

PICTORIAL GUIDE TO THE X-RAY DIFFRACTOMETER

1/10/02

The cardinal rule with the X-ray diffractometer is "be gentle; do not force anything". If something is meant to come apart it will do so with a light touch, assuming that the proper procedure is followed. Using excessive force risks misalignment of the diffraction system or damage to one of its components. In either case, the system will be out of commission and the remedy is costly.

The **Haskris Model R100** recirculating water chiller provides cooling water to the X-ray generator. The operator should be familiar with the following parts and controls:

- a. thermostat control
- b. ON/OFF switch and indicator lights
- c. flow meter for cooling water
- d. temperature and pressure gauges for cooling water
- e. supply and return valves for cooling water
- f. condenser water drain hose
- g. condenser water supply hose
- h. supply and return hoses for cooling water to and from the generator

The valve for the condenser water supply hose is just out of view to the right in this photo.

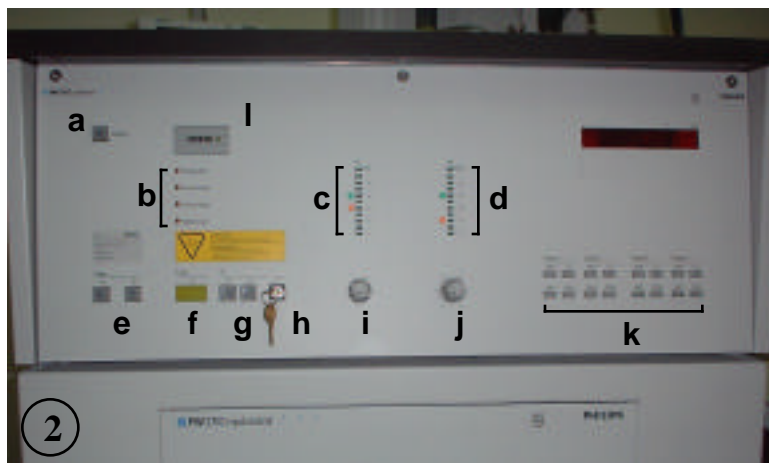
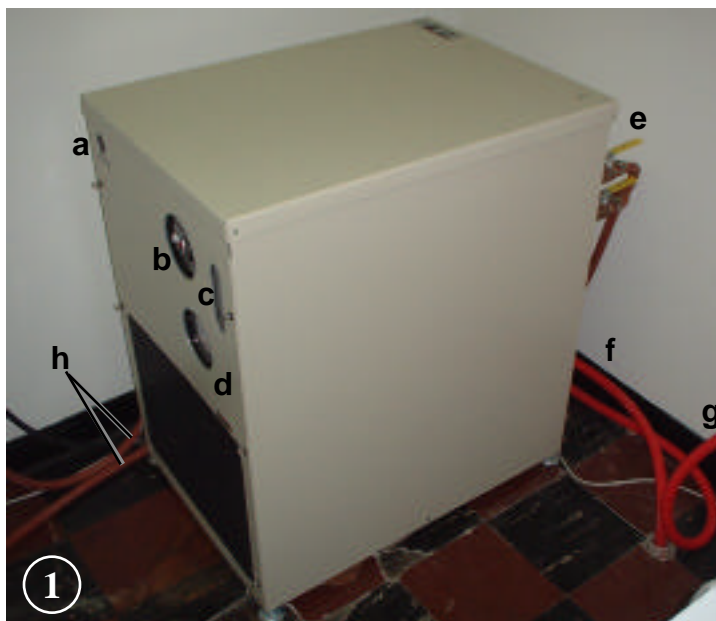
In general, all water valves related to the water chiller should be left OPEN.

When ever the X-ray generator is ON the water chiller must also be ON with all valves OPEN.

Controls for the **PW 1830** X-ray generator are as follows:

- a. shutters button
- b. fault indicators
- c. tension (kV) indicators
- d. current (mA) indicators
- e. power ON/OFF buttons
- f. X-ray ON lamp
- g. HT ON/OFF buttons
- h. HT lock key
- i. tension (kV) adjustment knob
- j. current (mA) adjustment knob
- k. shutter control buttons
- l. instrument hour meter

Refer to the start-up procedure for the operation of each control.



PW 1830 control panel

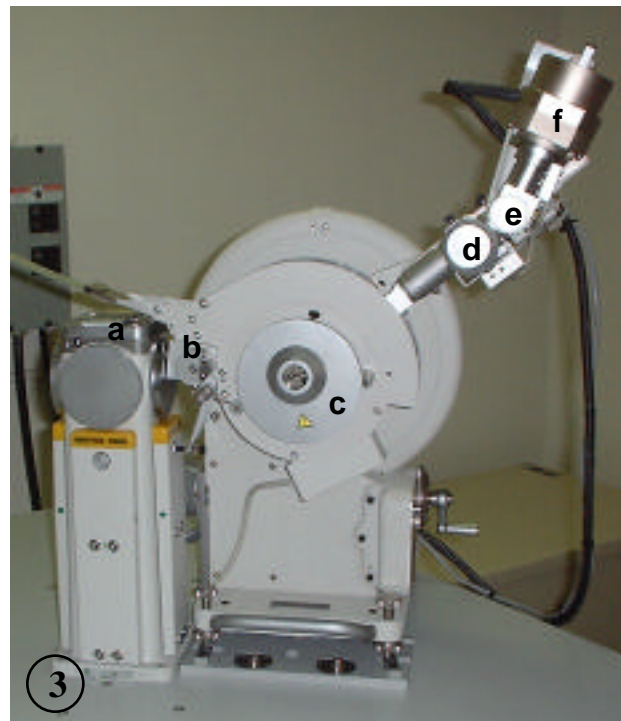
The X-ray tube is contained in the X-ray tube shield or X-ray "tower" (a), which is equipped with shutters for each of 4 ports. This is where X-rays are actually generated. Our diffraction system is set up to utilize X-rays from port (shutter) #4.

Other important parts of the **diffraction system** include:

- b. divergence slit assembly
- c. sample chamber
- d. receiving/scatter/soller slit assembly
- e. secondary monochromator
- f. detector

Parts of the system between the X-ray tower (a) and the specimen chamber (c) relate to primary or "incident" beam optics; parts of the system between the specimen chamber (c) and the detector (f) relate to secondary beam optics.

Our diffraction system is also fitted with a primary β filter, which is not used in conjunction with the secondary monochromator. The β filter disk lies between the X-ray tower (a) and the divergence slit assembly (b).



The **X-ray tube instruction card**, located at the top of the X-ray tower, indicates the target material and the power and tension ratings of the tube.

The target material in this tube is copper (Cu); the power rating is 2200 watts; and the tension (accelerating potential) rating is 60 kV.

The operator should be familiar with the relationship

$$P = V \times A$$

where P = power in watts, V = volts, and A = amperes. Thus, a limit of 2200 watts with 60,000 volts limits the current to less than 0.037 A, or 37 mA.

Routine analyses should be performed using considerably less than the rated power; 40 kV and 40 mA, i.e. 1600 watts, are normally sufficient.



When using the diffraction system, two dangers must be considered carefully. These are **X-RAY RADIATION** and **HIGH VOLTAGE**.

The diffraction system has safety circuits that are designed to protect the operator from both of these dangers. Nonetheless, **check to be sure that the shutter is closed (or X-rays are off) before opening or dismantling any part of the diffraction system.** Shutters are controlled from the PW 1830 control panel.

The mechanical device (interlock mechanism) shown here keeps the shutter button (g) depressed as long as the sample chamber lid (h) is in place. If the sample chamber lid is removed, the mechanism pivots counterclockwise (arrows), permitting the shutter button to come out, which causes the shutter to close. The interlock mechanism also prevents the divergence slit cover (i) from being opened unless the sample chamber lid is removed. Thus, access to the divergence slit assembly is also protected.

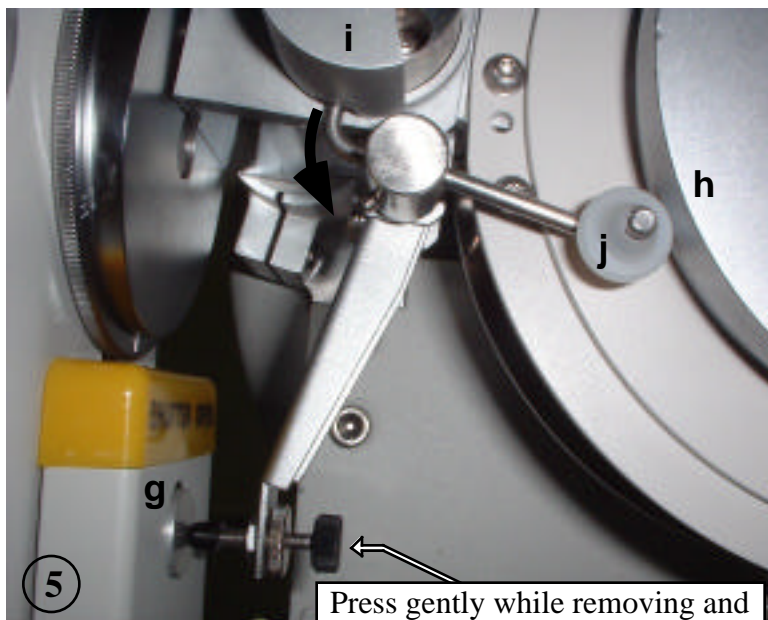
When opening the sample chamber, gently support the lid with one hand and loosen the knurled set screw (visible in photo 3) with the other hand. Then, reposition your hands such that the black, knurled adjustment screw on the interlock mechanism can be gently pressed as the lid is removed. This will pull aside the nylon roller (j).

The sample shaft (k) has a flat bottom surface and a spring clip (not visible) that holds the PW1172/01 sample holder (l) against it. The spring clip should be gently pulled down with a finger tip as the sample holder is inserted or removed.

It is important that bearing surfaces of the sample shaft (under side) and sample holder (top side) are clean and that the top surface of the pressed powder is flush with the top surface of the sample holder.

This photo shows a second sample holder at the entrance to the sample chamber as though to be inserted. The sample shaft holds only one sample at a time.

SAFETY CONCERNS



Press gently while removing and replacing the sample chamber lid.



Closing the sample chamber is the reverse of opening it. Be certain to engage the registration pin (*m*) and to gently press the black, knurled adjustment screw on the interlock mechanism before closing the lid. This will pull aside the nylon roller (*j*). Also, make sure that the roller rests against the larger diameter section of the lid and that the interlock mechanism engages the divergence slit cover correctly.

Do not over tighten the knurled set screw; this need only be snug (barely finger tight).

As described above, the sample chamber lid must be removed before the divergence slit cover can be opened. With the sample chamber lid removed and the interlock mechanism pivoted out of the way, rotate the divergence slit cover (*i*) 180° clockwise. The divergence slit (*n*) is inserted into or removed from its slot as shown. Do not insert the divergence slit too far; be careful not to pass the detent (click stop).

Closing the divergence slit cover is the reverse of opening it.

Note that, in this photo, the sample chamber (behind the hand) is open.

To open the receiver/scatter/soller slit assembly, loosen the knurled set screw a few turns, gently rotate the lid about 30° counterclockwise, and gently pull the lid straight off.

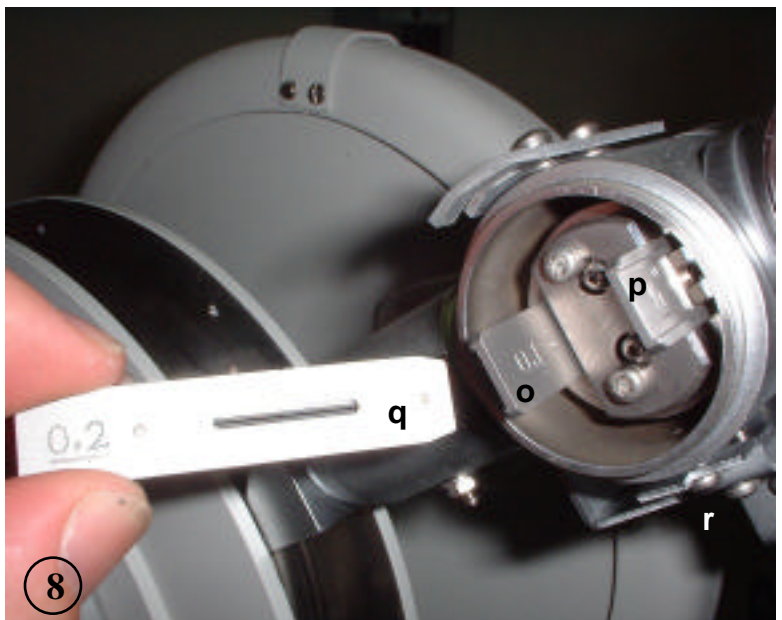
On our diffraction system the receiving slit (*o*) is toward the specimen; the scatter (antiscatter) slit (*p*) is toward the detector.

A 0.2 mm receiving slit (*q*) is also shown (held by finger tips) as though to be inserted. This assembly will hold only one receiving slit and one scatter slit at a time.

Do not insert the scatter slit too far; be careful not to pass the detent (click stop).

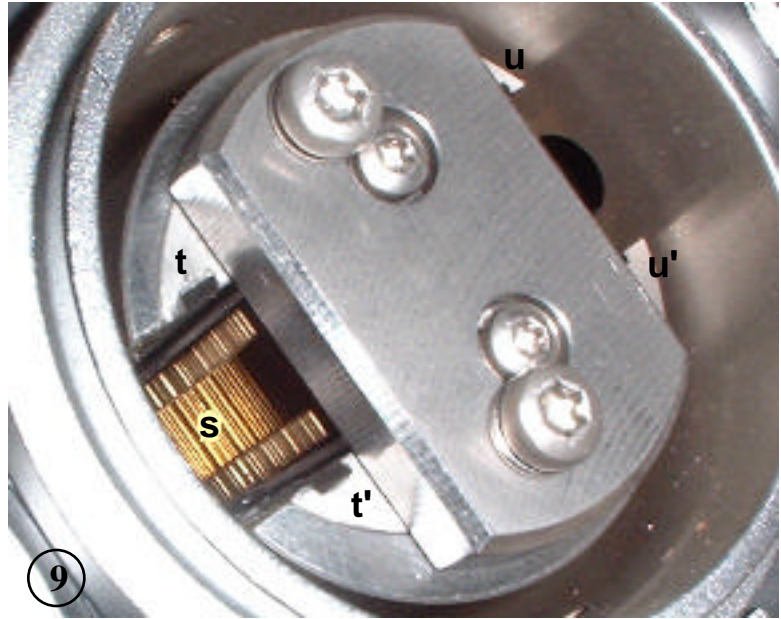
Replacing the receiver slit lid is the reverse of removing it. Be certain that the pin on the lower edge of the lid lies under the microswitch lever (*r*), such that the lever is depressed when the knurled set screw is tightened. This microswitch prevents the shutter from being open when the receiving slit lid is removed.

Do not over tighten the knurled set screw; this need only be snug enough to depress the microswitch lever.



Secondary soller slits (s) are sandwiched between the receiving slit slots ($t - t'$) and the scatter slit slots ($u - u'$).

Soller slits reduce axial divergence of the secondary X-ray beam. They consist of closely spaced (0.5 mm) vertical plates. With soller slits installed, the maximum axial divergence is 2.3° .



A word or two about slits

In general, slits (divergence, receiving, scatter, and soller) serve to refine the X-ray beam and to increase resolution, i.e. sharpness of peaks. However, this comes at a cost; slits get in the way of the beam and reduce its intensity. If low intensity is a problem and high resolution is not required, large slits are called for. In some cases, it may be advantageous to remove one or more slits. Different sizes of divergence, receiving, and scatter slits are available. These are easily replaced in the diffraction system. Soller slits should not be removed, except in extraordinary circumstances.

Divergence slit

A fixed divergence slit, in combination with the position of the goniometer, determines the length of the sample that is "illuminated" by X-rays. The length of the sample that is illuminated decreases with decreasing slit size and with increasing goniometer angles (θ and 2θ , 2θ on the display module).

The divergence slit is measured in degrees; the mounting plate is wider than the receiving or scatter slits.

Receiving slit

A good rule for receiving slits is to make the receiving slit width approximately equal to the line's full width at half maximum (FWHM). In most cases, a 0.2-mm receiving slit works well. For very narrow lines, 0.1 mm or 0.05 mm can be used.

The receiving slit is measured in millimeters; the mounting plate is the same size as the scatter slit and is narrower than the divergence slit.

Scatter slit

The scatter slit, in combination with the position of the goniometer, determines the length of the sample that is "seen" by the detector. The length of the sample that is seen by the detector decreases with decreasing slit size and with increasing θ values. The scatter slit size should be the same as that of the divergence slit.

The scatter slit is measured in degrees; the mounting plate is the same size as the receiving slit and is narrower than the divergence slit.