

X-Ray Production: General Overview of Apparatus

This lab serves as an introduction to the production of X-rays in the laboratory and how they are used in scattering experiments to determine the physical structure of solids. In this lab we will learn how X-rays are produced by the impact of high energy electrons on a metal target. We will determine the X-ray wavelengths by scattering the X-rays from a reference crystal with a known lattice-plane separation distance (d -spacing) using the technique of Bragg Scattering and subsequently measure the d -spacing for an unknown crystal. Finally we will investigate the attenuation of X-rays in different metals and in different thicknesses and thereby see how to obtain a monochromatic source of X-rays.

Preliminary Caution: *Exposure to X-rays can be dangerous, especially from prolonged exposures. The X-ray machine used in this lab has built-in safe guards to ensure that when in use, the exposure is minimal. The X-ray tube itself is enclosed behind a leaded-glass window to absorb the X-rays that are not along the beam path. In addition the large scatter shield cover has an Al/Pb back stop to prevent the direct beam from exiting the scatter shield. In addition, the leaded-glass window has a special interlock feature that will prevent the X-ray tube from being turned on when the window is open.*

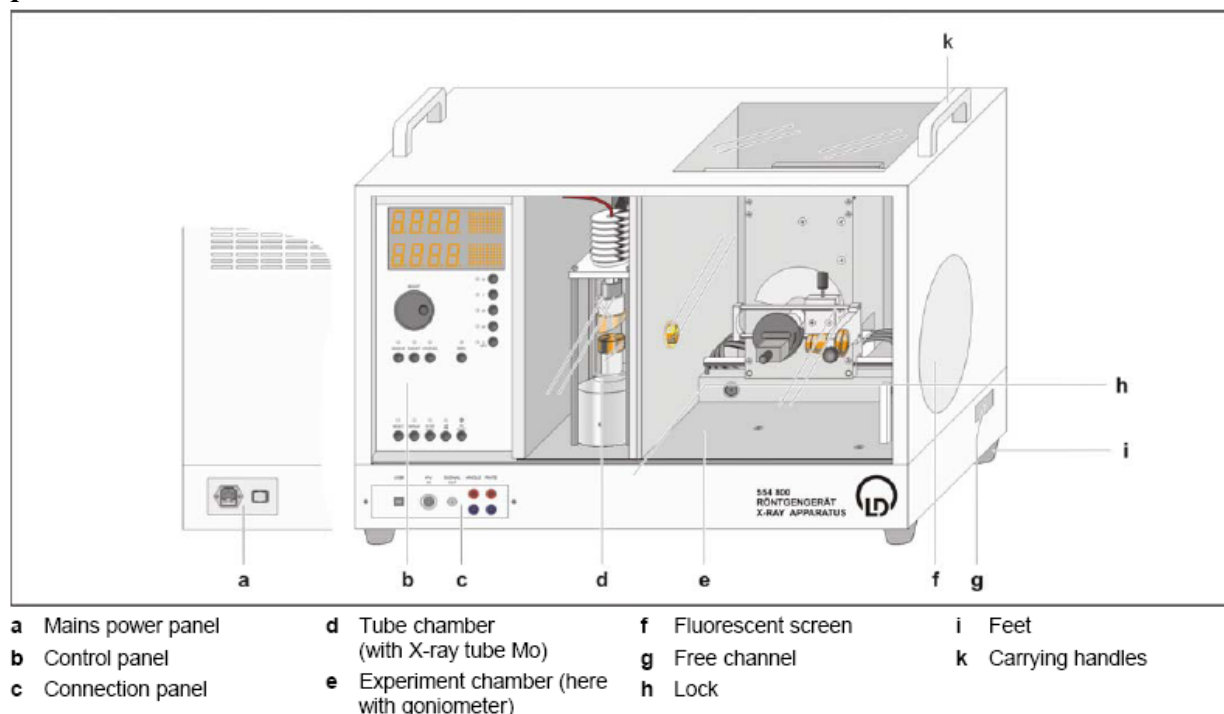


Figure 1. Important components of the X-ray apparatus (taken from Leybold-Didactic Instruction Sheet 554 800).

Part 1: X-Ray Apparatus

Figure 1 shows the major features of the X-ray apparatus that we will use for this experiment. The major components are the X-ray tube housed in the tube chamber (d) and the goniometer housed in the experiment chamber (e). The apparatus is interfaced to a computer via a USB cable and allows the user to control the power to the X-ray tube and the position and scan settings for the goniometer. Inspect the apparatus while reading sections 2, 6, 7 and 8 of the *Instruction Sheet 554 800* so that you understand the apparatus and its safety features.

As an initial check, follow the instructions below to familiarize yourself with the operation of the apparatus.

First time operation of X-Ray Tube

1. Make sure the X-ray apparatus is plugged into AC and switch on the device.
2. Press the key *U* to display the X-ray tube voltage.
3. Use the ADJUST knob to set the high voltage to 20 kV
4. Press the key *I* to display the X-ray tube emission current.
5. Use the ADJUST knob to set the emission current to 1.00 mA
6. Check to make sure that the lead-glass sliding windows are properly closed, then press the HV ON/OFF key – the high voltage indicator starts flashing and the hot cathode of the X-ray tube becomes luminous. The system is now generating X-rays.
7. Press the key *I* and use the ADJUST knob to vary the emission current *I* – the brightness of the hot cathode should change.

Use a radiation survey meter to survey the apparatus with the X-ray tube both off and on to make sure the apparatus presents no safety hazard with respect to exposure.

Part 2: Goniometer

The goniometer is a self-contained unit mounted in the experiment chamber which is equipped with two independently controllable stepper motors for rotating the target holder and sensor arm. The positioning of the target and sensor arms is controlled by manual controls on the control panel (b) or by the interface program X-Ray.exe which can be started by double-clicking the desktop icon on the computer.

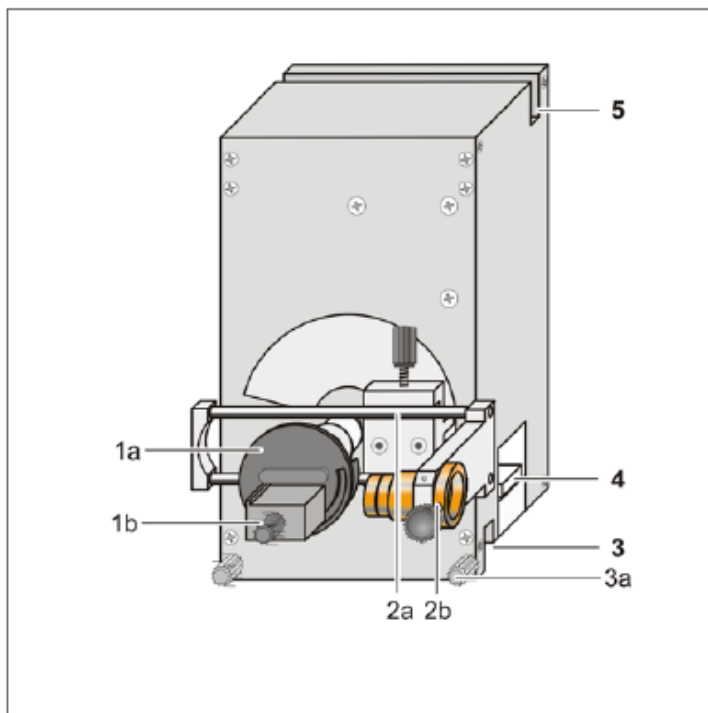


Figure 2. Goniometer apparatus. (taken from Leybold-Didactic Instruction Sheet 554 831)

- 1 **Target arm**
with target holder (1a), target stage (1b)
- 2 **Sensor arm**
with sensor holder (2a), sensor seat (2b)
- 3 **Bottom guide groove**
with knurled screws (3a)
- 4 **Terminal pin connector**
- 5 **Top guide groove**

The target stage and the sensor arm can be rotated independently or in a coupled mode. When placed in SENSOR mode, only the sensor arm moves when the ADJUST knob is rotated. Likewise when placed in TARGET mode, only the target stage moves when the ADJUST knob is rotated. In COUPLED mode, the target and sensor arm are rotated together in such a manner that (sensor angle) = 2 X (target angle).

Manually Positioning the Goniometer Arms

1. Press either the SENSOR or TARGET keys. [NOTE: the TARGET key doesn't seem to work, so skip that part.]
2. Set the desired value with the ADJUST knob – the set value appears in the display panel and the sensor or target arm moves to the desired angular position.

-OR-

1. Press the COUPLED key
2. Set the desired target value with the ADJUST knob – the set value appears in the display panel, the target arm moves to the desired angular position and the sensor arm automatically moves with twice the angular step width.

Positioning the Goniometer Arms Using the Computer Interface

1. If you haven't done so already, start the interface program by double-clicking the "XRay" icon on the desktop. NOTE: every time the x-ray device is turned off and then back on, you have to re-start the "XRay" program.
2. Select the "Bragg" tab for the Bragg scattering experiment and then select the "Settings" option from the menu bar.
3. In the Settings window select the "X-Ray Apparatus" tab
4. Press either the SENSOR or TARGET keys and set the desired angle by typing an angle (in degrees) in the box labeled " β =" – the set value appears in the display panel and the sensor or target arm moves to the desired angular position.

Installing Crystal on Target Stage

1. Position the target and sensor arms of the goniometer horizontally using the ZERO key.
2. Loosen the knurled screw of the target stage and lower the platform slightly.
3. ***Never touch the crystal samples with your bare fingers!*** Using the plastic tweezers, place the crystal flat on the target stage and push it backwards against the back stop.
4. Raise the target stage with the crystal up against the stop edge and tighten the knurled screw finger-tight.
5. Carefully check that the target is held firmly.

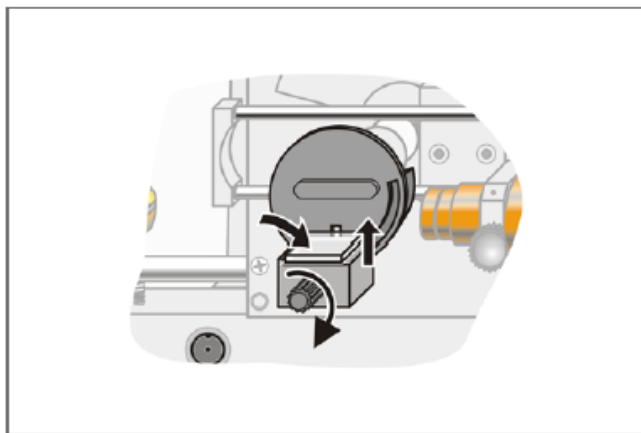


Figure 3 Mounting crystal on target stage.
(taken from Leybold-Didactic Instruction
Sheet 554 831)

Part 3: Zero Position of Goniometer

The zero position of the goniometer is determined by the target stage and arm position coincident with light sensors in the stepper motor apparatus, referred to as “light barriers.” Corrections to the light barrier zero position can be made relative to diffraction peaks from crystal lattice planes and stored in the memory of the X-Ray apparatus.

This setup uses the NaCl monocrystal to determine the goniometer zero position corrections.

(Note: the following steps 1-8 for manually zeroing the device appear not to work. Skip over this to the following.)

- ~~1. Press the ZERO key to return the target and sensor arms to the current zero position.~~
- ~~2. Mount the NaCl crystal on the target stage (see “Installing Crystal on Target Stage” above).~~
- ~~3. In coupled scanning mode, set the target to about 7.2° using the ADJUST knob~~
- ~~4. Set the X Ray tube high voltage $U = 35.0$ kV and emission current $I = 1.00$ mA and switch on the tube high voltage with HV ON/OFF.~~
- ~~5. Switch between sensor and target scanning modes and locate the maximum counting rate for the first reflection maximum of the NaCl monocrystal under manual control.~~
- ~~6. In coupled scanning mode, move the target back by 7.2° (even if this takes you into the negative range!).~~
- ~~7. Save the positions of the target and the sensor as the “zero position of the measuring system” by pressing TARGET, COUPLED and β LIMITS simultaneously.~~
- ~~8. To verify your adjustments, assume the angle 7.2° in coupled scanning mode and check the maximum counting rate.~~

Instead of zeroing the device manually, the “XRay” program on the computer can handle the zeroing. Click on the “settings” icon, then “crystal calibration” and then “start scan.” The device will automatically explore the count rate for various angles for both the sample holder and the detector until it locates the combination with the largest count. If you then click “Adopt”, it will re-zero the device such that the sample holder is treated as the angle 7.2° and the detector is treated as twice that angle.

NOTE: each time you put the crystal on the sample holder, you should re-do the automatic zeroing. It makes a noticeable difference in the quality of the data that you will collect with the device.

Part 4: Setting Up Automatic Scans

In the auto-scan mode, the goniometer arms move automatically when the SCAN key is depressed or the SCAN option is checked in the software interface. You can choose between scan modes “Target”, “Sensor”, or “Coupled”. All of the parameters needed to set up a scan can be controlled by using the XRay software control program. Besides setting the high voltage (U) and emission current (I) the following parameters must be set for a scan:

- Δt – time interval for counting x-rays at each angular position of the sensor
- $\Delta\beta$ – angular step size for sensor arm
- $\beta_{\min}, \beta_{\max}$ – lower (min) and upper (max) limit angles for the sensor arm

Setting Up an Automatic Scan Using the Computer Interface

1. If you haven't done so already, start the interface program by double-clicking the "XRay" icon on the desktop.
2. Select the "Bragg" tab for the Bragg scattering experiment and then select the "Settings" option from the menu bar.
3. In the Settings window select the "X-Ray Apparatus" tab
4. For Bragg scattering scans, select "Coupled" mode and enter the parameter values required for the scan
5. To start the scan, click on the "Scan" check-box – NOTE: If you need to stop the scan before it is finished, click on the "Scan" check-box again and the scan will stop and the x-ray tube will be shut down.