Data Collection in

D8 Powder X-ray Diffractometer

(Short/Long Arm)

Step by Step Tutorial

D8-Short/Long Arm

Mounting the Sample

1. Sample preparation is one of the important steps in collecting the powder diffraction pattern. The choice of the sample holder depends on the amount of the sample you have and also on the nature of the sample. Several sample mounts, are available in the X-ray Lab. Choose the one that suits your need. If you need help regarding the sample holder choice, talk to an expert.

2. Once you are ready with the sample, mount the sample in the instrument. You must be trained by an expert to mount the sample.

3. Before opening the instrument door, check for any abnormalities in and around the instrument. Give particular importance to, the panel showing the X-ray power (which should read **40 kV 40 mA**), shutter lights, the 2θ arm, or the sample stage.

4. Now open the **INSTRUMENT DOOR,** and mount the sample

SAFETY CHECK: EVERY TIME YOU OPEN THE INSTRUMENT DOOR

Check if the Shutter Lights (in the X-ray tube of the instrument) are in the Shutter Closed Position

If you do not understand where to look for the shutter lights, talk to an expert

Data Collection Tutorial

1. Login in to your account.

Check again if there is anything abnormal about the instrument. The instrument door must remain closed now.

Check if there is any error in the instrument. Look for either flashing red lights on the side, or in the panel showing the X-ray power (which should read **40 kV 40 mA**). If it shows any error message, get help from an expert.
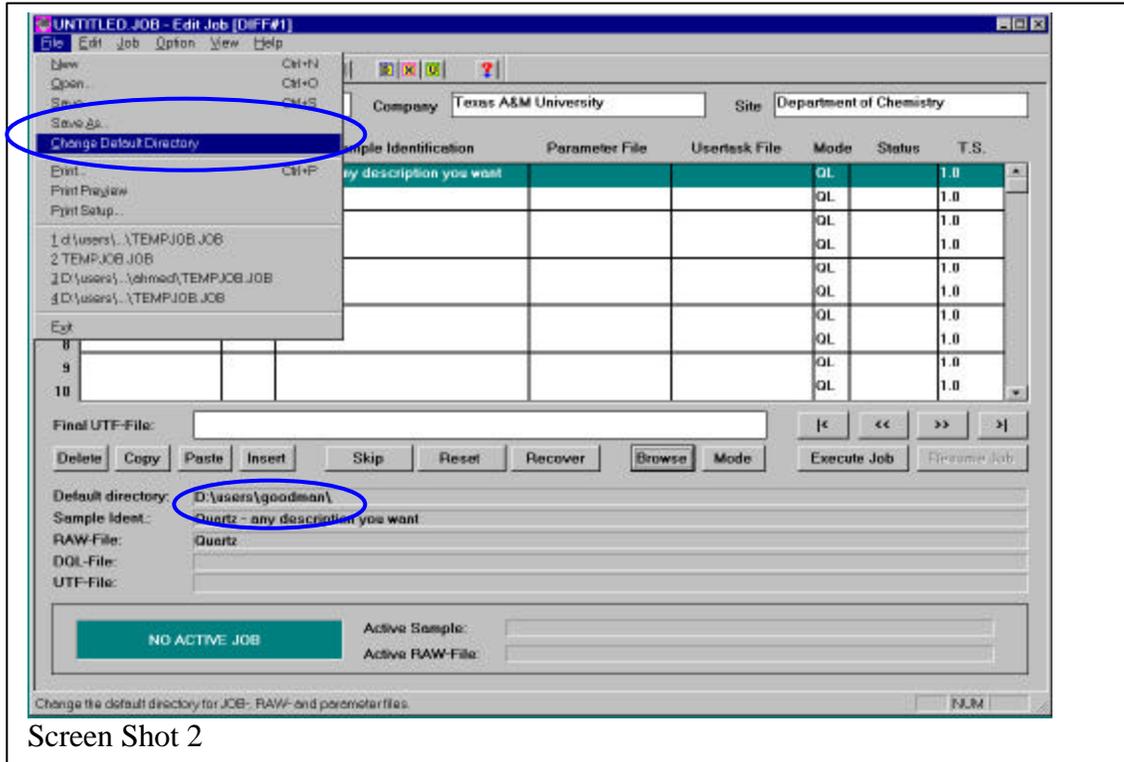
2. Now you can go to

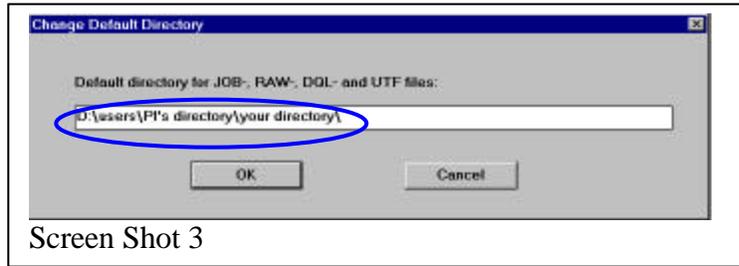- **Start ¾® PowderXRD ¾® JobMeasurement**.
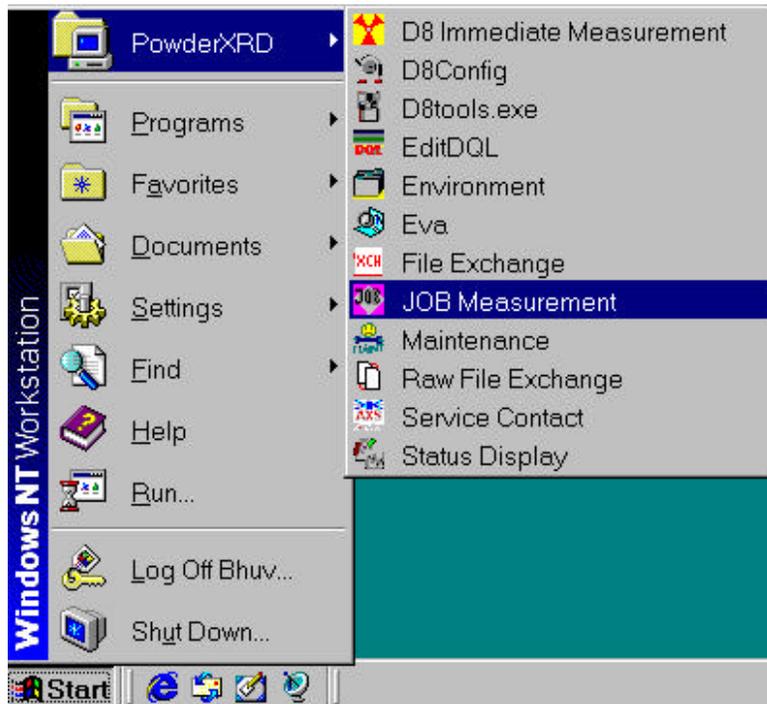
Screen shot 1

3. This will result in the following screen:

Screen Shot 2

Check for the Default directory: If it is not the directory you desire your files to go, then

change the default directory.

- **File ¾® Change Default Directory**

4. You will get the following pop-up screen

Screen Shot 3

Here enter the following D:\users\PI's directory\your directory\ (e.g.,

D:\users\Goodman\Lee\) and click OK.

Remember to insert the final backslash "\" at the end. If you miss the backslash, you will

have problems tracing your file. Also, your data collection may be overwritten, if you are

doing multiple samples.

5. Enter the file_name under **RAW-File** for your data, a brief_description under **Sample**

Identification.

Screen Shot 4
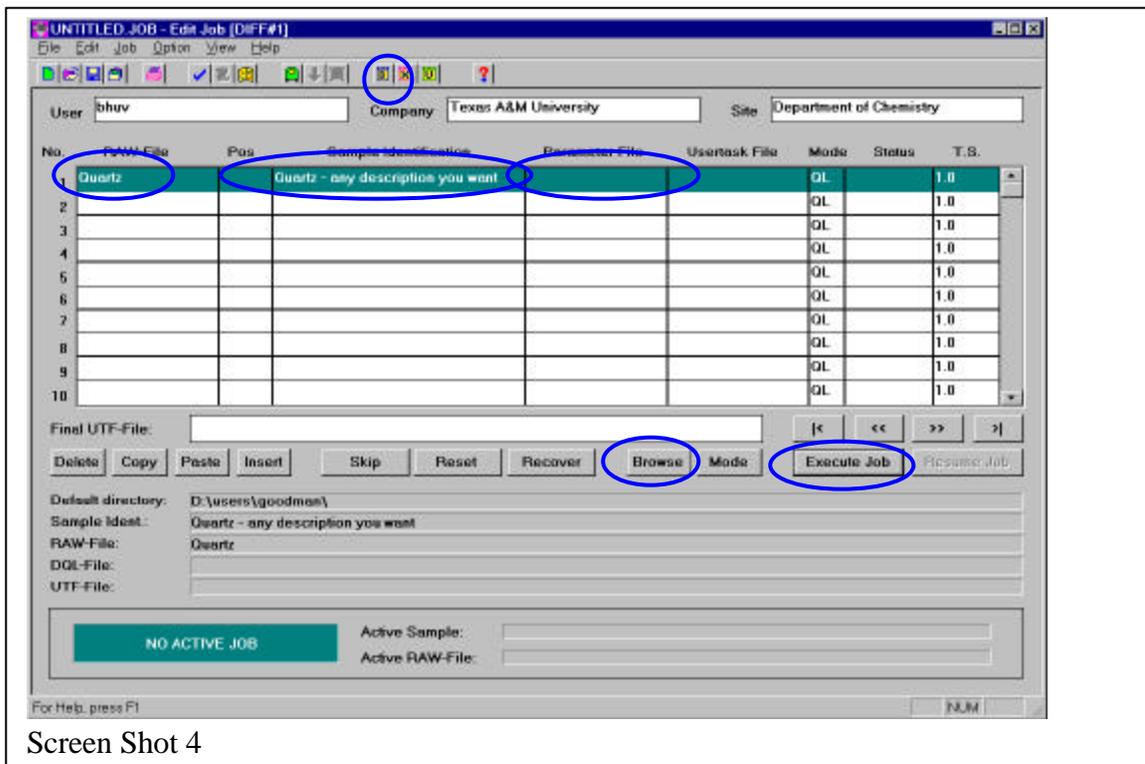
6. Select the .dql file under Parameter File (**Step 6a**)(if you have one ready). If you do not

have a file already made go to Step 7.

6a. Click on the space under the Parameter File, click on Browse, and select your .dql

file.

Click the button Execute Job
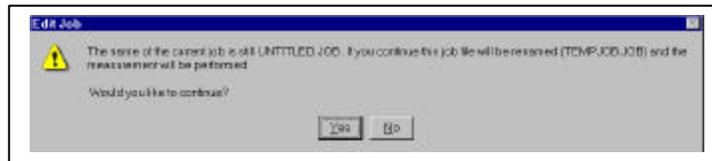
Say, yes to
this.
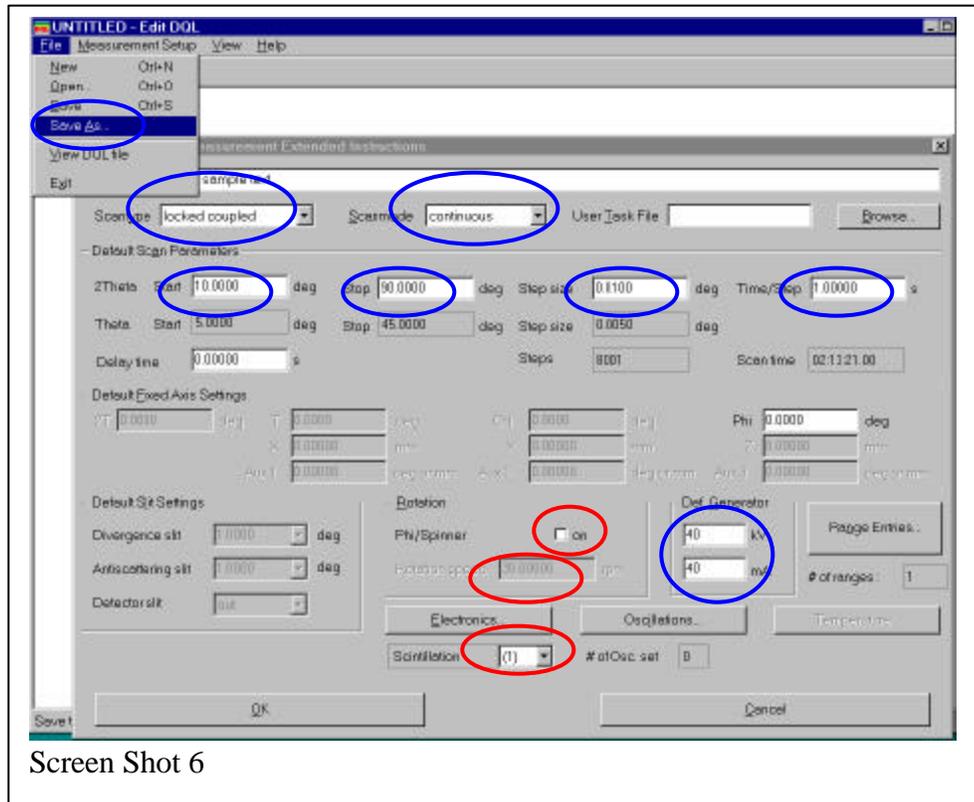
And your data collection Starts.

Go to **Step 9**.

7. If you do not have a Parameter file (.dql file) ready, click on the space under the Parameter File and then click on the button [D] . This will result in the following pop-up screen.

- **Measurement Setup ¾® Qualitative Extended**

Screen Shot 5

You will have

Screen Shot 6

Enter the **2Theta**

a. **Start** (minimum is 2, if you want to go below 2 please talk to an expert),

b. **Stop** (Usually 70 for patterns for identification purposes, could be upto 110),

c. **Step size** (usually 0.04, could be as low as 0.01 depending on your need), and

d. **Time/Step** (1 works fine for quick scan, use longer time for good data).

Check the following:

    a. Scan Type should be locked coupled. If it is not use the pull_down to select **locked coupled**.

    b. The **ScanMode** should be **continuous**.

    c. Def. Generator must be **40 kV** and **40 mA**.

For those using Long Arm instrument:

    a. Select the detector type (Just under the button for Electronics)

        1 represents Scintillation Counter

        3 represents a Position Sensitive Detector.

    b. Also you can choose to either spin the sample or not. If you choose to spin the sample, use a reasonable Rotation Speed (e.g., 5 to 10 rpm).
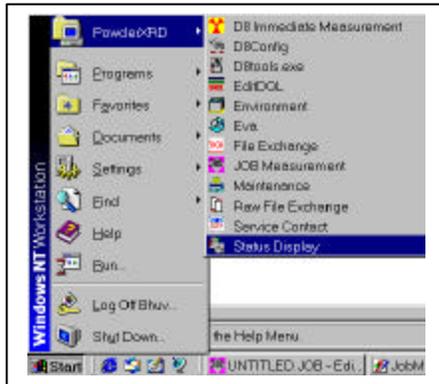
        i. File ¾® Save as ¾® give a file name ¾® close this window.

8. Now Go back to **Screen Shot 4**

Follow Step **6a**.

9. To see your data collection on-line,

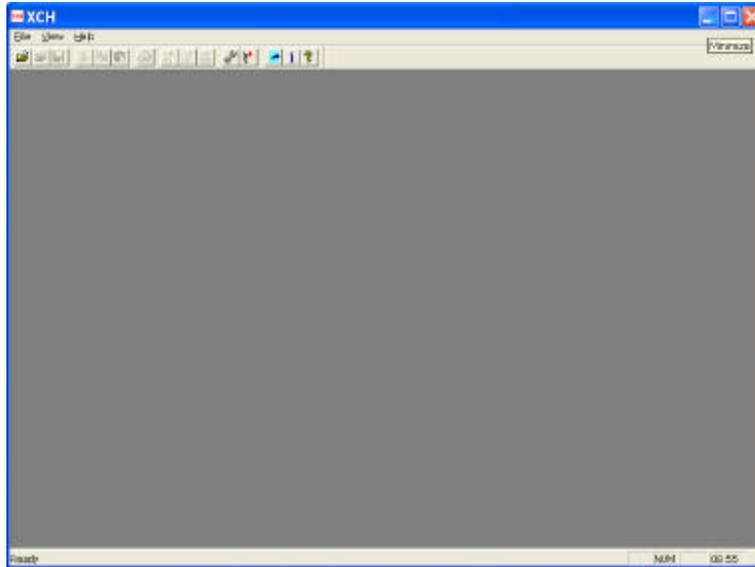Will result in a screen showing on-line data collection:
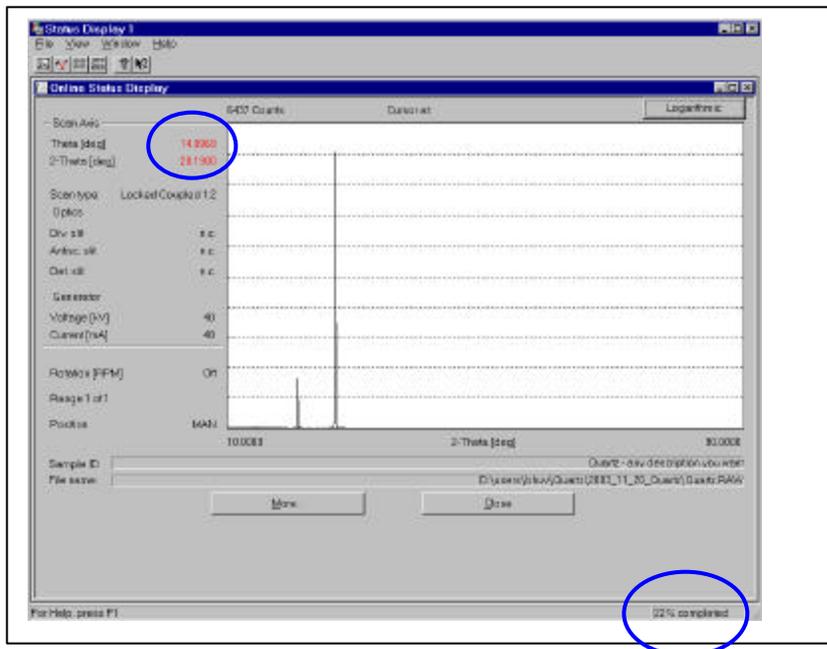
Two things of interest in this are your Theta and 2-Theta, to see where you are in your

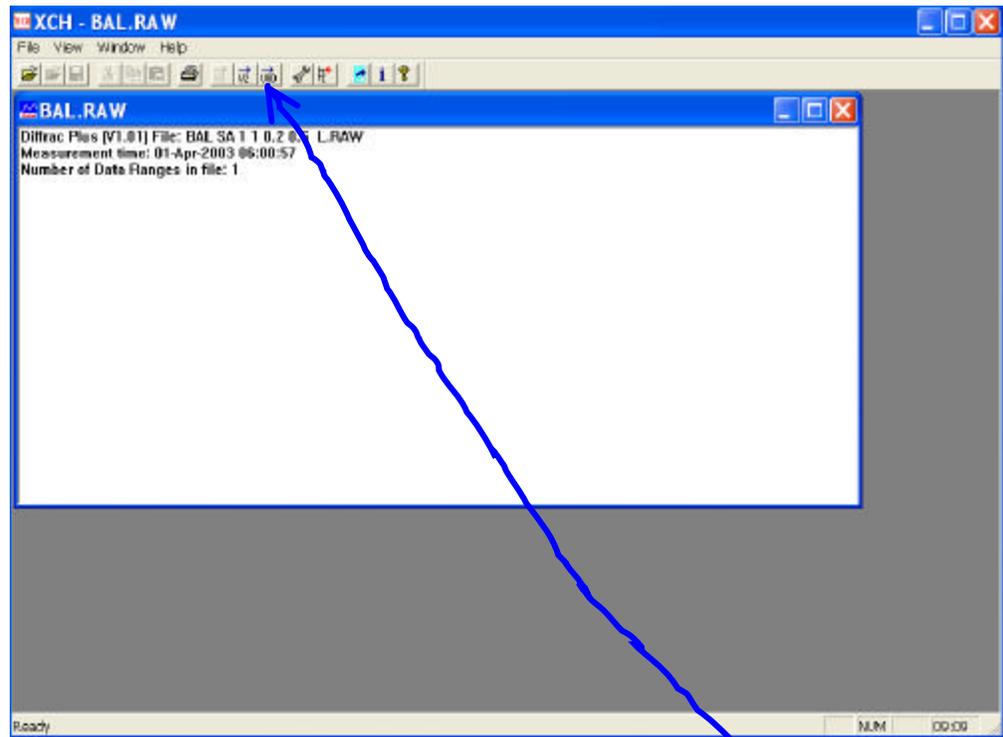measurement and the % completion. You can also find the peak position approximately.

To analyze the data, you can use EVA or other software.

To convert .RAW file to an ASCII (.UXD) file.
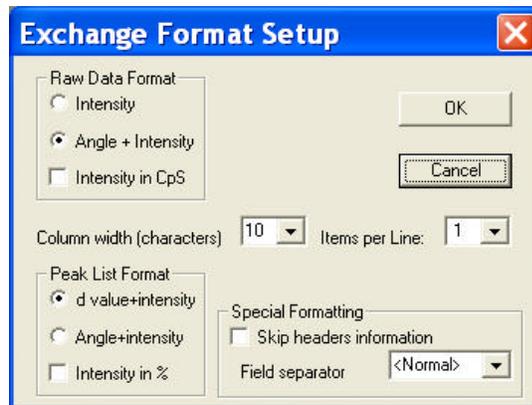
Open the Diffrac Files Exchange program.

- Open your .RAW (say BAL.RAW) file

- Check the format of the output file:

        File $\longrightarrow$ UXD format

Check if all the values are as given in the following screen.

If the screen you get is not matching to the above, change it accordingly.

- Click OK.

- Now click the icon

- Select the folder you want to save the ascii file (say, BAL.UXD), and save it.

The UXD file will contain several header lines which you may(!!!) or may not want followed by a two column data. The first column will correspond to Two-theta and the second the corresponding intensities.

Happy Powder Diffraction.

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