PHASE ANALYSIS
(Parallel Beam Optics)

I. Hardware Setup

1. Tube is in line focus.
2. Incident Beam Optics - **Mirror**:
   a. *Divergence Slit* - 1/2 °.
   b. Insert *Manual Attenuator - 115.7*.
   c. No *Mask*.
3. Diffracted Beam Optics – Parallel Plate Collimator **PPC**:
   a. No *Parallel Plate Collimator Slit*.

II. User Setup

4. Open the **Data Collector** Program.
5. Enter your **User Name** and **Password**.
6. Select *Instrument/Connect* in the **Data Collector**. The **Go OnLine** box will appear.
7. Select the **Mirror + PPC** for your optics.
8. Press the *OK* button.
9. Select the **Incident Beam** optics tab.
   a. Double click any item to prompt another window.
   b. Select the proper optic components in the new window.
   c. Set the **Automatic Beam Attenuator** to *Do Not Switch* and *Activated*.
   d. Press the *OK* button.
10. Select the **Diffracted Beam** optics tab.
    a. Double click any item to prompt another window.
    b. Select the proper optic components for the secondary beam side.
11. Press the **Instrument Settings** tab in the tree view.
    a. Double click any item in the tree view to prompt another window.
    b. Enter 0 in the *2Theta* field, 0 in the *Offset* field (this will set *Omega* to 0), 0 in the *Psi* field, and 0 in the *Phi*, *X*, and *Y* fields. Enter 5.0 in *Z* field.
    c. Press the *Apply* button to move to these positions.
    d. Press the **X-ray** tab. Set the generator power to 45 kV and 40 mA.

III. Zero Alignment

12. Select **Measure/ Manual Scan** from the main menu.
13. Enter the following parameters:
    a. *2Theta* in the *Scan Axis* field.
    b. Enter *0.01* in the *Step Size* field.
    c. Enter *2* in the *Range* field.
    d. *0.1* in the *Time per Step* field.
    e. Press the *Start* button.

f. Wait until scan is completed (greenish hairline appears in the graphic window at diffractometer's initial position).

14. If center of the peak is off from diffractometers zero value, the following has to be done (otherwise skip to Step 14):
    a. Press the right mouse button and select *Move* mode.
    b. Press and hold the left mouse button until the cursor is placed over the center of gravity of the peak. This will move the goniometer's *2Theta* to the selected position.
    c. Select the **Tools/ Sample Offsets** from the **Main Menu**.
    d. Enter *0* in the *2Theta* field.
    e. Close this window.

IV. Sample Preparation and Mounting

15. Prepare powder Sample:
    a. Place a glass slide on the tissue paper.
    b. Using applicator tip, spread silicon grease on the slide. Grease patch should be elongated and thin.
    c. Sprinkle enough powder on applied grease.
    d. Use another glass slide to lightly press powder into the grease. Lift the slide.
16. Put tissue paper on the bottom of goniometer. This will prevent powder from dripping into goniometer.
17. Use single sided sticky tape to mount sample on the stage. Long direction should be vertical.

V. Moving Sample into the Beam Position

18. In the **Instruments Settings** window, double click any item in the tree view to prompt another window.
19. Press the **X-ray** tab.
    a. Check the *Shutter Open* box.
    b. Click *Apply*. Note the Intensity value at the lower part of the main window.
20. Press the **Position** tab.
    a. Adjust the *Z* position of the sample stage until the counts read 50% of the value noted in step 19_b.
21. Press *OK*.

VI. Setting the Automatic Attenuator

22. Select the **Incident Beam** optics tab.
23. Double click **Attenuator** item to prompt another window.
24. Set the **Attenuator/Usage** to *Preset Intensity*. The *Activate* level should be *500,000* and the *DeActivate* level should be *450,000*.
25. Press the *OK* button.

VII. Performing the Measurement

26. Remove *Manual Attenuator - 115.7*.

27. Select the **Incident Beam** optics tab.
28. Double click **Positions** item to prompt Positions window.
29. Move *2Theta* to *80.00* **AND** *Omega* to *40.00*.
30. Click *OK*.
31. Select **Measure/Manual Scan**.
32. Enter the following parameters:
    a. *2Theta - Omega* in the *Scan Axis* field.
    b. Enter *0.05* in the *Step Size* field.
    c. *100* in the *Range* field.
    d. *1.0* in the *Time per Step* field.
33. Press the *Start* button.
34. After measurement is completed press **File/Save As** and save the file under apropriate name.
35. Close graphic window. Shutter closes.
36. Remove sample.
37. Prepare another sample and measure using the procedure listed above.

VIII. Completing the Lab session

37. Close graphic window.
38. Press the **Instrument Settings** tab in the tree view.
39. Double click any item in the tree view to prompt another window.
    a. Enter *0* in the *2Theta* field, *0* in the *Offset* field (this will set *Omega* to *0*), *0* in the *Psi* field, and *0* in the *Phi*, *X*, and *Y* fields. Enter *5.0* in *Z* field.
    b. Press the *Apply* button to move to these positions.
    c. Press the **X-ray** tab.
    d. Set the generator power to 40 kV and 10 mA.
    e. Click *OK*.
40. Close **Data Collector**.
41. Remove sample and tissue papers.

IX. Convert file to "*.csv" format:

42. In **File Manager** find your file (**Sample*X*.xrdml**) under **C:\Users\Group*X*** folder.
43. Right mouse click on the file. Select *Convert*.
44. Uncheck all options except *csv*.
45. Make sure *Convert To* is pointing to your directory.
46. Press *Convert*.