# Section IV: ALIGNMENT

## Unpacking

Carefully unpack the goniometer. Be sure to remove and inspect all items. Some pieces are small, packed separately, and are easy to overlook if you are not careful. Please inspect carefully any packing material before discarding.

If there are any signs of external damage to the crates or cartons, you must make note of them on the delivery receipt and/or freight bill. Notify the carrier immediately. Save all packing material for their inspection. Contact Brookhaven Instruments for further advice.

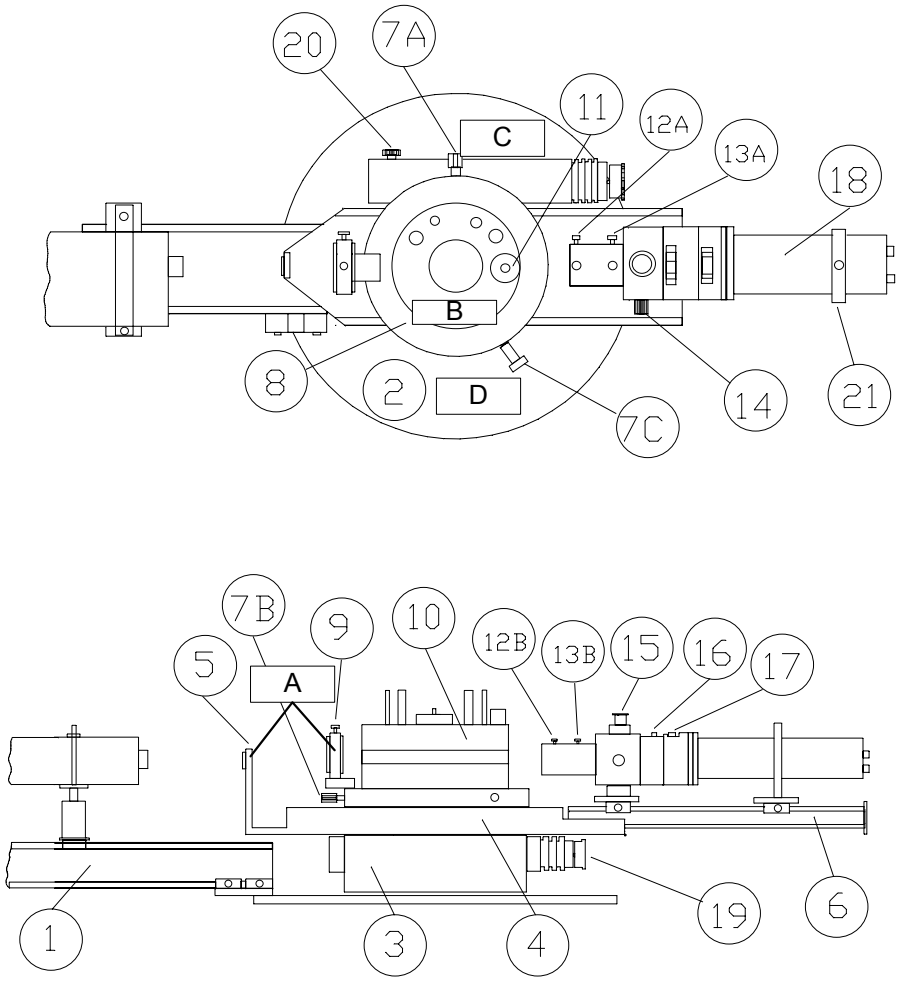
Do not return equipment to Brookhaven Instruments without requesting a return material authorization.

Most units are shipped in a wooden crate and 2 cartons. The crate contains the goniometer and sample cell assembly. The detector optics, PMT, sample cells and options such as the high voltage power supply and the filtration/circulation system are packed separately in the large white box with the BIC logo. The long carton contains the optical rail, part of the laser rail and mount option. The temperature controller, also an option, is packed in its own carton.

Do not leave any small pieces in the packing containers. Look for the o-ring that is part of the PMT housing, forceps and filters that are part of the filtration/circulation system, well-wrapped sample cells that look like packing material, any loose screws, etc. DO NOT THROW AWAY any packing material until you have finished the alignment and have identified all pieces.

Some of the parts of the goniometer are illustrated in Figure IV-1. These parts are listed in the legend that follows. Additional pieces not shown include the universal cell holder and the alignment cell. The following optional items are also not shown: the temperature controller, the filtration/circulation system, and the high voltage power supply.

Figure IV-1: Top & Side View of Assembled Goniometer



## Legend

1. Laser rail and mounts, optional
2. Precision-machined base
3. Turntable
4. Rigid rotating arm
5. Upright for 2 mm alignment aperture
6. Detector rail
7A,B. Center of rotation adjustment screws
7C. Center of rotation locking screw
8. Center of rotation adjustment table
9. Beam focusing and steering lens assembly
10. Sample cell assembly
11. Beam Stop
12A. Lens adjustment, horizontal
12B. Lens adjustment, vertical
13A. Slit adjustment, horizontal
13B. Slit adjustment, vertical
14. Mirror adjustment
15. Eyepiece
16. Pinhole wheel
17. Filter wheel
18. Photomultiplier housing
19. Angle adjustment
20. Clutch release
21. Support ring

## Laser Safety

BIC manufactures your goniometer in compliance with United States Laser Safety Regulations 21 CFR Subchapter J. This Federal regulation is administered by the Bureau of Radiological Health (BRH) under the auspices of the Food and Drug Administration.

BIC does not manufacture lasers, only the means for mounting the laser and adjusting the beam position. It is the user's responsibility for maintaining the laser as prescribed in the laser operator's manual.

Figure IV-1 depicts the positions of the warning and informational labels with capital letters. Figure IV-2 shows facsimiles of these labels in more detail.

Figure IV-3 shows the position of the rigid rotating arm for adjustments at or near zero scattering angle. After initial alignment, a black Delrin disk is mounted in the alignment upright (5). Previously, a neutral density filter was supplied. This item is no longer necessary. You should not look directly at the laser beam in the following alignment procedures.

This manual refers to laser power of 15 mW or less. CAUTION: HIGHER POWER LASERS MAY CAUSE BRH SAFETY LIMITS FOR VIEWING OPTICS TO BE EXCEEDED. The BRH limit is often too high for comfortable viewing. If you feel you need to reduce the intensity even further, please contact BIC for suggestions.

As a general rule: DO NOT LOOK DIRECTLY INTO THE LASER OR AT ITS DIRECT REFLECTION. Laser light is hazardous to the eyes. When the laser is in operation but the beam is not in use, block the radiation by using the beam attenuator. Most often this is a shutter located on the laser head. This is an excellent habit to form. It will also keep the output mirror of the laser clean, ensuring a longer time between cleaning.

Never turn on the laser while looking through the eyepiece (15). Likewise, never rotate the mirror into the down position (14) while looking through the eyepiece. Place a piece of white paper a few inches above the eyepiece. If you can see any light from the eyepiece falling on it, then reduce the beam intensity before looking into the eyepiece.

Figure IV-2: Laser Safety Labels

A

AVOID EXPOSURE
LASER RADIATION IS EMITTED
FROM THIS APERTURE

B

**DANGER**

LASER RADIATION WHEN OPEN.
AVOID DIRECT EXPOSURE TO BEAM

C

**BROOKHAVEN INSTRUMENTS CORPORATION**

750 BLUE POINT ROAD
HOLTSVILLE, NEW YORK 11742

**Manufactured:** April 1996
**Model:** BI-200SM
**Serial No.:** 400
**Power:** 15mW MAX Made in U.S.A.

THIS PRODUCT COMPLIES WITH
21 CFR 1040 AS APPLICABLE

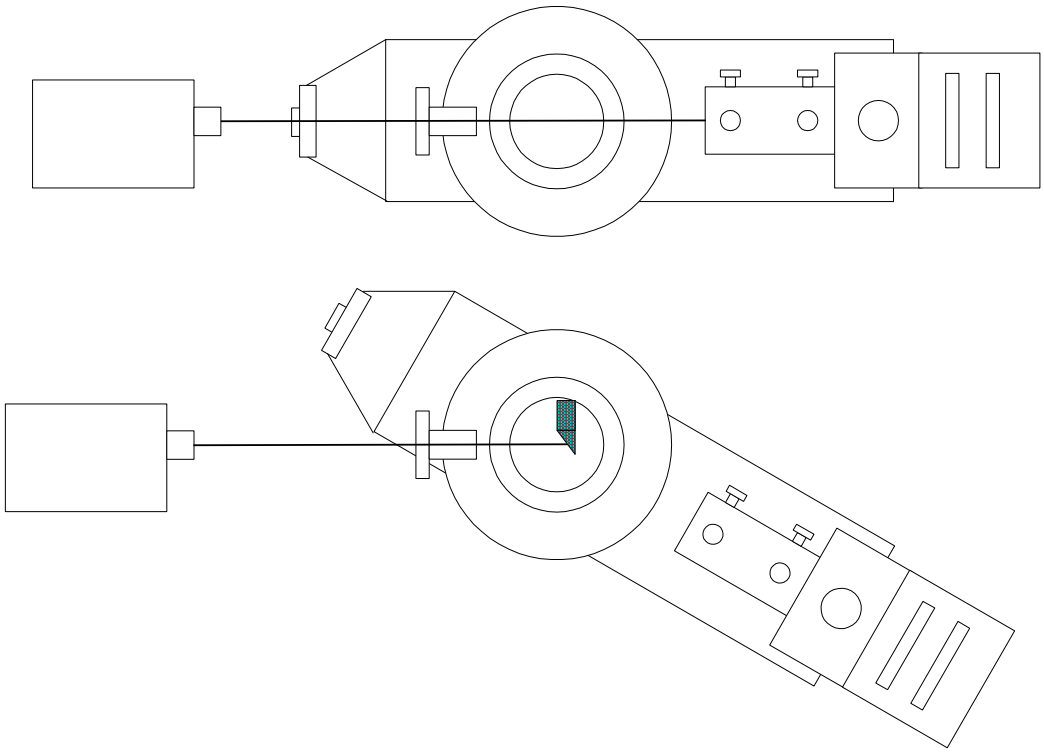
D

**DANGER**

LASER RADIATION
AVOID DIRECT EXPOSURE
TO BEAM

CLASS IIIb LASER PRODUCT

FIGURE IV-3



## Alignment Introduction

When the system is properly aligned, the laser beam will be focused horizontally onto the center of rotation of the sample cell. The cell will be concentric with the vat. Scattered light from the center of rotation will be focused onto the slit in front of the photomultiplier. To achieve this, it is necessary to mount various optical and mechanical pieces in sequence.

It is assumed that all the pieces have been purchased from Brookhaven Instruments. If the user has supplied components, appropriate modifications to these procedures are the user's responsibility.

A set of metric Allen wrenches, a flat-head screw driver, and a small pen light are the only tools needed for alignment.

Please read the previous section on laser safety. During the alignment procedure, the laser shutter is opened and shut in a precise sequence to avoid directly viewing the laser beam.

It is difficult to judge the size and position of very bright beams. If your laser power is adjustable, reduce it below 15 mW.

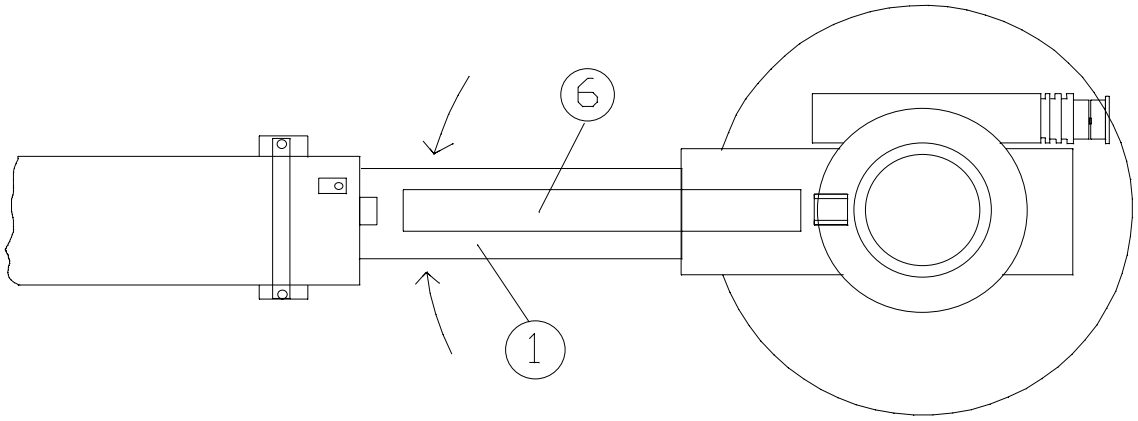
These are the steps you will follow to align your system:

1. Establish an approximate zero angle.
2. Align the laser.
3. Position and adjust the index matching vat.
4. Position the focusing lens and steer the beam.
5. Position and adjust the detector optics.
6. Establish the center of rotation.
7. Iteration of steps 3 through 6.
8. Position the beam stop.
9. Attach the PMT and make cable connections.

## Zero Angle

Your system was aligned at the factory. Most of the pieces are in the correct position or nearly so. The laser rail, if you ordered this option, is too long to be shipped installed. When first inserted into the clamping carriage it may not be properly aligned, so follow these procedures. The numbers in parenthesis correspond to those in Figure IV-1.

Figure IV-4: Establishing An Approximate Zero Angle




Place the machined base (2) on a sturdy table. Securely fasten the base to the table using screws or clamps. Slide the laser rail (1) into the clamping carriage on the base. A separate foot/carriage is provided with the laser rail option. Slide it onto the free end of the laser rail. Position the foot approximately one-third the distance from the free end. *Lightly* tighten the screws on the base clamping carriage. Securely tighten the screws on the separate clamping carriage, but do not secure the foot to the table.

Remove the black top and outer jacket from the sample cell assembly (10). Remove the three screws from the top of the brass manifold, and lift the manifold out of the assembly. Set it aside. Loosen the four set screws on the focusing lens (9) mount and slide the lens off. Remove the alignment upright (5) with the 2 mm aperture, located on the opposite side of the goniometer from the laser rail, and replace it with the detector rail (6). Fasten the rail to the rotating detector arm (4) using the two screws that are shipped installed in the rail.

Completely loosen the clutch release (20), allowing the rotating arm (4) to swing freely. Rotate the detector rail (6) until the angular scale reads 180°. The angular scale is located on the turntable (3). Tighten the clutch release. Sight along the detector rail toward the laser rail (1). They should be parallel with each other and with the focusing lens mount on the adjustment table (8). See Figure IV-4. If necessary, adjust the angle with the fine adjustment knob (19) until they are. You may need to gently tap the laser rail with a your hand. If necessary, loosen locking screw (7C) and rotate the adjustment table (8) until the center line of the lens mount is parallel to the detector and laser rails as shown in Figure IV-4. **Do not turn the adjustment screws (7A & 7B) to adjust table.** Secure the laser rail foot to the table, and gently tighten locking screw 7C.

Look at the coarse vernier scale on the turntable (3). If the angle no longer reads 180°, loosen the set screw (using a 2 mm Allen wrench) in the vernier. The set screw that allows adjustment of the vernier scale is located at approximately 175°. Then rotate the vernier until the permanent hash mark on the turntable lines up with the 180° marking. Loosen the set screw (using a 2 mm Allen wrench) on the angle adjustment (19), and rotate the knob until either one of the two zero markings lines up with the permanent hash mark on the motor housing. Tighten both set screws. Release the clutch, and rotate the detector rail until the angular scale reads zero. Completely tighten the clutch release. Use the angle adjustment (19) to obtain exactly zero degrees.

Place the optional laser mounts on the rail. If the laser used is not cylindrical, remove the crossbar by unscrewing the knurled, plastic-topped screws located on the mount posts. See Figure IV-5.

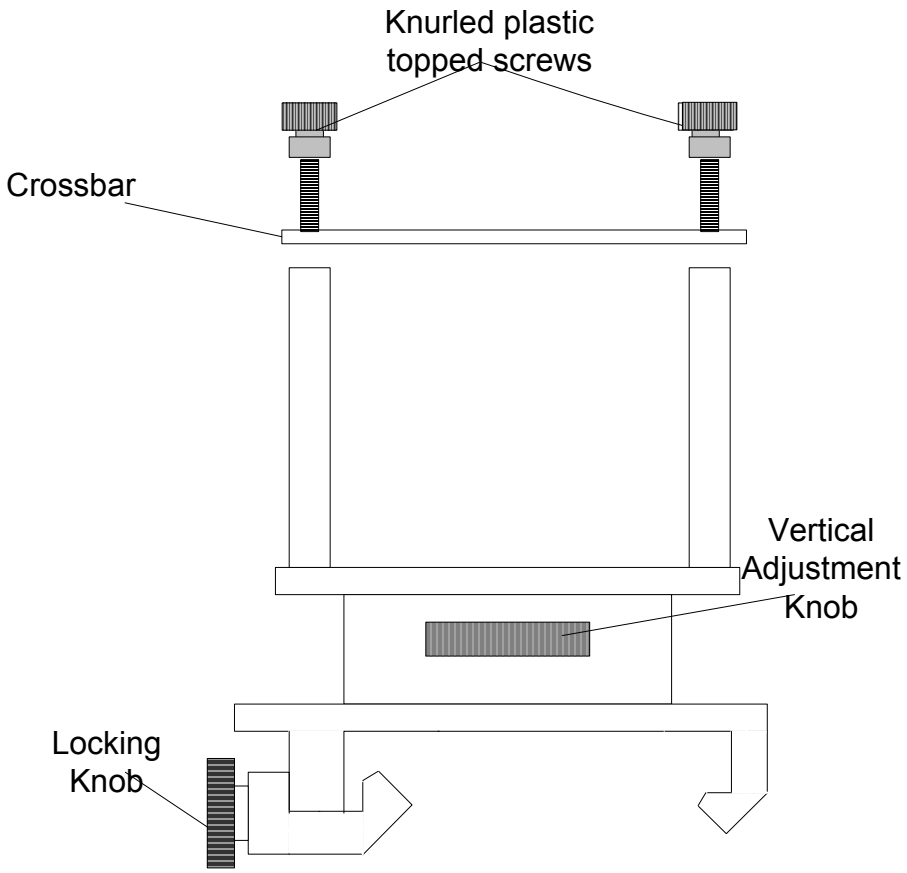
Position the mounts and laser as far as possible from the goniometer. Divergent stray light is then more easily blocked by the first aperture in the beam steering and focusing assembly.

Place the laser in the mounts. Cylindrical laser users should ensure that the linear polarization of the laser is vertical. Non-cylindrical lasers must possess linear polarization in the vertical direction. Contact Brookhaven Instruments if you are unsure of your laser's polarization.

Make the power and any cooling-water or duct connections to the laser. Turn it on. Warm it up for at least 15 minutes or until the laser has reached maximum beam pointing stability as described in the laser manual.

BLOCK THE BEAM WITH THE MECHANICAL SHUTTER LOCATED ON THE HEAD OF THE LASER. IF YOUR LASER IS NOT EQUIPPED WITH A SHUTTER, PLACE A ND3 FILTER IN THE PATH OF THE LASER, OR BLOCK WITH A NON-REFLECTIVE OBJECT.

Figure IV-5: Optional Laser Mount (BI-LRM)




Look underneath the center of rotation adjustment table (8). The table should be centered, with an equal gap as depicted in Figure IV-6A.

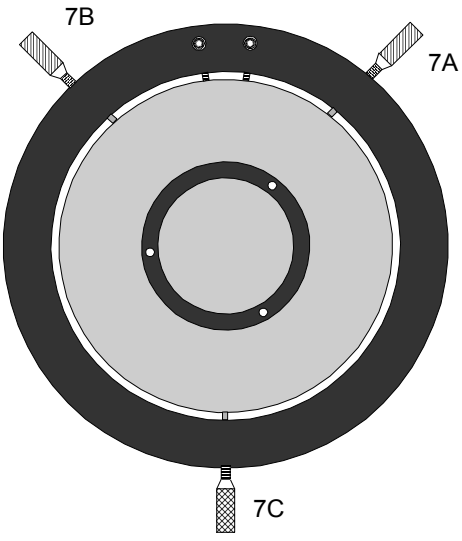



Figure IV-6A: View from Bottom of BI-200SM

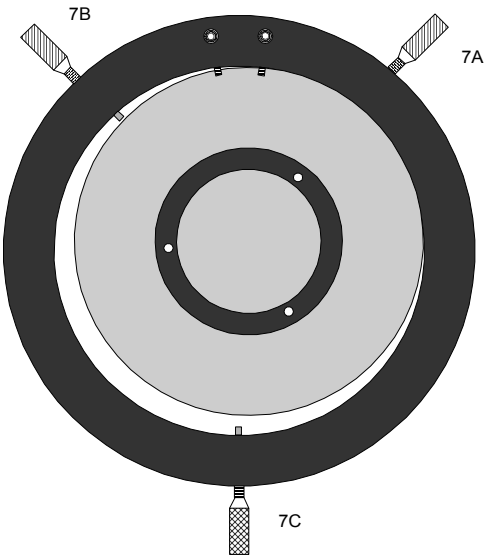



Figure IV-6B: Misaligned Goniometer Table

If it is not centered, as depicted in Figure IV-6B, loosen the locking screw (7C) and adjust 7A and 7B until the table has a uniform gap between it and the stainless steel plate.

Remove the detector rail. Attach the second upright (5) to the rigid rotating arm (4). See Figure IV-7. Insert the 2 mm apertures into the uprights. Unblock the laser beam.

With the laser warmed up, use the laser-mount adjustments to steer the beam cleanly through *both* apertures.

For most users who have not purchased cylindrical lasers, vertical adjustments can be made by using the large, knurled brass knob located in the center of the mount. To correct for horizontal beam placement errors, the laser must be physically moved within its mount.

For users with cylindrical lasers, use the bottom two positioning screws for height adjustment. Rotate the laser until the polarization mark ensures vertical, linear polarization. Secure the laser with the top screw.

When the laser is properly aligned, there are no bright spots visible on the apertures; instead, a slight corona effect is observed. Tighten the knurled brass knobs so the crossbar securely holds the laser in the mount.

**BLOCK THE LASER BEAM.**

Replace the second upright with the detector rail. Remove the 2 mm aperture from the upright (5).

Figure IV-7: Laser Alignment

Place the target on the last page of this section (page 4-31) on the wall, about 1 - 3 meters from the base of the goniometer. Center the target on the laser beam and tape the target to the wall, being careful to keep the spot centered.

## Vat Placement

Unpack and inspect the index matching vat very carefully. DO NOT HOLD THE VAT BY ITS CYLINDRICAL SURFACES. It should be clean with only dust clinging to its surfaces. Use dry air or compressed gas to remove the dust. Hold the vat securely while doing this.

Eventually the vat will need additional cleaning. Use a warm, mild soap solution. DO NOT USE ABRASIVES. Drip or blow-dry the vat. DO NOT DRY THE VAT WITH TOWELS. The beam enters the vat on the polished flat that is parallel to the axis of the cylinder. The part of the vat that must be free of water spots is a one centimeter-wide band running counterclockwise around from the flat toward zero degrees. The band starts about 1 cm from the base of the vat.

When dry, gently wipe this region by dragging a lens tissue across the surface of the vat onto which you have dripped reagent grade methanol. Use only the highest quality lens tissue on the vat. DO NOT USE ORDINARY LAB TOWELS OR TISSUES. Hold the tissue with forceps being extremely careful not to allow contact between the vat and forceps. DO NOT RUB THE VAT USING FINGER PRESSURE. A piece of dirt or dust trapped between the tissue and your fingers could scratch the surface. Brookhaven Instruments is not responsible for scratched glassware after delivery.

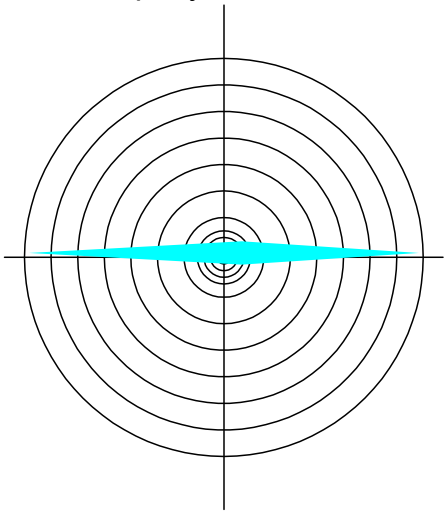
Clean any grit or dust from the inside of the black metal pot. Holding the the back of the vat with your fingertips (the “Brookhaven” label is at 90°, the back of the vat is opposite the “Brookhaven” label), carefully lower the vat into the metal pot, centering the flat entrance window perpendicular to where the laser beam will enter. Fill the vat with approximately 100ml of Decalin (cis+trans decahydronaphthalene), being very careful not to spill any Decalin on the outside surfaces of the vat. If you drip any Decalin, repeat the cleaning steps described above. Decalin

has a refractive index close to that of glass, and its vapor pressure is much less than that of toluene, the traditional index matching liquid. DO NOT USE WATER.

Unblock the laser beam. Adjust the vat within the pot until the exit beam centers on the target and the back reflection from the flat entrance window lies on the laser exit window. Adjust by rotation and translation or a combination.

Due to the cylindrical shape of the vat, the beam exiting the vat and striking the target will assume an elongated, horizontal elliptical shape. Center this shape on the target. See Figure IV-8. The back reflection from the flat entrance window is a spot. Center it on the laser exit window. This spot is superimposed on a weaker, elliptical reflection from the rear of the vat. It is the spot that you want to superimpose on the input beam, not the elliptical reflection. If the spot is not obvious to you, then temporarily block the beam inside the vat.

Figure IV-8: Vat Placement -Target Image of Laser when Vat is Properly Positioned



Lightly secure the vat using the three nylon screws around the sides of the pot while pressing firmly down on the top edges of the vat. Use a thin bladed, small handled screwdriver to tighten. Utilize only enough pressure such that the vat cannot be twisted by hand. Check that the image on the target and back reflection is correct.

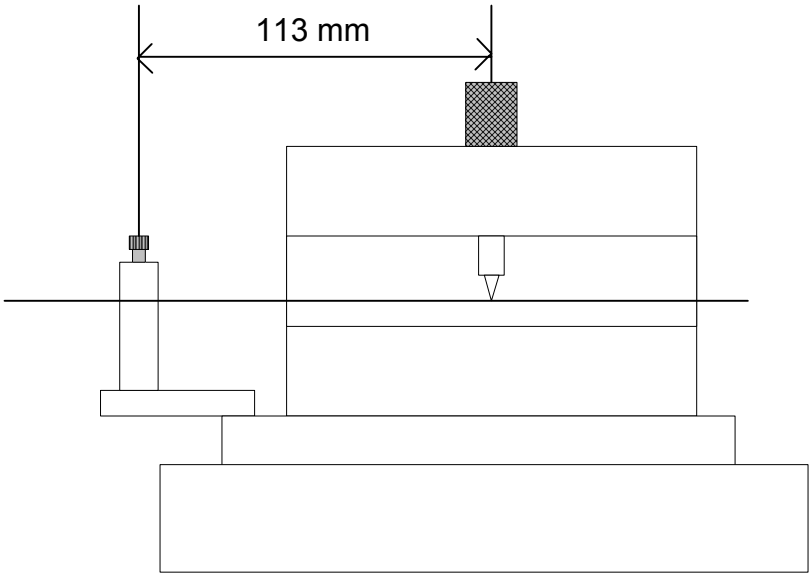
CAUTION: VATS ARE VERY DELICATE AND EXPENSIVE. USE ONLY LIGHT, EVEN PRESSURE ON THE NYLON SCREWS. A HEAVY HAND ON THE SCREWDRIVER WILL CRACK THE VAT.

Carefully replace the brass manifold, securing it finger-tight with the three screws. Refit the black outer jacket and the top to the sample cell assembly. Check that the image on the target and back reflection is correct.

BLOCK THE LASER BEAM.

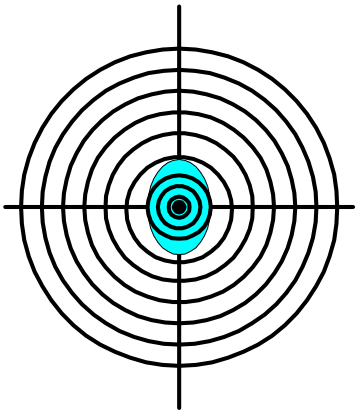
CAUTION: VATS ARE VERY DELICATE AND EXPENSIVE. THEY CAN BE CRACKED WHEN REPLACING THE MANIFOLD IF YOU ARE NOT CAREFUL.

Figure IV-9: Position of Steering Lens Assembly




Unblock the laser beam. Move the vertical lens adjustment, which is the brass screw on the top of the lens assembly, until the beam is vertically centered on the target. The image on the target is now a vertical oval. See Figure IV-10.

Figure IV-10: Image of Laser on Target

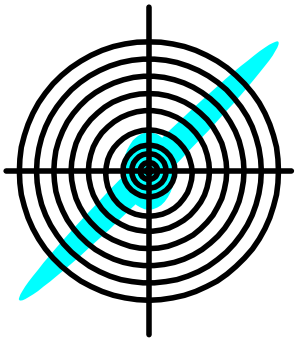


With the needle tip well below the beam, move the horizontal adjustment (located on the side of the lens assembly) until the beam strikes the needle. Notice the image on the target - it should show flare light as depicted in Figure IV-11. (Of course, the flare light could be off in the other direction, which would cause a mirror image of Figure IV-11.) Raise the needle by

turning it counterclockwise about one quarter turn. Turn the horizontal adjustment of the lens assembly until the laser beam strikes the needle again. Repeat this step until the beam is centered on the tip of the needle. (Many needle tips become hooked or bent, in which case it is sufficient to center the beam horizontally on the thicker needle shaft while maintaining the beam vertically centered on the target.) The flare light should now be centered horizontally as depicted in Figure IV-12.

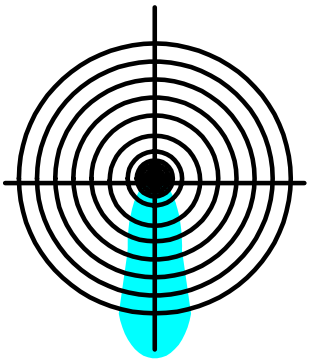
**BLOCK THE LASER BEAM.**

Figure IV-11: Image of Target for Lens Adjustments



Here the laser is striking the side of the needle. Notice the flare is on an angle. The needle is raised and the lens is adjusted horizontally until the laser strikes the tip of the needle, causing the flare to appear on the target as depicted in Figure IV-12. Some systems will cause a 4-pointed star pattern to appear instead of the more predominate downward flare seen below. When the laser strikes the tip of the needle and either pattern appears, the lens is properly focused.

Figure IV-12: Target Image with Properly Aligned Focusing Lens



**BLOCK THE LASER BEAM.**

## Detector Optics Adjustments

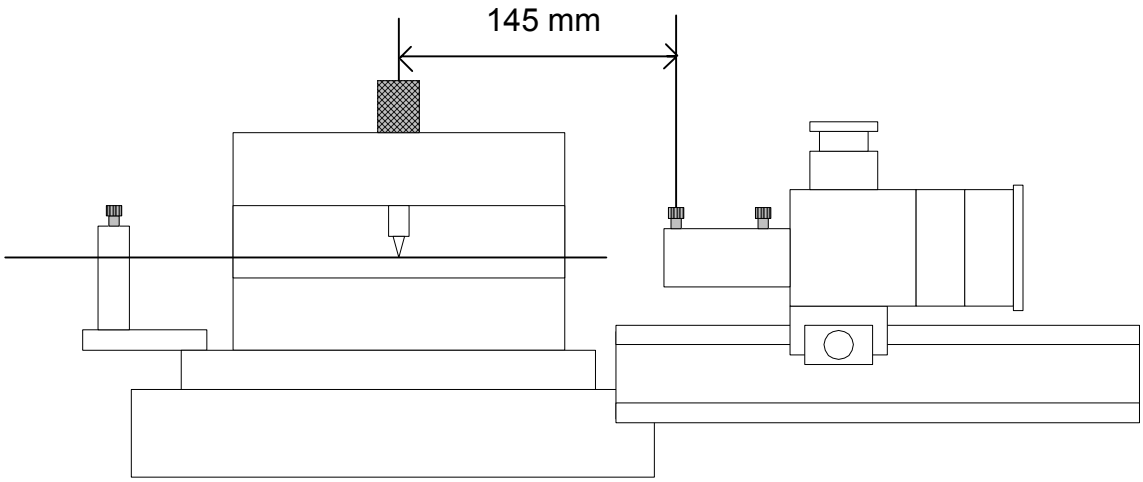
The front part of the detector optic consists of an adjustable lens that focuses light onto a slit. Behind the slit is a two-position mirror. When the mirror adjustment (14) is rotated counterclockwise, light focused onto the slit can be seen in the eyepiece (15). When the mirror adjustment is rotated clockwise, the light passes through the slit onto a variable pinhole and then through a filter into the photomultiplier tube.

Change the angle of the rotating arm to 20°. Mount the detector optic on the detector rail. Rotate the mirror adjustment (14) counterclockwise. Shine a penlight into the small entrance hole of the detector optic. Move the eyepiece up and down until the slit is sharply in focus. Lock down the eyepiece. Rotate the mirror adjustment clockwise.

Slide the detector optic along the rail until it is 145 mm from the center of rotation. Measure from the center of the vertical adjustment knob 12B. See Figure IV-13. Lock the detector optic slider so it cannot move along the detector rail.

Rotate the pinhole wheel (16) to the 3 mm position and the filter wheel (17) to the position marked "O" for open. Change the angle of the rotating arm to zero degrees. Unblock the laser beam.

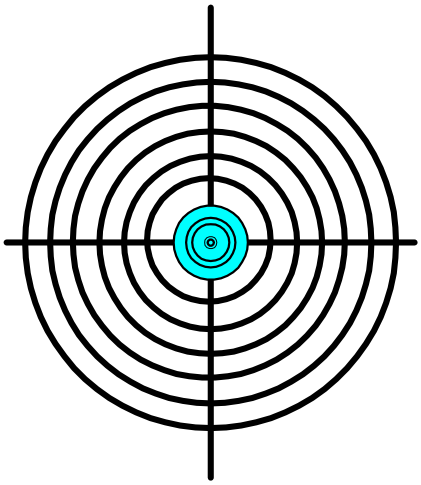
Figure IV-13: Position of Detector Optics.




Use the horizontal and vertical adjustments on the lens (12A & B) to maximize the intensity of the beam falling on the target. You will also need to move the slit in the horizontal direction (13A) and adjust the angle (19). Rotate the pinhole wheel to the 1mm position. Adjust the lens, slit, and angle until the intensity of the image on the target is maximized. The

image should be a bright circular spot with visible diffraction rings. If possible, reduce the intensity of the laser and turn off the room lights to better see the diffraction rings.

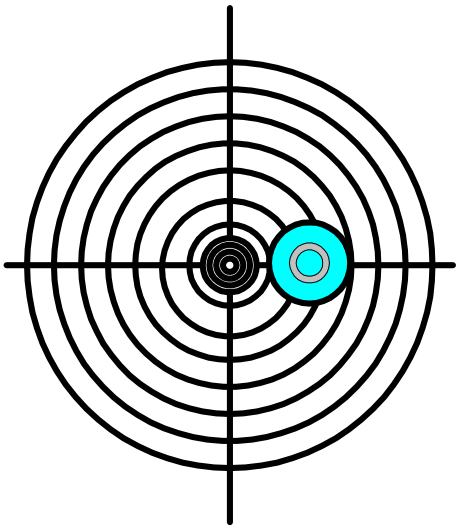
Figure IV-14: Target Image of Laser Through Detector Optics



Most likely you will not be able to superimpose the light onto the center of the target like it is in Figure IV-14. At this point, it is more important to get the image centered vertically. Use adjustment 12B to accomplish this.

Rotate the pinhole wheel. Notice the position and uniformity of the image at each position. If the image on the target is not centered, as depicted in Figure IV-15, use the lens (12A & B) and the horizontal slit (13A) adjustments to "walk" the beam toward the center of the target.

Figure IV-15: Target Image of Laser Through Misaligned Detector



To "walk" the beam, do the following:

Move the slit horizontally (13A), turning clockwise if the image is to the right of being centered on the target, counterclockwise if to the left, until the image begins to disappear. Then adjust 12A to maximize the laser intensity image. Change the angle (19) slightly until the maximum intensity is observed. Repeat this process until the uniformly bright image is centered on the target.

Check the images using the other pinhole positions. Find the lens and slit position where all the images are uniformly bright, maximized, and without halos.

**BLOCK THE LASER BEAM.**

Reset the zero angle using the vernier scale on the turntable and the fine adjustment knob (19). This is your new zero angle.

## Center of Rotation

Rotate the mirror adjustment (14) counterclockwise. Aim a penlight into the initial aperture of the detector optics. Observe the light as it passes through the slit. Adjust the eyepiece until the slit appears focused.

Insert the alignment cell into the sample cell assembly. Release the clutch (20) and change the angle to 20°. Aim a penlight at the needle. Hold the penlight at the smallest angle possible between the light and the front of the detector optic. The needle of the alignment cell should be visible, though probably not centered, in the eyepiece. Rotate the needle adjustment up and down until you can easily see the tip. The reflection off the side of the needle will be a bright band of light, on an angle in relation to the slit. The tip of the needle will appear to point toward the center of the goniometer. If necessary, adjust the slit horizontally (13A) until the needle is exactly centered in the slit.

Unlock the center of rotation adjustment table (7C).

**A.** Change the angle to 155° while looking through the eyepiece. Unless the sample cell assembly is perfectly centered on the turntable, the needle will appear to move across the slit.

**B.** Use adjustment screw 7A to halve the distance the needle tip is displaced from the center of the slit. This should be a minor correction. Do not rotate the screw more than one half turn in either direction. If greater adjustment appears to be needed to center the needle image, refer to Figure IV-6A and Figure IV-6B. If the rotation table appears centered, contact Brookhaven Instruments for assistance.

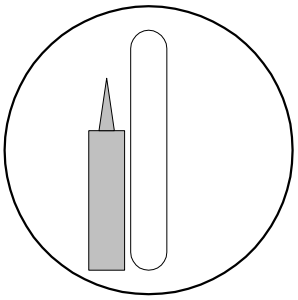
**C.** Change the angle to 20° while looking through the eyepiece. The needle should appear to move much less. Center the slit on the needle using the horizontal adjustment (13A). Repeat steps A, B and C until the needle is centered within the slit.

**D.** Change the angle to 90°. Use adjustment 7B to center the needle in the slit.

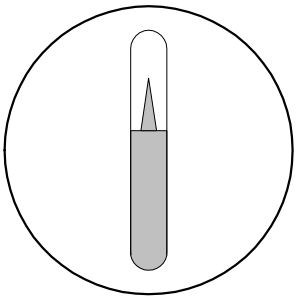
Repeat steps A, B, C, and D until the needle does not appear to move in the slit. When you are satisfied, use adjustment 7C to lock the center of rotation table. Return the detector optic to 0° and engage the clutch (20). Rotate the mirror adjustment (14) clockwise. Unblock the laser. *If the image is still uniformly bright, and the intensity is maximized and centered on the target,* skip to the section entitled "Beam Stop." If not, continue with the next section entitled "Iterations."

Figure IV-16 Center of Rotation Adjustments

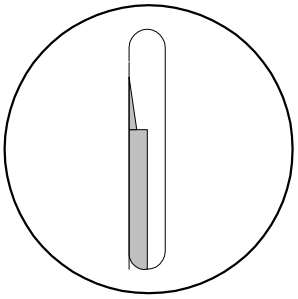
At 20°, the needle is out of the slit



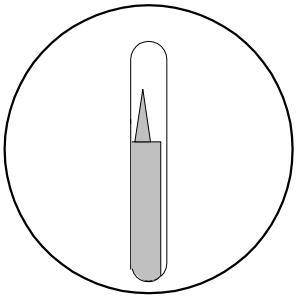
Move the slit (13A), until the needle is centered



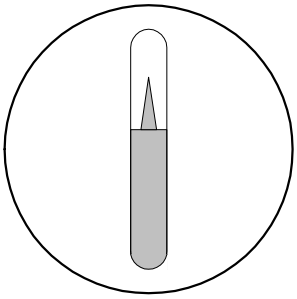
After rotation to 155°, the needle may look like this



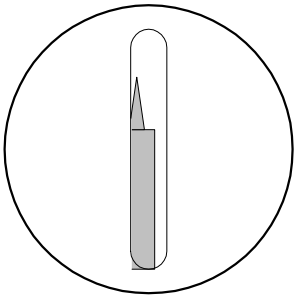
Halve the difference (7A).



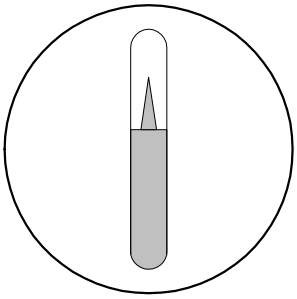
Move the slit (13A) until the needle is centered



At 90°, if the needle is not centered, then...



Use adjustment screw 7B to center the needle



## Iterations

If the center of rotation adjustment table has been moved significantly, then the vat has changed its position, and the beam may no longer hit the needle tip or the center of the target. In this case execute the following steps.

Release the clutch and change the angle to 20°. Center the lens within its mount by using the vertical and horizontal adjustment screws. Rotate the needle adjustment clockwise until the needle intercepts the path of the laser. *Slightly* raise the needle, about one quarter turn counterclockwise. Loosen the locking screw (7C). Gently grasp both 7B & 7C. DO NOT TURN THESE SCREWS. Using the screws *for gripping purposes only,* slowly rotate the table until the needle again intercepts the path of the beam. Continue this procedure until the flare from the needle on the target appears as described in the section entitled "Detector Optic Adjustments" and depicted in Figure IV-12. Tighten the locking screw (7C).

Remove the focusing lens assembly (9). Check the back reflection and the elliptical image on the target. If they are centered, replace the lens assembly to its original position. If they are not, block the laser beam, then carefully remove the brass manifold. Unblock the laser beam, and rotate and translate the vat until the reflection and image are correct. Then replace the lens assembly. Use the horizontal adjustment on the lens assembly until the beam strikes the center of the needle as described earlier. This should be a minor correction, if necessary at all.

Change angle to zero degrees and engage the clutch. Adjust, if necessary, the detector lens and slit and angle until the brightest, most uniform beam is obtained on the target.

Check the center of rotation. Make adjustments if necessary.

Repeat these steps until no further changes can be made.

Remove the alignment cell. Replace the shuffle plate on the cell-side of the steering lens. The larger opening should be on the lens side. Position the plate until the beam passes uniformly through it as viewed from the laser side of the lens. Check that the image on the target is not changed. Lock down the plate. Replace the other shuffle plate; position it in the same way; lock it down.

## Beam Stop

Mount the periscopic beam stop (11) on the top of the sample cell assembly. Twist it until the back reflection, as seen on the shuffle plate of the steering lens assembly, is centered on the optical axis. Secure the beam stop mount by tightening the two screws on the top of the mount.

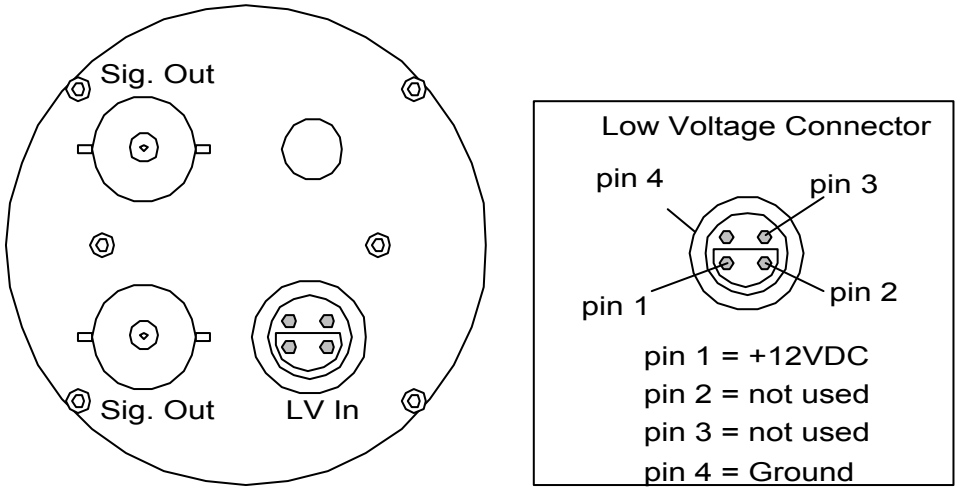
**Mounting The PMT**

Make sure that the o-ring in the PMT housing (18) flange is seated correctly. Hold the housing up against the end of the detector optic. Use the three, M5 screws in the rear of the detector optic to secure the PMT housing. The screws have brass washers. First, use your fingers to tighten the screws evenly all around. Then use an Allen wrench to tighten the screws. Do not over tighten.

Slide the PMT support ring (21) onto the detector rail. This piece is necessary in systems where the PMT housing is longer than our standard. It is not supplied unless the BI-DS2 option is purchased. Brookhaven's standard PMT and housing do not require additional support. Position the ring about 1/4 the distance from the end of the PMT housing. Lock the slider on the rail. Support the PMT in the ring by lightly tightening the three support screws with your fingers. These screws are only for support, not for positioning. DO NOT TIGHTEN with a wrench.

**Power & Signal Connections**

Figure IV-17: Standard Configuration for BIC PMT

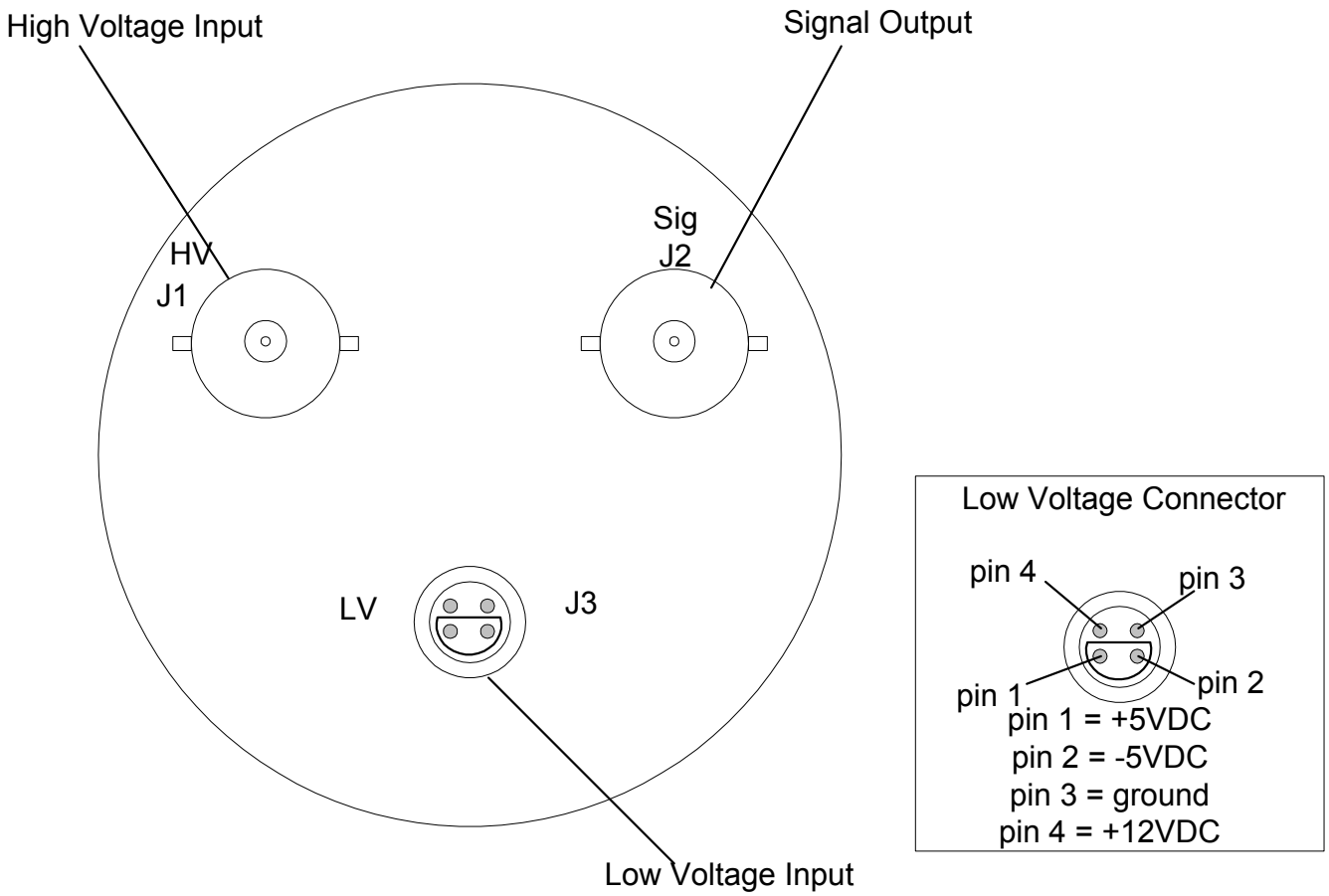


The standard Brookhaven PMT has the High Voltage power supply internally, so only signal and low voltage cables are required.

For signal hook up, attach the long, thin coaxial cable (RG 174) with one BNC and one miniature Lemo to either connector marked "Sig. out" on the PMT and the connector mark "A" on the BI-9000AT correlator board. The signal output employs TTL levels with a 50 Ohm output impedance.

For low voltage, attach the long, gray coaxial cable supplied with LEMO connectors at both ends to either low voltage output of the correlator to the low voltage input on the tube. This supplies the power to the amplifier/discriminator *and internal high voltage. Be sure to make this connection after the PMT housing is attached to the detector optics.* **Never supply low voltage to the PMT when it is not attached to the detector optics.** Doing so can easily destroy the PMT.

Figure IV-18: Power and Signal Connections for Optional BI-DS2



Signal

There are three connectors on the rear of the BI-DS2 PMT housing marked J1 H.V., J2 SIG, and J3 L.V. For signal hook up, use the short, thin coaxial cable (RG 174) with a BNC connector on each end to connect the PMT and the BI-HV connector marked "Sig. A in." Then attach the long, thin coaxial cable with one BNC and one miniature Lemo to the connector marked "Sig. A out" on the BI-HV and the connector marked "A" on the BI-9000AT correlator board. The signal output employs TTL levels with a 50 Ohm output impedance.

Use a coaxial cable (RG 58U) with BNC connectors at either end to connect the signal output (J2 SIG) to Input A on your BI-2030AT correlator.

High Voltage

For the high voltage connections, use the short, thicker, coaxial cable (RG 59) supplied with MHV (mini high voltage) connectors at both ends to connect the high voltage input (J1 H.V.) to the negative output on your HV supply. **Never connect or disconnect cables with the high voltage on.** Your BI-HV power supply has been preset at the factory to operate your PMT at peak performance. Contact the factory if you need to change the HV setting. **Do not turn on the high voltage power supply at this time.**

**Do not try to force a BNC connector onto the MHV connector**. The connectors should lock together easily by pushing and twisting. If excessive force is required to lock them together, then you have probably tried to mate the wrong pair. The female BNC and MHV connectors on the rear of the PMT housing look similar. The males, on the cables, however, are different. The male MHV pin is further recessed, and it is surrounded by thicker insulation. **If high voltage is applied, mistakenly, to the signal output on the PMT housing, damage to the internal amplifier/discriminator circuit will occur.**

Low Voltage

For the optional EMI tubes, use the short, gray coaxial cable supplied with LEMO connectors at either end to connect the low voltage input (J3 L.V.) to the low voltage output on the BI-HV, marked "LV out." Connect the long, gray coaxial cable supplied with LEMO connectors at both ends to either low voltage output of the correlator to the low voltage input on the BI-HV, marked "LV in."

## Motor Connections

If you ordered the BI-CON stepping motor controller option with a Brookhaven correlator, then use the thick, round, gray cable to make the connection from the 15-pin delta connector on the motor housing of the goniometer to the port marked MOTOR on the rear of the BI-2030AT correlator or on the rear panel of the computer for the BI-8000AT or BI-9000AT.

Experience has shown that many problems can be solved by making sure that all connectors are securely fastened before making measurements.

## Temperature Control

For some light scattering measurements it is important to maintain a constant and known temperature. This is particularly true for diffusion coefficient measurements. The diffusion coefficient is inversely proportional to the viscosity of the solvent or suspending liquid. Viscosity is strongly temperature dependent, and the percentage change per degree can be large.

For those cases where temperature control is necessary the plumbing has been provided in the goniometer. Many standard temperature baths with external circulation capability are suitable. The ones provided by BIC are typically capable of regulating temperatures over a larger range than the sample cell assembly can maintain (approx. +5° to +80 °C). It is up to the user to recognize this fact and maintain the temperature within these limits.

At the low end of the range, condensation may become a problem. Consider insulating all hoses, inlet and outlet ports, and any exposed metal surfaces on the sample cell assembly. It may be necessary to blow a dry gas onto the exposed portions of the index matching vat to prevent condensation there. At the higher end of the temperature range a heat loss may require additional insulation.

Above approximately 80°C temperature stability decreases. Gradients in the index matching vat and sample cell contribute to systematic and random errors.

The system has been designed to maintain ± 0.2 °C stability over the range +5° to +80 °C, and over a length of about 3 cm from the bottom of the cell. Thus, the temperature control is suitable for use with most light scattering experiments within the range specified, except critical point studies. In this case the user is advised to provide his or her own

temperature control. For the most exacting work the user is advised to measure the temperature of the sample directly.

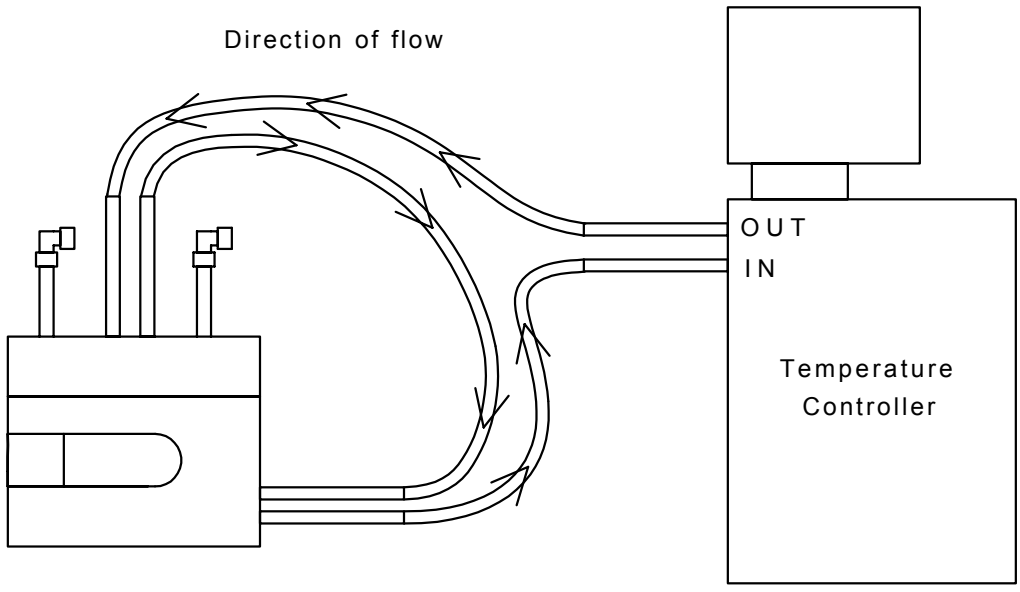
## Setting Up the Controller

**Be sure to read carefully the instructions supplied with the temperature controller before turning on the power**. The reservoir in the controller needs to be covered with the bath liquid to within 2 or 3 cm from the top, and care should be taken when externally circulating liquid since the bath level will drop. Never expose the heater coils to air while operating because severe overheating and damage may occur. **Do not use pure water as the temperature control fluid in the bath**. Instead of water, use ethylene glycol (antifreeze). It is less corrosive and does not evaporate as easily. A 50% v/v aqueous mixture is also suitable.

Use hose clamps on all I/O ports. The inside diameter of the tubing should be nominally 3/8 inch (9.5 mm) as this matches the outside diameter of the 4 ports of interest.

Liquid circulates through a copper coil surrounding the sample cell holder sleeve. Then it flows through a hollow chamber under the sample cell assembly. This combination should prevent severe temperature gradients over the range specified. Severe gradients in the cell can cause convection. This may result in sinusoidal features in the correlation function, which complicate, unnecessarily, the data analysis.

Figure IV-19: Connecting the Temperature Controller




Attach the circulator output to either large-diameter port on the top of the sample cell assembly. See Figure IV-19. Connect a short hose from the

other port to one on the sample cell assembly's bottom edge. Finally, connect the remaining port to the return port on the circulator. Tygon tubing is sufficient for use with ethylene glycol. Test the compatibility of the tubing with other liquids before using.

Arrange the hoses such that they do not interfere with any of the mechanical parts of the goniometer, especially when changing angles. It is convenient to use a ring stand for this purpose.

## Equilibration Time

When changing temperature, you should allow enough time for the sample to come to thermal equilibrium. The actual time depends on the circulator bath size, flow rate, tubing length, and the temperature change. There is also a lag in time between thermal equilibrium of the circulator bath and the sample. With circulators supplied by BIC it takes about 15 minutes for the sample to reach 40 °C starting from room temperature. An additional 15 minutes is required from 40 °C to 70 °C, and the sample temperature will be about 0.5 °C less than the circulator bath temperature unless the hoses, brass I/0 ports, and black, metal dust cap are insulated.

When cooling it takes longer to reach equilibrium. From room temperature to 5 °C it takes about 1 hour for the temperature of the circulator bath to stabilize. The sample temperature is about 1.5 °C higher unless extra, external insulation, is provided.

## Placement of the Controller

Small vibrations are produced by the circulation and refrigerator pumps. These vibrations may cause problems with more delicate measurements. Consider isolating the circulator from the table on which the goniometer is mounted. Placement on the floor is one option, though tubing length may be excessive.

## Circulation/Filtration System

To reduce stray light, index matching liquid surrounds the sample cell. This translates the air-glass interface to the outer surface of the vat which has a much larger diameter than the cell. Thus, a much wider range of scattering angles is usable without interference from stray light. In addition, stray light from scratches on the outer cell walls is minimized.

The ideal index matching liquid has a refractive index around 1.5, a low vapor pressure, and is easy to pump around a filtration system to remove dust. We recommend using cis-trans decahydronaphthalene, trade name Decalin, as the index liquid. Toluene, a known carcinogen, is no longer recommended for index matching. **Do not use water.**

The optional BI-FC filtration/circulation system consists of a pump, tubing, filter holder, clamp, tweezers, filters and connectors. The Teflon, graphite gear pump turns at 1550 rpm, delivers about 0.130 liters per minute of a 1 centipoise liquid and has a normal operating pressure of 15 to 20 psi. Operate the pump only when it is necessary to remove dust from the index liquid. Initially, for a dusty liquid in a dusty vat, this may take several minutes. Thereafter, the pump need only be used for a minute or two. A filter change will also be required after the first few minutes.

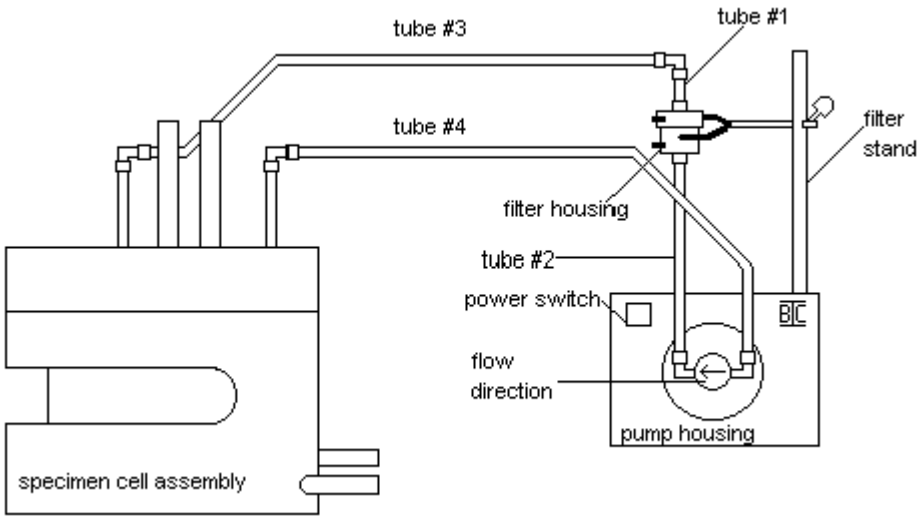
## BI-FC Connections

See Figure IV-20. Attach tube #3 to one of the Teflon elbow connectors. Insert the tube until it bottoms in the connector housing, and then tighten the nut by hand. Attach the other end of the connector to one of the small diameter ports located on the specimen cell assembly. Use the same procedure to attach tube #4 to the other port.

The NPT elbow connectors should already be attached to the pump head. If they are not, remove the pump head to attach them. Otherwise, they can be cross threaded and damaged. Attach tube #4 to the inlet side of the pump head. (Look at the arrow on the pump head.) Attach tube #2 to the outlet side. Attach the filter housing, with the filter installed, to the other end of tube #2. Clamp the filter housing to the filter stand. Attach tube #1 to the output side of the filter using one of the Teflon elbow connectors. Fasten this connector to the free end of tube #3. Check all connections.

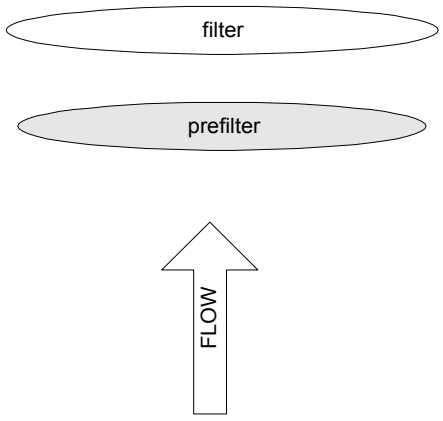
The filter housing, a 47 mm in-line type that is self-sealing (no o-ring), is also made of Teflon. The flow direction is marked on the side of the clear section. One hundred nylon filters are provided with the BI-FC. The pore size is 0.22 micron. This filter is compatible with most liquids. Always handle the membrane with the smooth-tip forceps that are included with the BI-FC system. Also supplied are 100 prefilters.

Figure IV-20: Filtration/Circulation System




Open the filter housing using the two, green plastic wrenches supplied, one on each end, fitted over the housing. Wet a filter with the index matching liquid and place the filter in the housing. Wet the prefilter, and place it with its smooth side against the filter. Center it as best you can; this will help prevent leakage. To seal the filters inside, tighten the housing with the wrenches. Later, if leaks develop around the housing, tighten it further. See Figure IV-20.

Figure 21: Direction of flow for the BI-FC




If you need more tubing, you may order it directly from the Cole Parmer Instrument Company, Chicago, Illinois, telephone 1-800-323-4340 (from the U.S.). Ask for the ¼ inch O.D. Teflon PFA tube, part number E06375-02.

## Running the BI-FC

Before starting the pump you must add more liquid. Fill the vat until the level of the liquid reaches the middle of the cell holder sleeve. Turn the pump on.

While it is running, check the level in the sample cell area. If necessary, add liquid until all the tubes and the filter housing is full. When the system is full, the level of the index liquid should be a few millimeters above the bottom of the cell holder sleeve. Run the pump until, at low angles, little or no dust is visible. Check for leaks. Tighten connections until the leaks stop.

With the pump stopped, the filter holder may be opened for filter replacement. If the amount of dust is not noticeably reduced after several minutes, change the filter. To change the filter, use the plastic wrenches to open the filter. Place a clean beaker under the filter to catch the liquid that spills. This liquid can be reused.

Do not pump the liquid during measurements of the scattered light. Pump vibrations may interfere with measurements.

Consider purchasing the optional BI-SFS sample filtration system for cleaning pure liquids and sometimes the samples themselves.

Figure IV-22: Examples of filter discoloration

TARGET