Rietveld Refinement Steps

Friday, January 24, 2014 8:29 AM

#1 - open expgui
#2 - navigate to a new folder (remember! No spaces in folder names)
#3 - make a start.exp file and click "read"
#4 - click "create"
#5 - leave next box for experiment title empty
#6 - under phase table click "Add Phase"
#7 - under "import phase from" field select crystallographic information file .CIF
#8 - select Co3O4 and add it (accept the pop up screens confirming spinel origin and symmetry operators
#8 - click add new atoms
#9 - follow steps 6-8 for the CoO phase as well.
#10 - now that phases are added we need to add the experimental XRD patter we will refine against. Go to powder tab and select "Add new Histogram"
#11 - Select the .raw file corresponding to a given temperature
#12 - click add instrument parameter file and select the file provided for the homework (this will automatically set the Co radiation)
#12a oops! Click Edit Background. Under function type select #1 Shifted Chebyschev
#13 - under scaling turn off the overal scale factor and turn on the scale factor for each phase
#14 - we can now do our first refinement. Click powpref, accept new values and click genles and accept modification values
#15 - click liveplot to see graphically how good a fit is achieved
#16 - notice that some peaks don't correspond to the phases we want to refine! These are from the sample holder, we need to ignore them
#17 - go to powder tab and select "Set Data and Excluded Regions"
#18 - zoom in on regions, click "add region" click on beginning of unwanted peak and end of unwanted peak. Repeat for all bad peaks. Tip! Use the impurity image provided.
#19 - We now can look at liveplot and see what seems to be the error. Both peak position and shape look wrong.
#20 - let's refine position of peak first by refining unit cell lattice parameters under phase tab
#21 - run powpref/genles to see improvement
#22 - you can increase # of refinement cycles under LS Controls tab, default is 3, change it to 10
#23 - go to Constraints tab and we must constrain UVW to be the same for both phases
#24 - constraints tab, click profile constraints, click add constraint, select U V and W click continue, highlight phase 1 & 2, click save
#25 - go to profile and refine U
#26 - run powpref and genles
#27 - refine W
#28 - run powpref and genles if chi^2 is less than 4 you are probably done.
#29 - to get wt fraction to to lstview tab up top
#30 - scroll down to phase / element fraction for phase 1 and 2
#31 - write down the wt. fraction of each phase

NOTE! Do not refine the zero of the instrument, we had it calibrated before we ran our sample and it is close to actual zero, it doesn't need to be refined.