Standard Operating Procedure:
Bruker D8 Discover

(You must be trained by MSE Staff or Authorized Trainer to use this machine)

Last Revision: 2008/12/16

Location: Gerstacker Building B115

Hazards: (The following materials and equipment associated with this procedure present exposure or physical health hazards. Safety precautions are prudent and mandatory.)

This unit produces a high intensity X-ray beam. Take all precautions to avoid exposure to the x-rays.

Warning: Beryllium!
Do not touch the front window of Area detector as it contain Beryllium. Fumes of the dust from Beryllium and its compounds can be hazardous if inhaled!

Engineering Controls: [Prior to performing this procedure, the following safety equipment must be accessible and ready for use: (e.g. chemical fume hood, biological safety cabinet, laminar flow hood, chemical spill kits)]

None

Protective Equipment: [Prior to performing this procedure, the following personal protective equipment must be obtained and ready for use: (acid resistant gloves, safety eyewear, lab coat, chemical splash apron)]

You must wear radiation dosimeters when operating this equipment. The dosimeters will be checked every 3 months to determine whether you have received any x-ray exposure.

Waste Disposal: (This procedure will result in the follow regulated waste which must be disposed of in compliance with environmental regulations)

None

Accidental Spill: (In the event that a hazardous material spills during this procedure, be prepared to execute the following emergency procedure)

Nat applicable.

Typical Experiment Sequence:

1: Sign in Log sheet.
2: Launch **GADDS** Program
3: Load and align sample with **Camera-Laser System**
4: Set up scan and save data
5: Quit GADDS program and leave X-Ray Power in standby mode.
6: Sign out Log sheet.

Procedures:

1: Sign in Log sheet.
Check the machine status on either left or right column before using the machine. The "Ready" and "On" lights should be on. The "X-Ray ON" lights on top of machine should be on.

On the left control column:

STOP button: If hit it switches off the control electronics. The X-Ray source is turned off and all moving drives will stop instantly. The stop button should only be used in emergency situations and not for normal shut down of the diffractometer system.

 **Light button**: This button turns ON/OFF the fluorescent tube located at the ceiling of the enclosure.

 **Fan button**: This button turns ON/OFF the fan located on top of the enclosure.

System Instrument-Key Switch: This key will lock/unlock the system. During normal operation the switch must be in position "unlocked".

Open Door button: This button must be pressed to open the front door. If the Open Door button is activated while the tube window is open the X-ray shutter will close automatically.

Ready light: Status of High Voltage Generator. Illuminated orange display indicates that the high voltage generator is operating. Flashing display indicates the high voltage generator is ramping up.

On light: Illuminated green display indicates the control electronics are ready.

Alarm light: Illuminated red display indicates a pending system alarm, flashing red light indicates a pending warning.

Busy light: Illuminated yellow display indicates that a measurement is in progress. Flashing yellow display means that the system is not ready.

Right column:

Stop button: Function same as **Stop** button on left column.

 **Power OFF button**. It switches off the control electronics, the high voltage generator and all components connected to the AC outlets.

 **Power ON button**. It switches on the control electronics, the high voltage generator and all components connected to the AC outlets.

High Voltage- Rotary Switch. Used for turning high voltage on or off.

Ready, On, Alarm and **Busy** lights have same functions as those on left column.

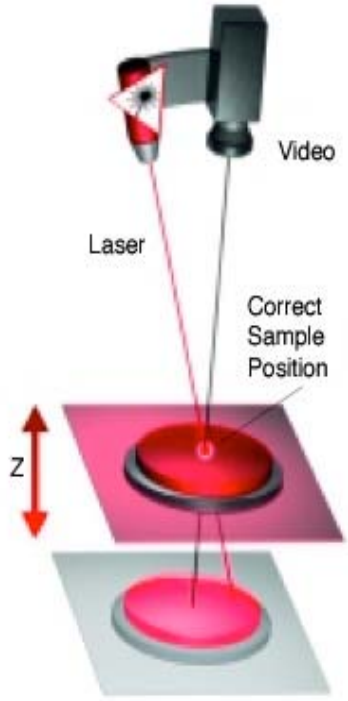
2: Open GADDS program, it will prompt "set generator to user setting: 40 KV and 40 mA" Click Yes.

3: For new user/new project, go to Project\new, input info accordingly. Use working directory C:\frames\users\"unique name"\"project name"

4: Install Sample:
Before you open the D8 door, push **Open Door** button on either side of control column, then open the window. Put your sample on the XYZ table and position sample approximately directly under the camera.

5: Sample alignment:

D8 Discover use **camera-laser system** to align a sample.



All the components including X-ray beam, camera, laser have been aligned to aim at a point which is called universal center of the D8. To align your sample surface at this universal center, you need to use the Manual Controller to move the XYZ table. To activate Manual controller, press Ctrl+Shift+M in GADDS program. Then press “L” to turn on **Laser**.

The Manual Controller: It is general controller and not all the functions will be needed for the D8 Discover. The axes we used here are:

1- θ1 (X-ray source)
2- θ2 (HighStar 2D Detector)
5- X
6- Y
7- Z
8- Camera Zoom

Push "↑"or "↓" button will increase or decrease the position of axis.
Push "+" or "-" will increase or decrease the speed of movement.

!!!Warning: Make sure all the axes will not run into each other when you try to move any axis since there is NO safety switch installed for this purpose. The "**Emergency Stop**" can be used if needed.

Open VIDEO program, Turn on the light as needed.
First, Zoom out camera may help you easily find your sample in the video window. Push "8" on controller it will display Z=x.xxxx, Push "↓" until x.xxxx close to 1. Use "+" or "-" will increase or decrease the speed of movement as needed.
Move X,Y to move interested area of your sample under the camera.
Move Z to focus on your sample. The laser spot should be in the center of video window once your sample is in focus.
Zoom in camera to Z=7.000 to fine tune your sample position.

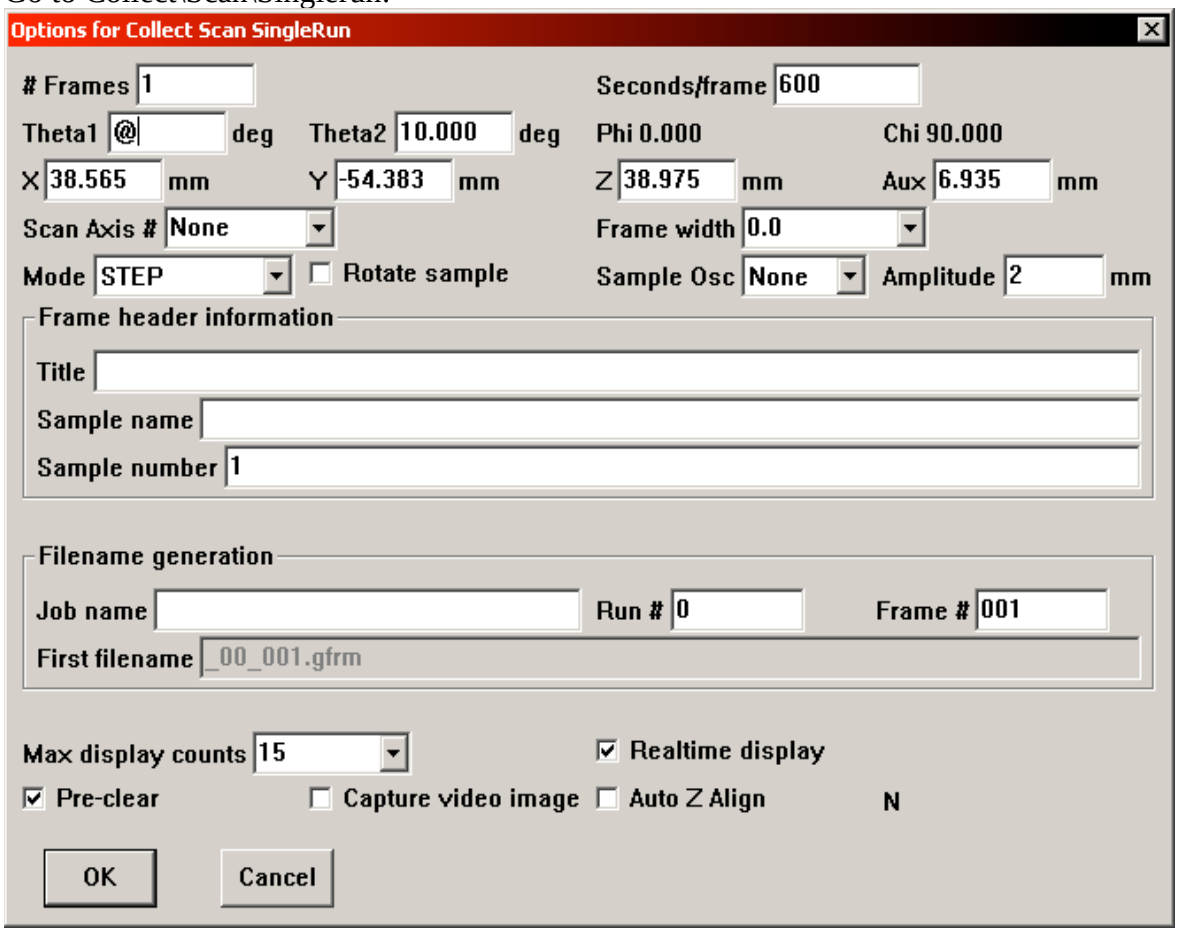
(Note: you also can use command to move axis. It is very useful if you want to move axes to exactly position.
!!! Warming: Always leave one finger on “STOP” button when you are using command mode. Press “STOP” button immediately if the moving part is going to hit other part.
On controller, press F1/DRVC and it will display Drive, command (axis, command, parameter). If you want to drive Zoom to 7, press 8,2,7, then ENTER. The camera will zoom in to 7. Press Shift+F1/DRVC to leave command mode.)

Once the Laser spot is in center, your sample is aligned and ready to take data. Turn off Laser by push “L” in GADDS program.

!!!Warming: You can use controller to move θ1 and θ2 to your desired angle. Please make sure detector will not hit by direct beam (usually θ1+θ2 >12° at 150 mm sample to detector distance)
To exit Manual control, press Esc.

6: Set up a scan and taking data:
For new user /new project, you need to go to Project\new, input info accordingly. Use working directory C:\frames\users\”unique name”\”project name”

Go to Collect\Scan\Singlerun:



Typical **SingleRun** setting for new user:
Frame:1
Time:300 sec
Use"@" for θ1, θ2, X, Y, Z, Zoom, it will display all current axis values.
!!!Warning: Make sure all axis values in the setting are the same as the actually positions before hit "Enter" or OK. Scan Axis: None
Frame width: 0
Mode:

Sample Osc: None
Amplitude:

Check file name which is "Jobname"_"Run#"_"frame#", change it as needed.
Pre-Clear: Check for Yes.

Once you are done with setting, click OK or Enter.
(!!! Do not hit "Enter" or "OK" before you finish setting or the scan will start and/or axes will move unexpectly)
To stop a scan in the middle of run: Ctrl+C
Data will be saved in .gfrm.

7: Frame intergration:

Go to Peak\Integrate\Chi. Click OK. Use 1,2,3,4 to change 2θ range and Chi range for integration. Then hit Enter.

Data can saved in the following File Formats:
Diffracplus: can be opened and processed by EVA or Jade software.
Plotso: Text file and can be opened with notepad

Note: For Powder diffraction, you can use script language (in slm file) to automate the data collections, integration and merge. The following is an example for collecting 2θ from 22 to 68 with 30cm of "Sample to Detector distance". You can modify this file to fit your sample.

```
!make sure sample is aligned.
!this program is to collect data from 21.5 to 68.5 in 2theta and for 3 frames and works.
!integrate the frames and save to a single file.
!%1=Title %2=NAME %5=Scan time for 1 frame

SCAN /SINGLERUN 3 /THETA1=15 /THETA2=15 /X=@5 &
 /Y=@6 /Z=@7 /AUX=@8 /AXIS=Coupled /WIDTH=15 /SCANTIME=%3 &
 /TITLE=%1 /SAMPLE=%2 /NUMSAMPLE=0 /NAME='%1'-'%2' /RUN=01 &
 /FRAMENO=001 /DISPLAY=7 /REALTIME /CLEAR /MODE=Step &
```

/OSCILLATE=None /AMPLITUDE=1

LOAD '%1'-'%2'_01_001.gfrm /DISPLAY=7 /SCALE=-n &
/OFFSET=0.0
INTEGRATE /CHI 21.5 38.5 -100 -80 /NORMAL=5 /STEPSIZE=0.02
INTEGRATE /WRITE %1 /FILENAME='%1'-'%2'_01 /FORMAT=DIFFRACplus
/APPEND/SCALE=1.0

DISPLAY /NEXT
INTEGRATE /CHI 36.5 53.5 -98 -82 /NORMAL=5 /STEPSIZE=0.02
INTEGRATE /WRITE %1 /FILENAME='%1'-'%2'_01 /FORMAT=DIFFRACplus
/APPEND/SCALE=1.0

DISPLAY /NEXT
INTEGRATE /CHI 51.5 68.5 -96.5 -83.5 /NORMAL=5 /STEPSIZE=0.02
INTEGRATE /WRITE %1 /FILENAME='%1'-'%2'_01 /FORMAT=DIFFRACplus
/APPEND/SCALE=1.0

system "merge /B /A /s '%1'-'%2'_01.raw /R1:3 '%1'-'%2'_merged.raw"

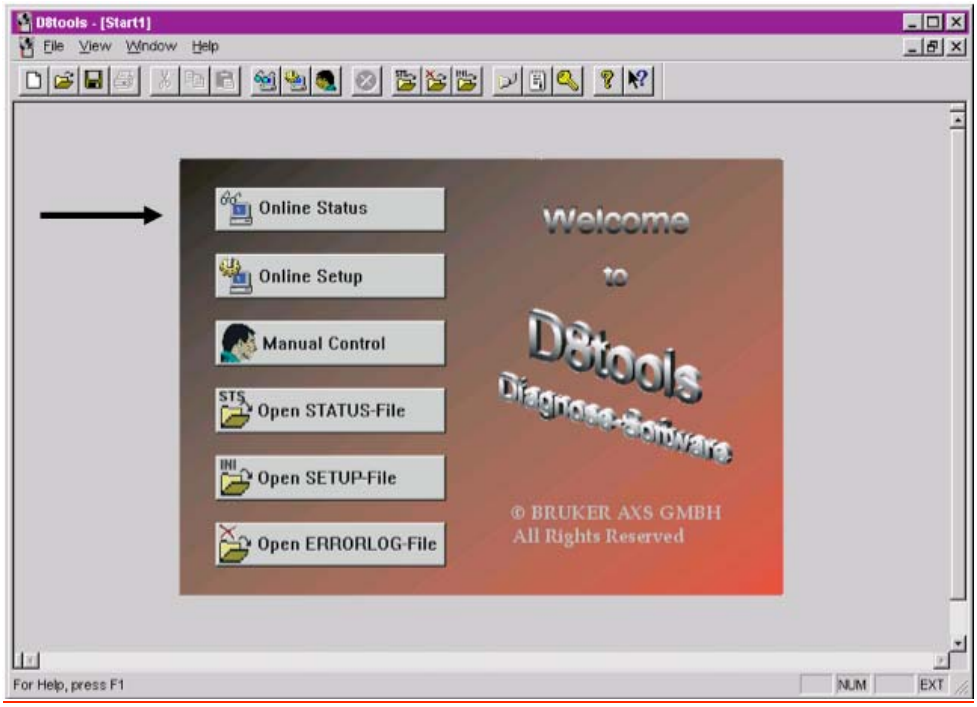
8: Take your sample out and close GADDS program.
Push “Open Door” button before your open glass window. Close window after taking out your sample.
To quit GADDS program, go to Project\Exit. Leave Generator at 20KV, 5mA

9: Sign out Log sheet.

Common Errors and Fixes:
There are many reasons that could cause D8 malfunctioning. Correct the problems before you restart the X-Ray. Program **D8 Tools** usually is used to troubleshoot and find out the fault parts.

X-Ray suddenly is turned off. (adapted from David Martin Group notes)
The general procedures to turn the X-ray back on:

1. Quit the GADDS program
2. Open the D8 tools program
3. Click Online Status



4. select “X-ray generator” , then “X-ray software safety circuit capture register”
5. Under “Utilities” select “Xray”, then “Reset Xray software safety circuit capture” (ignore warning if present)
6. Click OK. Password is “password”
7. Under “Utilities” select “Xray”, then “Reset Xray hardware capture register” (ignore warning if present)
8. Select “X-ray generator”
9. Under “Utilities” select “Xray”, then “Switch-off circuit ON”
10. HV switch on machine: Turn on Xray

Drive isn’t responding:

1. Open D8 Tools
2. Open DRIVE to affected drive
3. Select STOP icon on toolbar
4. Select from menu bar “Utilities”/”Pos. Drive”/Reset
5. That may be enough, but to be safe
   a. Note current position setting on drive
   b. Click “Settings” on drive pop-up menu (at bottom)
   c. Note reference position
   d. Use toolbar arrow down/up icon “reference down/up” to move current position to reference position. Once it has stopped moving, click double-arrow toolbar icon to right “adjust” to fine-tune it (get position to right of decimal point)
6. Close D8 tools.