BRUKER X-Ray Diffraction

Warning: This instrument may only be operated by those who have been trained by AAF staff and have read and signed the AAF laboratory policies.

1. Computer should be on. User: Bruker XRD Password: brukerxrd
2. Software (should be open)
   A) Open DIFFRAC.Measurement Center (to take data) Login Name: Engineer Password: (blank)
      i) Click on COMMANDER Theta, Two Theta, Detector, Twin_Primary, Twin_Secondary, Voltage, Detector (again) should all have a green check next to them. If not notify Steve H. If Phi and Rotation are not check – do not worry…
   B) Open DIFFRAC.EVA (to analyze data)
3. Samples: sample holders
   A) Small circular "puck" sample holder. - powders
      i) Fill from the center out. Flatten sample to same vertical height as top of puck. Place puck in spring loaded holder. Carefully place sample in experimental position. Magnetic holder.
   B) Large circular "puck" sample holder. - powders one side – bulk samples (<13mm high, < 50mm diameter). Mount sample in puck and place in experimental position Magnetic holder
4. Run white corundum sample to check Bruker XRD!! Run at 40mA/40KV (standard power)
   4.1 mount std corundum sample in spring puck holder.
   4.2 using a wizard file (*.bsml files - user created 5.1 below or use std from SEH) - using with standard BB geometry (slits 0.6 and 5mm) no Ni filter 30-38 2 theta scan (40mA/40kV power 2min total time step size 0.02 2theta)
   4.3 Run XRD via START JOBS tab see 5.2 below
   4.3 $K\alpha_1$ Cu peak at 35.15deg 2theta and >350cps. See management if different
5. Set-up Diffraction Event/Scan (Make an Event/Scan File)
   5.1 In DIFFRAC.measurement center program
      A) Click on Wizard
      B) Top Wizard (between View and Help) click on open (desktop/xrd data/event_template/bb_10_70deg20min norotate.bsml)
      C) Edit and save Event/scan file in your folder.
         i) "most of the time" only need to change, start/end two-theta, time/step, rotate sample or not, as needed. Other changes contact Steve H
         ii) Save Event/Scan file in your folder
   5.2 After sample placed in position, Click Start Job
      A) Sample id as needed (ASCII)
      B) Experiment Name - XRD Event/Scan File (insert as needed)
      C) Result file Name - XRD data file – to be saved in your data folder (desktop/xrd data/… (add as needed)
      D) Start Jobs (lower right corner) message not initialized can be safely ignored if all the parameters in 2Ai (above) are indicated (checked green)
   5.3 Click COMMANDER (to see real time display).

6. XRD Data Analysis DIFFRAC.EVA
    - 6.1 Open data file
    - 6.2 File/Import from files/pick a data file (desktop/xrd data/…) - open
    - 6.3 Left click and drag right to zoom in area of interest (right click and unzoom …)
    - 6.4 Remove Background
        - A) Data Command/Tool/Background Adjust curvature slide as needed. Click on Background Subtracted (as needed)
    - 6.5 Strip $K\alpha_2$ (zoom out to entire scan and all peaks will/should have their $K\alpha_2$ removed – only do stripping if must)
        - A) Data Command/Tool/strip $K\alpha_2$
            - i) Vary intensity ratio slide to get best $K\alpha_1$ peak. (0.55 usually close)
    - 6.6 Fit Peak (position, FWHM…) Data command/Tool/create area
        - A) Select Area - left click on left side of peak – drag to right side of peak release.
        - B) Peak information shown/calculated - Observed Maximum position, Gravity center, FWHM, crystal size (assumes instrument resolution is 0.00?)
    - 6.7 Peak search/find data Command/Tool/Peak Search (zoom to scan area of peaks needed)
        - A) Adjust width slide bar to include peaks as observed.
        - B) Click append to list
        - C) Click on Peak List - Data Command/Create/Peak Column View Shows list of all peaks found. Delete or Edit peaks as needed. (right click peak – delete…)
    - 6.8 Search Data Base Data Command/Tool/Search/Match(scan)
        - A) Chemical filter: Pick element(s) that are Mandatory, excluded, etc (click on individual elements until correct color is seen)
        - B) click REBUILD
        - C) Database Filter ICDD best (not in data taking computer)
            - i) # of elements in formula (minimum and maximum)
            - ii) density min/max
        - C) Click REBUILD
        - D) Candidate List - click SEARCH Brings up possible matches. Can scroll through matches with mouse. Checking a "match" box displays standard match with/on data. The more boxes checked the more "matches" displayed.
        - E) Data command/Create/ DB View/ Brings up the information of the match
            - i) System(cubic…)
            - ii) Space Group
            - iii) a,b,c
            - iv) I/I_c (for phase weight comparisons)
            - v) HKL's seem to be random????

7) PLEASE CLEAN AREA WHEN DONE
8) Return Tube and Detector to 15deg: (on COMMANDER check the "Theta" and "Detector" at 15 deg and then press " Position All Checked Drives"
9) Fill out Bruker XRD usage book