarrow \beta_{Tot} = \frac{0.9\lambda}{DCos\theta} + 4\epsilon\frac{Sin\theta}{Cos\theta}$$

$$\Rightarrow \beta_{Tot}Cos\theta = \frac{0.9\lambda}{D} + 4\epsilon Sin\theta$$

Strain

Estimation of strain and crystallite size from Williamson-Hall plot.

Estimation of crystallite size of an unknown sample:

- Calculate the instrumental error of the known sample.
- Calculate crystallite size of the unknown sample.
- Calculate the error in crystallite size estimation.
- Plot Williamson-Hall plot and calculate strain and crystallite size.
- Compare the both crystallite size values and discuss the reasons if any discrepancy in values observed.
- Discussion.