Single Crystal XRD using Bruker D8 Venture Duo

On APEX4:

Left side bar **right side window**

- Create new project New sample (top left) (always save other people's old projects), fill in crystal information

On diffractometer:

- Mount crystal
- Close the door when detector is moving
- Physically center with goniometer
  - Up/down movement is higher one
  - Left right is lower one
- Turn on the light bottom at right side of the diffractometer

On APEX4:

Left side bar **right side window**

Set UP -> Center Crystal: Click Center once mounted

- Open doors on diffractometer
- Align once, rotate 90
- Align again, rotate 90
- Align again, rotate 180
- Rotate around to check if it is centered
- Once it can rotate without misalignment use the camara to take measurement of crystal size (each tick is 20 micron (0.02mm), radius is 100 micron (0.1mm))
- Put the size in Set UP -> Describe

Evaluate -> Determine unit cell

- Run (it will go through each step automatically to find a unit cell), if the unit cell is not right, you can manually adjust the threshold, and manually go through the process.

Collect -> Calculate Strategy

Anode: Mo, Resolution: 0.4A for solid state

Symmetry: lower than your space group, check the HKL range at top window, make sure cover both positive and negative HKL

Parameters for the strategy determination:

Crystal to detector: 40mm standard (50mm if long axis), click ok

Shortest normalized exposure time: 0.5

Select Scan Parameter:

Frame angle: 0.5 degree,

Frame time: depending on crystal can change time per step, <5s. we use 2 sec for iridates

Collect -> Run Experiment

Add Crystal video into 2

Append strategy

Validate to make sure it'll run

Execute

After collection:

Evaluate -> Determine unit cell

Select image (folder at top), don't use fast scans use actual scans (samplename_001), when you go to last scan should be _0180

Harvest spots

Min I/signal = 3, harvest

Once harvested, index, index

Pick group, accept, set

Bravais, select one you want

Refine, use more peak, refine until it converge, now have new unit cell

With the new unit cell, start processing:

Reduce data -> integrated images:

Select unit cell you just made

Import runs from experiment (make sure you don't use the fast scans or use fast scans when missing low angle data)

resolution limit: 0.4

Refinement options:

XYZ box size to 0.8,0.8,1.2 or 1,1,2 depends on your crystal

Integration options:

More options -> Algorithm -> Monte Carlo simulation: 32, start integration

Reduce data -> Scale

Symmetries: (Laue and Point)

Mu*r : 0.1 or 0.2

Start

Refine

Examine data -> Analyze data (x-prep)

This will export required files for refinement