**Instructions for Bruker D8 ECO Powder Diffractometer**

**Getting Started**

Enable the instrument in Coral, using the Coral computer (also analysis computer) next to the computer controlling the instrument. Please pay attention to the name of the instrument, as we now have two Bruker D8 machines in the facility.

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, 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. When you come in, there should be a “generator stable” light on top and the “computer connected” light on the bottom. The legend for the different symbols is on the front of the instrument.

This instrument uses Cu radiation, = 1.54A. A Ni filter is used to eliminate k-beta radiation. Settings for the tube will be **40kV** and **25mA**. (Do not exceed these values).

The detector is a Si strip detector with 160 channels. It is a high speed detector meaning that data can be collected very quickly. Longer hold times per step will greatly increase the total amount of counts in a scan.

There are two buttons on the upper right side of the instrument. The top button is for the inside light. The bottom button is pushed to allow the door to open, which opens straight out. The safety interlock is with the shutter; it is safe to open the enclosure doors with the X-rays on.

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

**Sample Mounts**

The sample mounts are made of PMMA. They can be used on the flat side or on the cavity side. We are having several modified to take the round low background holders

**Collecting Data**

Open **Diffract.Measurement Center** (at the bottom of the screen)

**Diffract.suite** is the measurement program. Right now the user name is: lab manager with no password. Data can be saved on the C: drive in the D8 Eco users folder, or on the Xray server. The server allows you to access files from any of the computers in the lab.

Open “**Commander**”. You will see a list of the axes and slits with 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 25mA for the X-ray generator. Select Set**. DO NOT EXCEED** these values. (This instrument uses a 1 KW generator, not the 3KW generator that is on the other D8 instrument.) There is no warm-up time.

To move an axis or a slit, 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).

The screen to the right is where data will be displayed. Below the display area is where you set up your diffraction scan. The setup for a powder scan should be: Scan type: coupled 2theta/theta; Scan mode: continuous PSD fast. Enter the start and stop angles, the step size and the time/step in seconds. The total number of steps and total time for the scan will be displayed. The PSD opening has a maximum value of 2.7 deg, which is normally used. Select START. Because of the way data is collected with this detector, the scan will appear to start at an angle lower than the specified starting angle. You will see the data displayed on the main screen as it is being collected. 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 ECO users/your folder.

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

This option allows you to program a single scan, or multiple scans and will automatically save the data. When you have created a scan, as indicated above, it can be saved as an experiment with a .bsml extension. Open the “Start Jobs” option. Load your .bsml file under Experiment Name. Under Result file name, give the data a file name; it will be automatically saved. 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.

**Low Angle Scans on the D8 ECO**

Low angle usually applies to the range 1 to ~15 deg.

You can minimize low angle noise and scatter by changing parameters for the diffraction scans as follows:

Make sure the air scatter screen is in and is almost touching the sample

Set the divergent beam slit to 0.1 or 0.2 mm. The normal setting is 0.6 to 1.0 mm.

Set the scatter slit to 3 mm. The normal setting is 9 mm.

Set the PSD opening to ½ of your starting angle.

These steps will greatly reduce the low angle noise but will also lower the intensity of your peaks. Since you can subtract the background in JADE or EVA, you may want to just use the normal settings.

**Fluorescence Suppression**

The settings on the SSD detector can be changed to suppress fluorescence effects in Fe-bearing samples.

To the right of the detector (SSD 160) box, click on the box that shows the detector settings. Change the “lower discriminator” from 0.11 to 0.19, then select apply. This will lower the high background due to fluorescence. After completing your scans, change the “lower discrminatgor” value back to 0.11. The peak heights are also lowered, so it may be preferable to subtract the background in JADE or EVA.

**Analyzing Data: EVA**

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

Under file select “Import file” to open the data. (Selecting Open will only show you EVA files.) EVA can be used to find peak positions, subtract background, do search-matches, etc. 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). The analysis computer for the D8 ECO has both EVA and JADE on it for data analysis.