**Instructions for Bruker D8 Discover with Vantec Detector**

**Getting Started**

Enable the instrument in Coral, using the Coral computer (also analysis computer) next to the computer controlling the instrument.

The instrument will normally be in a standby condition with X-rays on and at low power. The yellow leds on both sides of the instrument are the indication that x-rays are on. Yellow leds on the tube housing also indicate that the x-rays are on. When the shutter is open, a red leds will be on above the yellow ones.

On the left side of the instrument as you face it there are two lights below the yellow leds. These lights indicate the condition of the instrument. The conditions when you come in should show a “generator stable” light on top and the “computer connected” light on the bottom. The legend for the different symbols is on the left side of the instrument.

Two different tubes on this instrument, Cu = 1.54 and Co  = 1.78. The computer reads which tube in on the instrument, which is also indicated on the top of the tube. Filters are used to eliminate k-beta radiation; Ni is used for Cu and Fe is used for Co. The filters are in a rack inside the right side of the enclosure. There are 4 different collimators for the tube (0.3, 0.5, 1.0 and 2.0 mm). The collimators mount magnetically to the front of the tube assembly.

The large sample stage is an X-Y-Z stage with +/- 45 mm of translation in X and Y and -45 mm translation in Z. The stage also has a rotational axis (phi).

The detector is a 2D Vantec detector, which has been calibrated during installation. It should not need to be re-calibrated, but let me know of any issues. At the default distance of 200 mm from the sample, the detector collects a range of ~30 deg in 2theta.

The enclosure doors slide apart. Push in buttons on inside of handles. The safety interlock is with the shutter; it is safe to open the enclosure doors with the X-rays on. Open the doors to the instrument and put in sample. There are various sample holders and spacers in the left side of the instrument. Most of the sample alignment is done using the Z axis.

**Emergency shutdown:** There are two red buttons on either side of the instrument and a 3rd button inside the enclosure that are shutdown switches. In the event of an emergency (i.e. electrical fire, flood, etc) push any of these buttons to shut down the instrument.

**Measurements**

On the computer, open **Diffract.Measurement Center** (lower left portion of monitor). If the computer has been re-started, the username is Cornell University (no password).

**Diffract.suite** is the measurement suite. Right now the user name is: lab manager with no password. Data is saved on the computer, on the C: drive in the D8 users folder. (Eventually, we will have the X-ray server working in Bard)

A list of programs in the Diffract.suite appears on the left side of the screen and across the top of the screen. “DaVinci” shows the setup for the instrument, including tube, collimator and detector distance. Check this if you are unsure of which tube is mounted.

**“Commander”** is used to collect data manually, move axes, etc. The procedure described below is for this program option.

Open “Commander”. You will see a list of the axes and their current positions (in bold numbers) and the current settings for the current and voltage (also in bold). The column to the right of these number is where you enter the values that you want to set.

Start by entering 40kV and 40mA, if the Cu tube is in. Enter 35kV and 45mA if the Co tube is in. Click on set to have the values come up. There is no warm-up time.

To move an axis, enter the new value (the box next to it should get checked automatically. If it doesn’t, check the box). Then select the box that says “position all checked drives” (at the top of the column of boxes with blue check marks).

Below the screen where data will appear, there are 3 tabs: “main display”, secondary display, video and info. Select the video option to align the sample height. Turn on the laser (check the Laser 1 box). Using the Z slider first, align the height of the sample. When the laser spot is in the center of the cross-hairs, the height is correct. Then position the sample using X and Y, if needed. After aligning your sample, turn off the laser.

You are now ready to collect data. The range of 2theta values that is collected in a single frame of the Vantec detector is ~30 deg. You need to decide if you need multiple frames to collect the range of 2theta values that you want. The range of scan types is shown below the main screen. A coupled 2theta/theta “scan” is what we normally collect. The scan mode should be “step”. Time is the # of seconds/step. The “steps option” is the # of positons that you need to collect the required range of 2 theta values (you will usually need between 1 and 3 steps to collect your data). The 2theta value will be the position of the Center of the detector. Data will be collected +/- 15deg from that position. So, for example, positioning 2theta at 20deg to start gives you a frame that has data from 5 deg to 35 deg. (You should not positon 2theta below 18 deg. This will collect a range down to ~3 deg to 33 deg. Positioning the detector at a lower angle will result in collecting the direct beam which doesn’t damage the detector but doesn’t result in good data for you.) The increment is how much do you want the detector to move for each data acquisition. An increment of 15-20 degrees gives a reasonable overlap area. The “stop” for 2theta should adjust automatically based on the # of steps and the increment. Select “Start”.

You will see the data displayed on the main screen as it is being collected. There is a delay; it is not instantaneous.

When all the data has been collected, the shutter will close automatically.

**Save the data!!** The data is not automatically saved. You must go to file—save results. Go to the C:/D8 users/your folder. All frames will be saved and incremented .001, .002 etc.

**Setting up a job, (or macro).**

This option allows you to program a single scan, or multiple scans and will automatically save the data.

**Select Wizard, then New** (at the top of the page)

This option will allow you to create a macro (or a job) that you can save and run at anytime. The advantage of this option is that the filename is entered into the job and the data is automatically saved.

From the “create a new experiment” window, select XRD, and then OK. A window on the left side of the screen shows you options that will be set up for the experiment.

Under the XRD basic, Method 1, Davinci is checked off. The current setup for the instrument appears. Make sure it is what you want for your data acquisition.

Select XRD setup. This is where you setup the condition for data collection. (2theta position, # of steps, time/step). (the next option VCT/VSS is not active)

If you don’t wish to add anything further to the job, save it in your directory. It will have a .bsml extension.

You can also continue to add to the job by giving multiple positions for data acquisition if you want to. Select XY positons. This option allows you to program data acquisition on a grid, X or Y line scans, “free” positions, etc. Under the “positions” option, select the box next to “style” to set up the type of multiple scans that you want to do. Then save the job.

**Open Start Jobs.** Load your .bsml file under Experiment Name. Under Result file name, give the data a file name; it will be automatically saved. If you are doing an X-Y scan, the file names will be incremented, 001, 002, etc. Select the button that says “Start Jobs” at the lower right portion of the screen.

**Shut Down**

When you are finished collecting data, lower the voltage and current to 20kv and 5ma, which are the standby settings for both tubes.

**Analyzing Data: EVA**

Open Diffrac.EVA at the bottom of the screen. This is the Bruker analysis program.

Under file select Import file. If you have more than one frame for your data, select all frames. Eva will combine the frames into a single frame and display it on the screen.

There are 5 icons at the upper left of the display window that give you options for integrating the data, which produces an x-y plot of intensity vs 2theta. We normally use the “wedge cursor”.

Once you have selected the area to be integrated, select the “integrate cursor” option under the tool menu at the left side of the window. You will get an X-Y plot of the data. This file can be exported (Export scan) to a .raw file which can be used with EVA to add files together or subtract background,etc. Eventually we will have the JCPDS data base connected to the D8 computer to be used with EVA.

This data can be also exported to a .xy file that Jade can read. (the .raw file doesn’t seem to open properly in JADE).

On the C: drive of the D8 computer, there is a folder **“Xray-data**”. Move any data you want to analyze using Jade into this folder. There is a short cut to the folder on the desktop of the Jade computer making it easy to access your data in Jade. EVA is also running on this computer so you can analyze data whenever you need to.