

Using the X-Ray Diffractometer

0. Warnings

- A. Be very careful not to expose the roll of x-ray film, even when closed, to light.
- B. Be careful with the developing chemicals, read warnings on label, avoid slashing on cloths.
- C. Do not operate the x-ray unit if the shielding is not in place OR you are not trained
- D. Be careful working in the dark. Practice working with safelight on.

I. Alignment of Camera to x-ray port.

- A. X-ray unit OFF
- B. Remove end of lead shielding
- C. Remove entrance and exit cones from camera
- D. Install camera
- E. Sight through camera and align so that the line through the center of the entrance and exit, meets the center of the x-ray port
- F. Check that the manual shutter is open and the correct filter is in place.(For Cobalt x-ray tube use an Fe filter that will block some wavelengths of the emitted x-rays.)

II. Sample Preparation

- A. Measure ID (Inside Diameter) of the camera. (needed for data analysis later.)
- B. Use a very fine powder. Use a mortar and pestle if necessary.
- C. Put some kind of adhesive (water, glue, . .)on the glass-rod sample holder, avoiding end to be placed in mount on the camera
- D. Roll glass rod in the powder and get it covered.
- E. Install glass rod in mount on the axis of the camera

III. Film Preparation

- A. Practice this before doing for real.
- B. **ALL film handling should be done in TOTAL DARKNESS**
- C. Carefully assemble all you equipment and parts in the darkroom. Organize them in such a way that you can find them in the dark. (The order of use seems like a starting point.
 1. Camera with lid off, opening toward you, entrance and exit cones removed . Camera lid and entrance and exit cones near camera.
 2. Film cutter/punch ready, stop pin in correct hole.
- D. **All darkroom lights out!**
- E. **Handle film only by its edges.**
- F. Open cabinet, remove square box (about 4" X 4"X 1½"), open box, unwrap film
- G. Cut film to length
- H. Immediately Rewrap roll of film, rebox film, put box in cabinet, close cabinet.
- I. On film cutter/punch move pin to punch position. Punch one end of film, rotate end-for-end and punch other end.
- J. Place film in camera, cut ends up. Catch on little hooks at top, use tightening thing to force film against camera's inside surface.
- K. Install camera's lid and end cones
- L. Cover camera before bringing it out of the darkroom
- M. Have the main room lights off.

IV. Exposure

- A. Install camera entrance cone toward x-ray port.
- B. Connect drive belt, plug in rotator drive motor and check that the sample holder part is rotating.
- C. Install door on shielding,
- D. Put radiation sign just outside of the door

- E. Turn on X-ray unit **REQUIRES trained operator with radiation badge and key.**
 1. Cooling water ON at manual valve. Key in and turn, Set unit timer to 3 hours
 2. Push START (as in startup) button (clunk from cooling water valve)
 3. Push ON (as in x rays ON) button (twin green lights, clunk, delay and then x ray red light)
 4. Turn up Voltage to 35 KV and Current 20 mA.
 5. Use shutter timer, Set for 1-2 hour as needed, Open shutter
 6. Guard the room while the unit is on.
- F. When exposure is complete: Turn Voltage and Current all the way down. Push OFF button
Do **NOT** push STOP button yet, so cooling water stays on.
- G. Record usage.

V. Developing the Film

(see: <http://www.kodak.com/US/en/health/scientific/products/autoradFilms/tips.shtml>)

- A. Practice this before doing for real.
- B. **ALL film handling should be done in TOTAL DARKNESS**
- C. **Handle film only by its edges.**
- D. Prepare your chemicals and developing equipment.
- E. **All darkroom lights out!**
- F. Open the Camera and remove the cones.
- G. Place film in developer for 5 minutes (use timer), use tongs to insure it is totally submerged
- H. Rinse film in flowing water for 1 minute (timer or count)
- I. Place film in fixer for 5-10 minutes (use timer) agitate every 30-60 second, make sure film is totally submerged.
- J. Rinse film in flowing water for 1 minute (timer or count)
- K. Take film to main room, look at it, and celebrate or curse as appropriate.

VI. Shutdown

- A. X-RAY UNIT
 1. Push STOP button (Clunk as internal water valve closes.)
 2. Check Voltage and Current dials are fully counterclockwise.
 3. Turn key off and remove key, Return key to the *keeper of the key*
 4. Turn OFF the water at valve.
- B. Record usage. if not already done.
- C. X-ray sign back in room
- D. Developing chemical back in bottles
- E. Darkroom equipment washed dried, put away.
- F. Dark room counters clean and dry.
- G. Darkroom Light OFF
- H. Shielding back together.
- I. X-ray camera and film cutter/punch stored.
- J. Main room cleaned up.