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AN ALIGNMENT PROCEDURE FOR THE KRATKY SMALL-ANGLE X-RAY CAMERA

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METALS AND CERAMICS DIVISION

AN ALIGNMENT PROCEDURE FOR THE KRATKY SMALL-ANGLE X-RAY CAMERA

J. W. Anderegg

Molecular Anatomy Program, Oak Ridge National Laboratory
(Also supported by the National Institute of General Medical Sciences, the National Cancer Institute, and the National Institute of Allergy and Infectious Diseases)

and

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Metals and Ceramics Division

FEBRUARY 1970

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AN ALIGNMENT PROCEDURE FOR THE KRATKY SMALL-ANGLE X-RAY CAMERA

J. W. Anderegg,[1] P. G. Mardon,[2] and R. W. Hendricks

ABSTRACT

A precision alignment procedure has been developed for the Kratky small-angle x-ray camera. This method uses only a proportional counter to measure the x-ray intensity distribution in the incident beam, as opposed to other methods which require fluorescent screens and x-ray film. During the progress of this work, two useful auxiliary alignment slits were also developed. Their use is described in detail. The roles of previously developed vacuum bellows seals and a diffracted beam graphite monochromator in the alignment procedure are discussed.

INTRODUCTION

The small-angle x-ray camera based on the collimation geometry proposed by Kratky[3] has become increasingly important in the fields of metallurgy, physics, chemistry, and biophysics. Effective use of this instrument requires that all slit edges in the collimation system be parallel to each other and parallel to the line view of the x-ray tube focal spot. In addition, the collimation system must be accurately pointed at the focal spot. This report presents a step-by-step procedure for precisely achieving these conditions.

Two views of the assembled camera are shown in Fig. 1. Those parts which play a role in the alignment procedure have been labeled. The identity of each labeled part is given in Table 1. It is to be noted

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[1] On leave from the Biophysics Laboratory, University of Wisconsin, Madison, Wisconsin.

[2] On leave from AERE, Harwell, England.

[3] O. Kratky and Z. Skala, Z. Elektrochem. 62, 73 (1958).

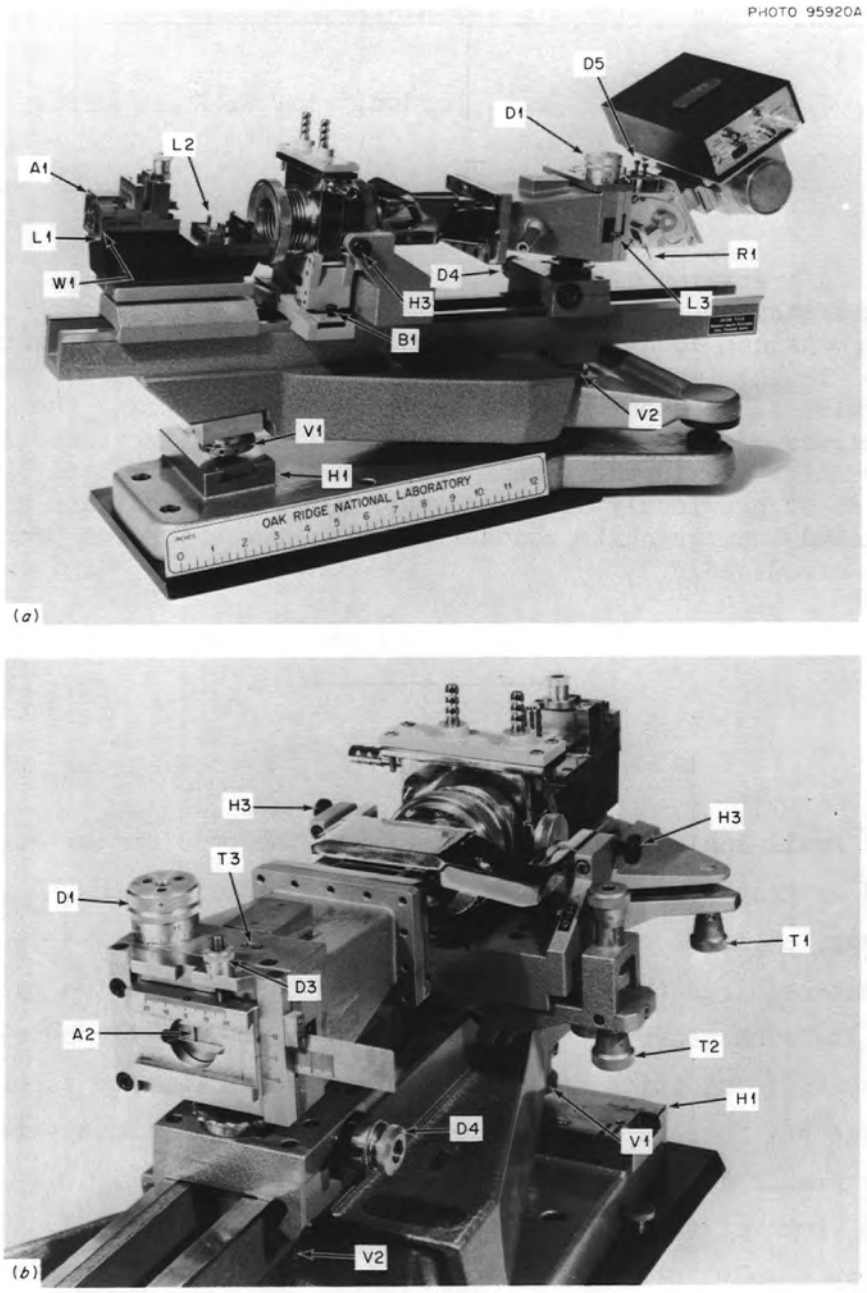

Fig. 1. Two Views of a Modified Kratky Small-Angle X-Ray Scattering Camera. (a) Side view and (b) rear view. Nonstandard components of this unit consist of (i) bellows seals between collimator vacuum cover, specimen chamber and vacuum path to detector, (ii) diffracted beam graphite monochromator box and preamplifier mounted on detector, and (iii) special alignment slits A1 (front) and A2 (rear).

Table 1. Definition of Kratky Camera Variables and Symbols

Symbol	Function
Alignment slits	
A1	Adjustable position front alignment slit (1 mm wide)
A2	Adjustable position rear alignment slit (1 mm wide)
A3	Special Kratky alignment device
Locking bolts	
B1	Vacuum sample chamber locking bolt
B2	Collimation system locking bolt (required only with bellows)
Displacement micrometers	
D1	Receiving slit (upper edge only) displacement micrometer
D2	Specimen chamber displacement micrometer
D3	Detector/monochromator displacement micrometer
D4	Receiving slit (lower edge) displacement micrometer (determines the scattering angle 2θ)
D5	Monochromator radiation shield displacement screws
Horizontal translations	
H1	Front horizontal camera translation micrometer
H2	Rear horizontal camera translation micrometer
H3	Vacuum tank horizontal translation screws
Collimation slits	
L1	Beam length collimation slit
L2	Beam length collimation slit
L3	Receiving slit length
Rotation screws	
R1	Graphite monochromating crystal 2θ adjustment
Tilt micrometers	
T1	Collimation system tilt micrometer
T2	Vacuum tank tilt micrometer
T3	Receiving slit (upper edge only) tilting screw
T4	Specimen chamber tilt micrometer
Vertical translation	
V1	Front camera height adjustment pivot
V2	Rear camera height adjustment pivot
Collimation slits	
W1	Entrance slit width
W2	Nonexistent – determined by W1
W3	Receiving slit width

that both cameras in use in this laboratory have been modified in several important respects, as discussed below.

A diffracted beam monochromator has been mounted between the receiving slit (slit 3) and the proportional counter. A small (30 × 5 × 1 mm) flat piece of hot-pressed pyrolytic graphite is used as the monochromating crystal.[4,5] Also, a low-noise preamplifier (ORTEC model 109PC) has been mounted directly on top of the detector in order to minimize the length of the detector-preamplifier cable and hence reduce the electronic noise of the x-ray detection system.[6,7]

Through the use of flexible metal bellows, the collimation system, specimen chamber, and vacuum tank have been connected together so that the entire x-ray path from the collimation system through the sample to the detector slit is in vacuum. This eliminates the windows (usually mylar or cellulose acetate) on the rear of the collimation system, on both sides of the specimen chamber and on the front of the vacuum tank. The resulting decrease in background scattering varies from a factor of about 15 at the larger scattering angles to over 50 at scattering angles of a few milliradians. The details of construction for each of these modifications will be described in a forthcoming publication.[8] In the interest of completeness, the alignment procedure described here will include the use of these modifications. For cameras not so equipped, the required changes in the procedure should be self-evident.

To aid in the alignment procedure, two special slits have been constructed. Slit A1 [Fig. 2(a)] is a 1.0-mm-wide vertical entrance slit, which may be translated horizontally in a dovetail mounted immediately in front of the length collimation slit L1 [Fig. 2(b)]. Slit A2 is a 1.0-mm-wide vertical receiving slit which may be translated horizontally in a dovetail cut in the front surface of the standard detector

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[4]C. J. Sparks, Metals and Ceramics Div. Ann. Progr. Rept. June 30, 1966, ORNL-3970, p. 57.

[5]C. J. Sparks, to be published.

[6]R. W. Hendricks, Rev. Sci. Instr. 40, 1216 (1969).

[7]R. W. Hendricks, to be published.

[8]R. W. Hendricks, to be published.

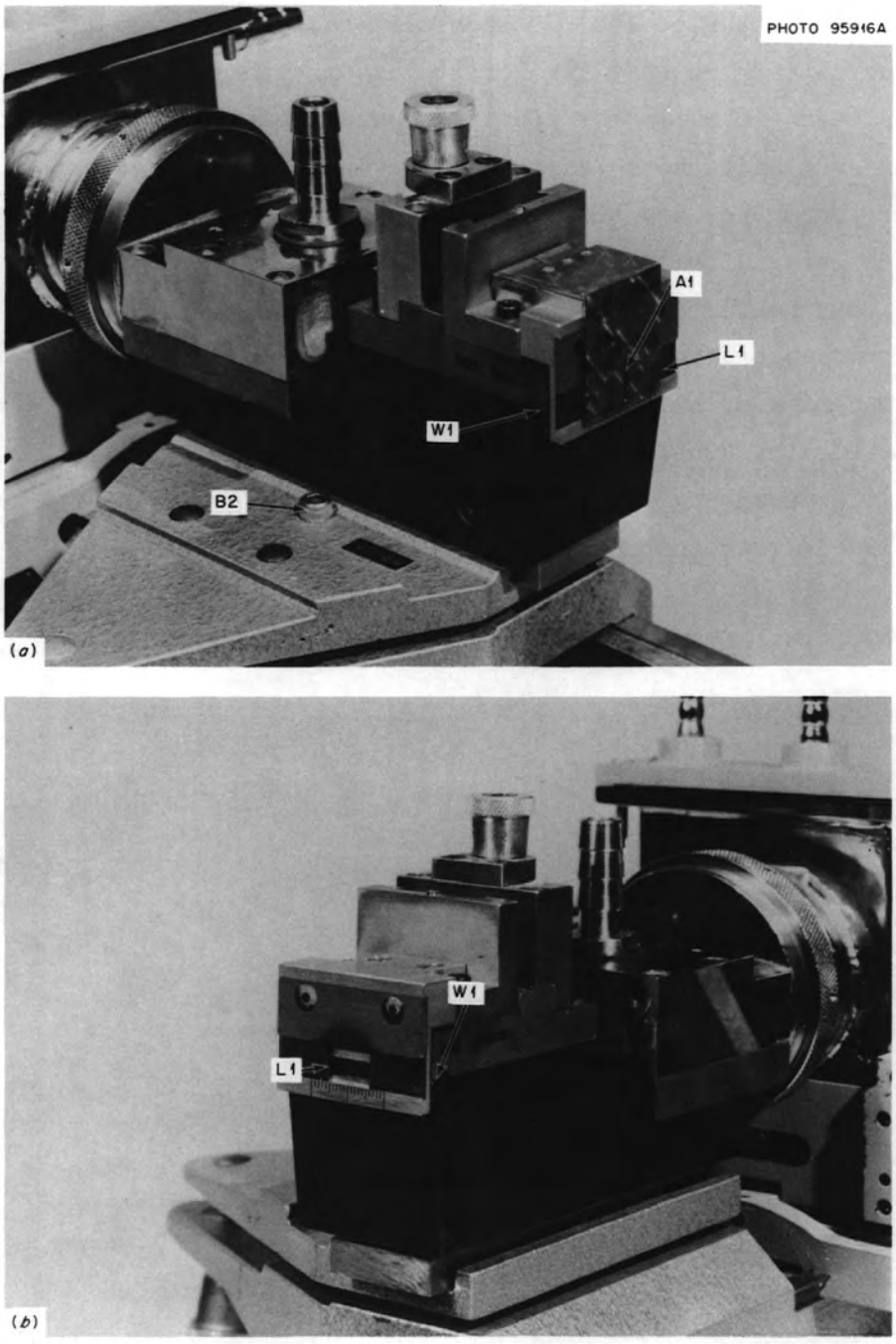

Fig. 2. Kratky Camera Entrance Slits. (a) Special alignment slit A1 mounted on the standard length collimation slit L1. Also note special locking bolt B2 required with systems equipped with vacuum bellows. (b) Length collimation slit L1.

mounting bracket [Fig. 1(b)]. Each slit has an engraved metric scale and fiducial mark so that its horizontal displacement can be measured. Care was taken to align the fiducial marks so that a setting of 0.0 mm centered each slit on the camera axis. The advantage of these slits is primarily that they may be mounted or dismounted from the camera without interfering with the setting of any other slit in the collimation system, thus making it easy to check the alignment at any time. As will become apparent in the following discussion, these auxiliary slits are a convenience, not a necessity. Their functions may be performed by using L1, the double-slit alignment device, and the special slit A3 (Fig. 3) for scanning the length of the beam. The latter two devices are available from the manufacturer.

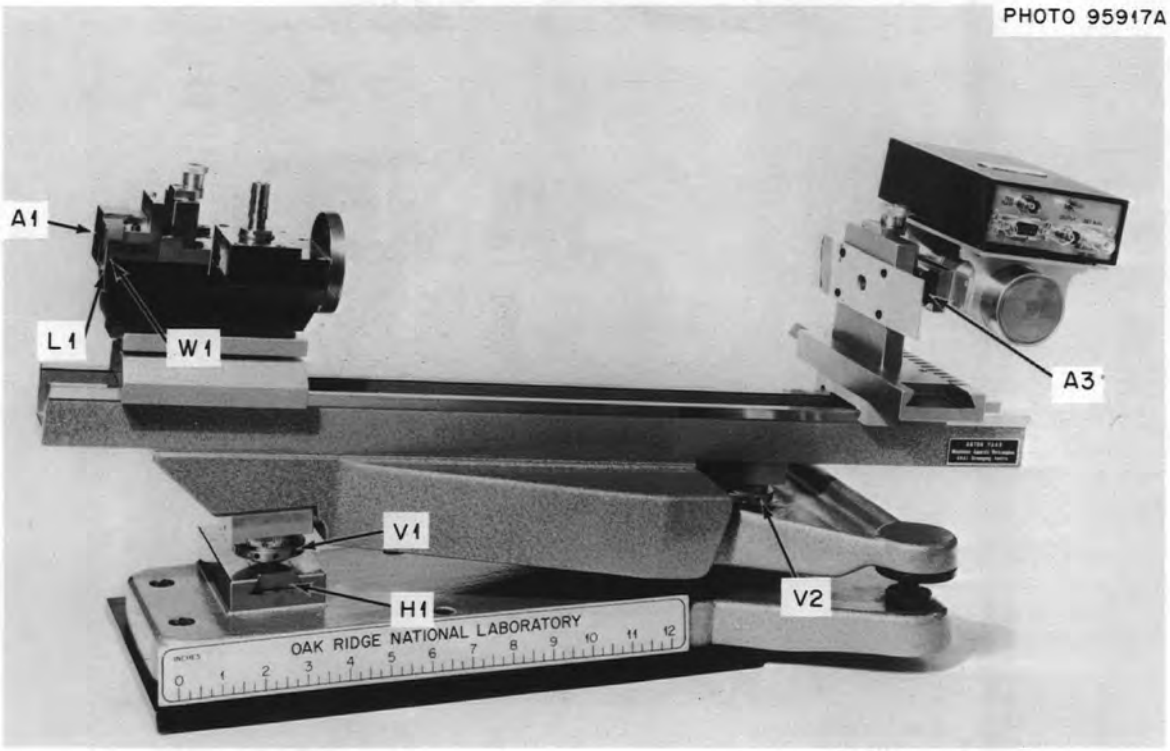

Fig. 3. Special Kratky Alignment Slit A3 for Measuring the Transmitted Power along the Length of the Incident Beam.

Several steps in the alignment procedure require that slit W1 (Fig. 2) be changed. It is necessary that the camera not be moved relative to the focal spot during such a change. The radiation shields between the tube face and the entrance slit must be designed with this requirement in mind. In our laboratory, this problem has been solved by mounting each camera on a slide which moves in a dovetail with a motion parallel to the camera axis. The dovetail and slide have a fiducial mark and metric scale, respectively, which allows the camera to be backed away from the tube face a known distance. After interchange of the slits, the camera may be returned into the radiation shields to precisely the same location.

A schematic drawing of the camera is shown in Fig. 4 to locate the important camera parameters. Horizontal and vertical cross sections of the collimation system are shown in Fig. 4(a) and (b), respectively.

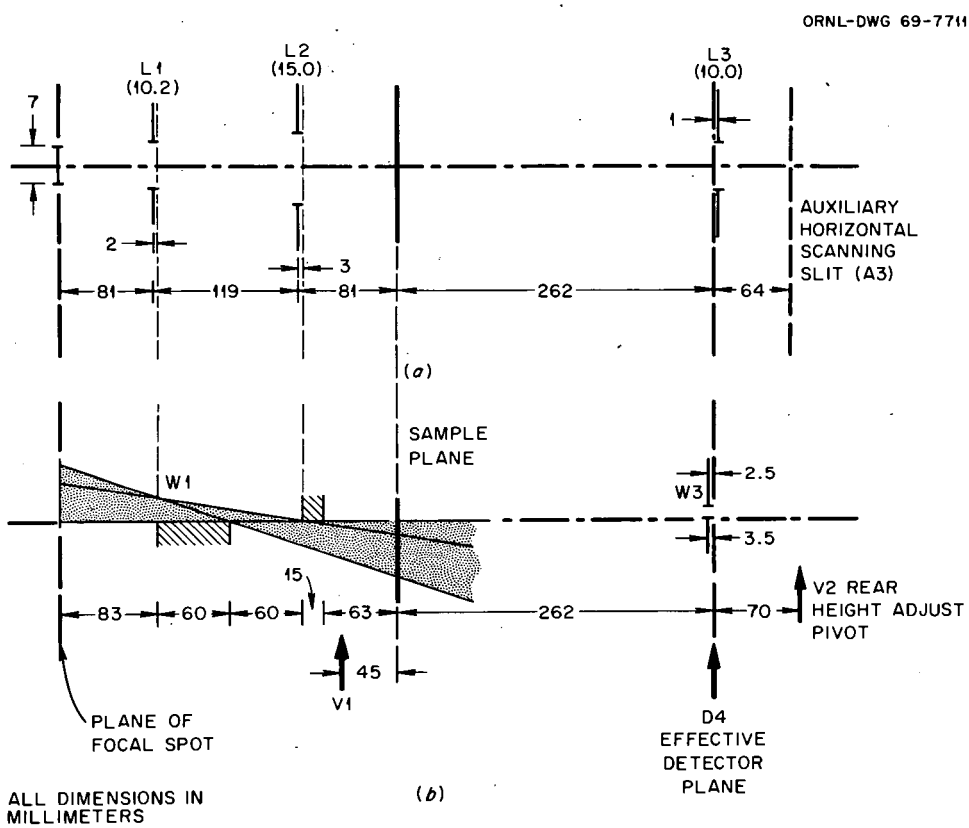

Fig. 4. Schematic Drawing of Collimation System. (a) Horizontal cross section of camera. (b) Vertical cross section of camera. Dimensions are those used in this laboratory (in millimeters).

The dimensions required to compute the width and length weighting functions from the results of Hendricks and Schmidt[9] and Buchanan and Hendricks[10] may readily be obtained from these diagrams. The dimensions shown are not critical, but are those in use in this laboratory.

It is important to note that the special Kratky focal spot (dimensions 2.8 × 7.0 mm) has been used in this study. If other focal spot dimensions are used, appropriate changes in the location of slit A1 must be made in steps B and C of the following discussion to ensure that the detector is viewing the ends of the focal spot.

THE ALIGNMENT PROCEDURE

A. Preliminary Adjustments

Mount the components of the camera as shown in Fig. 1, p. 2. Adjust the height of the front and back vertical translations V1 and V2 so that the camera takeoff angle is approximately 6° from the horizontal and so that the collimator is aimed by eye to point at the focal spot. Adjust slides H1 and H2 to make the camera axis perpendicular to the tube face and centered on the exit port of the x-ray tube. Put in a 20-μ entrance slit W1. In front of it mount the length slit L1 and the auxiliary vertical slit A1. The jaws of L1 should be wide open. Set the length of the second slit L2 to 1.0 mm centered about 0.0 mm (the camera axis). Place the vacuum cover over the collimator, but do not evacuate. Ensure that the specimen chamber is empty. Mount all radiation shielding and slide the camera up to the tube face. Set the detector slit W3 at 0.000-mm vertical displacement by adjusting the 2θ micrometer D4. Open the receiving slit width W3 and length L3 as large as possible (2.5 × 30.0 mm). Directly behind L3 insert the second

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[9]R. W. Hendricks and P. W. Schmidt, Acta Phys. Austriaca 26, 97 (1967).

[10]Mary G. Buchanan and R. W. Hendricks, Program Weight: A FORTRAN IV Program for Evaluation of Weighting Functions Used in Small-Angle X-Ray Scattering, ORNL-TM-1950 (September 1967).

auxiliary vertical slit A2. Mount the detector behind A2. If a monochromator is to be used, do not mount it until later. If the camera is equipped with vacuum bellows, release the O-ring seals, but tighten locking bolt B2. The purpose of B2 is to keep the collimating system from moving when the camera is later evacuated. Turn on the x-ray tube at low power (typically 45 kv and 1 ma).

With A1 centered at 0.0, make a horizontal scan of the direct beam with A2. (It may be necessary to raise or lower V2 to get the collimator pointing at the focal spot. It may also be necessary to use some attenuating foils to reduce the intensity of the direct beam.) If the profile of this scan is not centered on zero, the vacuum tank is not centered on the camera axis. Adjust the pinning screws H3 in the appropriate direction and re-scan. Repeat until the tank is centered.

If the auxiliary slit A1 is available, the camera is now ready to set the final lengths of slits L1 and L2. If A1 is not available it will be necessary to narrow L1 to 1.0 mm and use it in place of A1. In order to achieve maximum incident power on the sample, it is necessary for the entire focal spot to illuminate as much of the sample as possible without any part of the direct beam striking any surface in the system except the designated collimation slits. With the aid of a scale diagram such as shown in Fig. 4(a), the length of L2 is computed without regard to L1 so as to irradiate a maximum amount of the sample. It is important that the most divergent rays do not strike the sample holder or the inside walls of the vacuum path to the detector. Once L2 is determined, the length of L1 is computed to just intercept the rays from the edge of the focal spot to the edges of L2. Having set L1 and L2, replace auxiliary slit A1 and the vacuum slit cover. Do not evacuate the camera.

B. Tilting the Collimator Parallel to the Focal Spot

In the previous section, the vacuum tank was centered on the camera axis, and the final lengths of the collimation slits L1 and L2 were set. In this and the following five sections, we shall step-by-step align the collimator parallel to and pointing at the focal spot and align all

succeeding edges which limit the direct beam so that they are parallel to the collimator.

Set A1 at +3.0-mm displacement and A2 at +10.0-mm displacement. As shown in Fig. 5, the detector will now see a section of the focal spot which is centered 1.8 mm from the right-hand end of the focal spot. Make a vertical scan of the entrance slit across the focal spot by adjusting V1. Repeat the process on the left-hand end of the focal spot by setting A1 at −3.0 mm and A2 at −10.0 mm. If one end of the focal spot is higher than the other, the two vertical scanning curves will be displaced from each other as in Fig. 6(a). Compute how much tilt of the collimator is required to bring the two curves into coincidence. Loosen locking bolt B2; adjust T1 for the proper tilt; retighten B2 and repeat the scans. Acceptable final alignment is illustrated in Fig. 6(b).

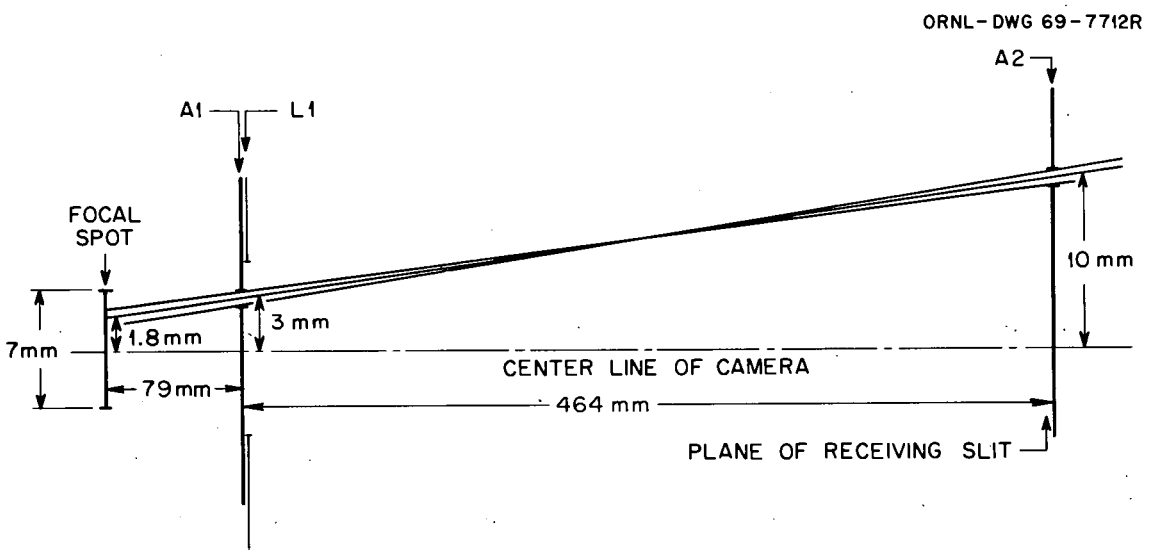

Fig. 5. Ray Diagram of Geometry for Adjusting the Tilt of the Monochromator.

The vertical scans of the focal spot discussed in the preceding paragraph can be used as a measurement of the width of the focal spot. From the diagram in Fig. 4, p. 7, it can be seen that moving the vertical height adjustment (V1) through a certain distance results in the

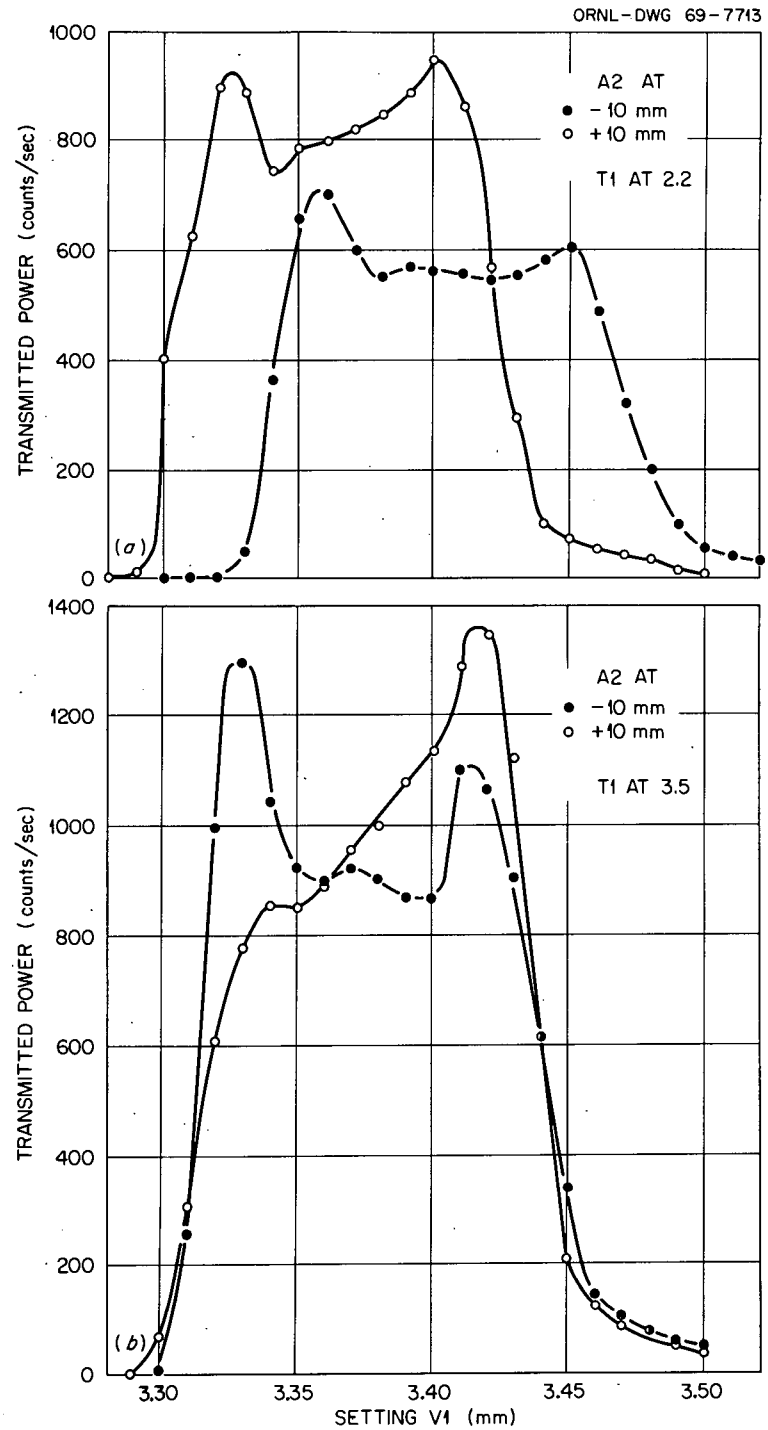

Fig. 6. Collimator Tilt Alignment Curves. (a) Misaligned. (b) Aligned. The curves in (b) do not superimpose perfectly because of the intensity variations along the focal spot (see section on "Measuring the Focal Spot Intensity Distribution," for discussion).

observed part of the focal spot moving by 613/377 = 1.63 times that distance. Hence, in Fig. 6(b) the complete scan of the focal spot required a movement of V1 of 0.16 mm which corresponds to a motion of 0.26 mm in the plane of the focal spot. The observed scanning curve width is the true focal spot width plus the width of the scanning beam. With W1 set at 0.02 mm the width of the scanning beam in the plane of the focal spot is (143/60)(0.02) = 0.05 mm. Unfolding this from 0.26 mm we get 0.21 mm for the width of the focal spot in our example.

C. Pointing the Camera Axis at the Center of the Focal Spot

Vertical Center

Set the vertical height adjustment V1 so that the collimator is at the center of its vertical scan across the focal spot as determined in step B, p. 9. In the example of Fig. 6(b), a setting of 3.38 would be appropriate.

Horizontal Center

Set auxiliary slit A1 at +3.0 mm, and scan slit A2 across the receiving slit. In this situation, A2 is viewing the region very near the right-hand end of the focal spot. Shift A1 to −3.0 mm and repeat the A2 scan. If the camera axis is not pointed at the center of the focal spot these curves will not be symmetric about the zero of A2 translation. If such is the case, compute the required horizontal camera translation, move H2, and repeat the procedure until the curves are symmetric about the A2 zero as shown in Fig. 7. If the misalignment is very bad, it may be necessary to adjust H1 as well. For final alignment, H2 motion is to be preferred because of its greater lever arm and hence greater sensitivity.

D. Adjusting the Jaws of W3 to be Parallel

Remove auxiliary slit A1. With auxiliary slit A2 at −10 mm and W3 wide open, raise the lower edge of W3 using the 2θ micrometer D4 until the intensity is reduced to one-half. The bottom jaw is now in the

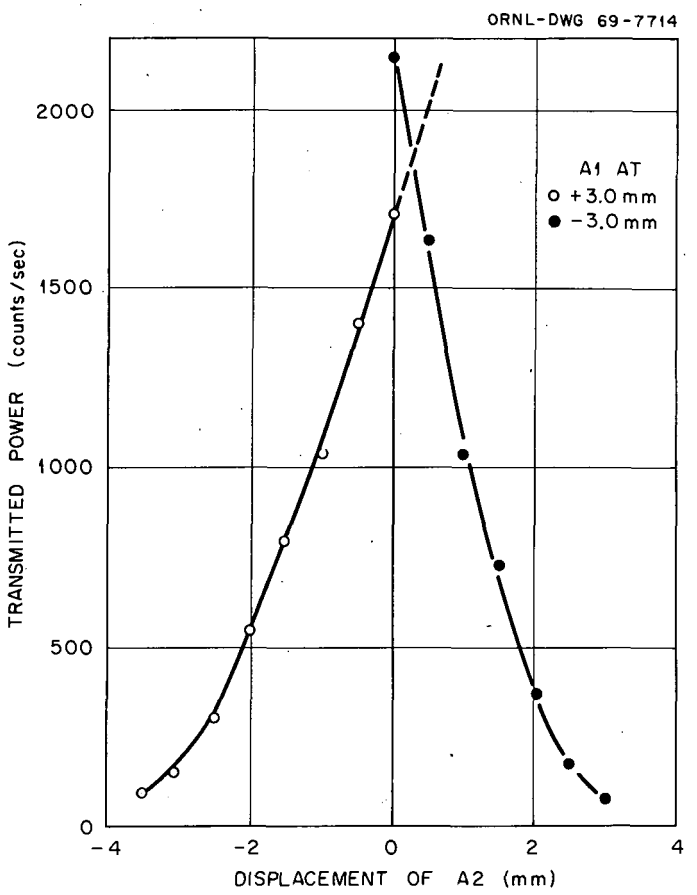

Fig. 7. Horizontal Scan of the Two Ends of the Focal Spot Required for Pointing the Collimator at the Horizontal Center of the Focal Spot. Figure illustrates a slightly misaligned collimator.

center of the beam. A "closing curve" is now measured on W3 by recording the x-ray intensity as a function of D1, the upper jaw displacement micrometer reading. Only the last 0.03 mm are important; make readings every 0.005 mm. Plot the curve and note the setting at which it extrapolates to zero opening, as in Fig. 8. Now open slit W3 again and repeat this process with slit A2 at +10 mm. It may be necessary to readjust the height of W3 to put the bottom jaw in the center of the beam since the bottom jaw is not necessarily parallel to the beam at this point. If the two settings for zero opening do not coincide the jaws are not parallel. Tilt the upper jaw with screw T3 until the settings for zero opening at −10.0 and +10.0 mm coincide as closely as possible. Satisfactory results are illustrated in Fig. 8 for three positions of A2: −10, 0, and +10 mm. Note that the order of the curves for 0 and −10 mm

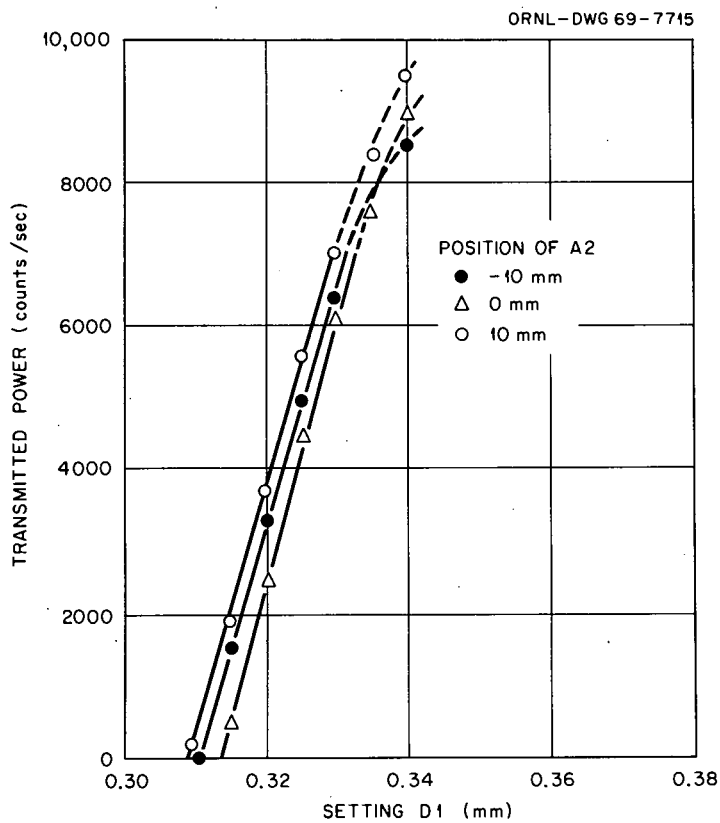

Fig. 8. Closing Curves for an Aligned Receiving Slit. The curves do not superimpose perfectly because the receiving slits are apparently not perfect straight edges.

are reversed, thus suggesting that the failure of the closing curves to agree more closely is because the slit jaws are not straight edges. Similar observations have been made independently by Patel and Schmidt.[11]

E. Tilting the Receiving Slit to be Parallel to the Collimated X-Ray Beam

Set the width of the receiving slit W3 to 20 μ. With the auxiliary slit A2 at −10 mm, make a vertical scan of the collimated beam (a rocking curve) using the 2θ micrometer D4. Repeat the scan with A2 at +10 mm. If the peaks of these two curves do not coincide, the receiving slit is not parallel to the entrance slit W1 and hence is not parallel

[11] I. S. Patel and P. W. Schmidt, private communication.

to the collimated x-ray beam. Typical results for a misaligned slit are shown in Fig. 9(a).

Set the 2θ micrometer (D4) at a vertical displacement corresponding to the high angle side of one of the rocking curves. Measure the intensity for A2 at +10 and −10 mm. Adjust the tilt micrometer T2 until the intensities are the same at both settings of A2. At this point repeat the complete rocking curves. Further minor adjustments of T2 may be required to achieve the best average match over the entire rocking curves. Typical results for an aligned receiving slit are shown in Fig. 9(b).

It is appropriate to account for the use of the narrow (20 μ) entrance and receiving slits. Since the rocking curve is the convolution of the receiving slit with the direct beam, to achieve high resolution in this step of the alignment procedure (i.e., good separation of the two rocking curves for small misalignments) it is necessary that (a) the direct beam be narrow and (b) the receiving slit be narrow compared to the width of the beam.

F. Tilting the Specimen Chamber to be Parallel to the Collimation System

Leave slits W1 and W3 at 20 μ and set W3 at the peak of the rocking curve. Insert an empty liquid sample holder into the specimen chamber. With auxiliary slit A2 at −10 mm, raise the chamber vertically (motion D2) until the beam just starts to get through the 1-mm slot in the sample holder. Repeat with slit A2 at +10 mm. Tilt (D4) the sample holder until the beam emerges at the same point in each case. Results for a misaligned chamber are shown in Fig. 10. For the aligned chamber, the curves will coincide.

G. Evacuating the Camera

Remove the liquid sample holder. Seal both sets of bellows. Check that B2 is securely tightened; if it is not, then when the camera is evacuated the air pressure will cause the bellows to collapse and the collimating system will tilt. Evacuate the camera. Steps B, E, and F

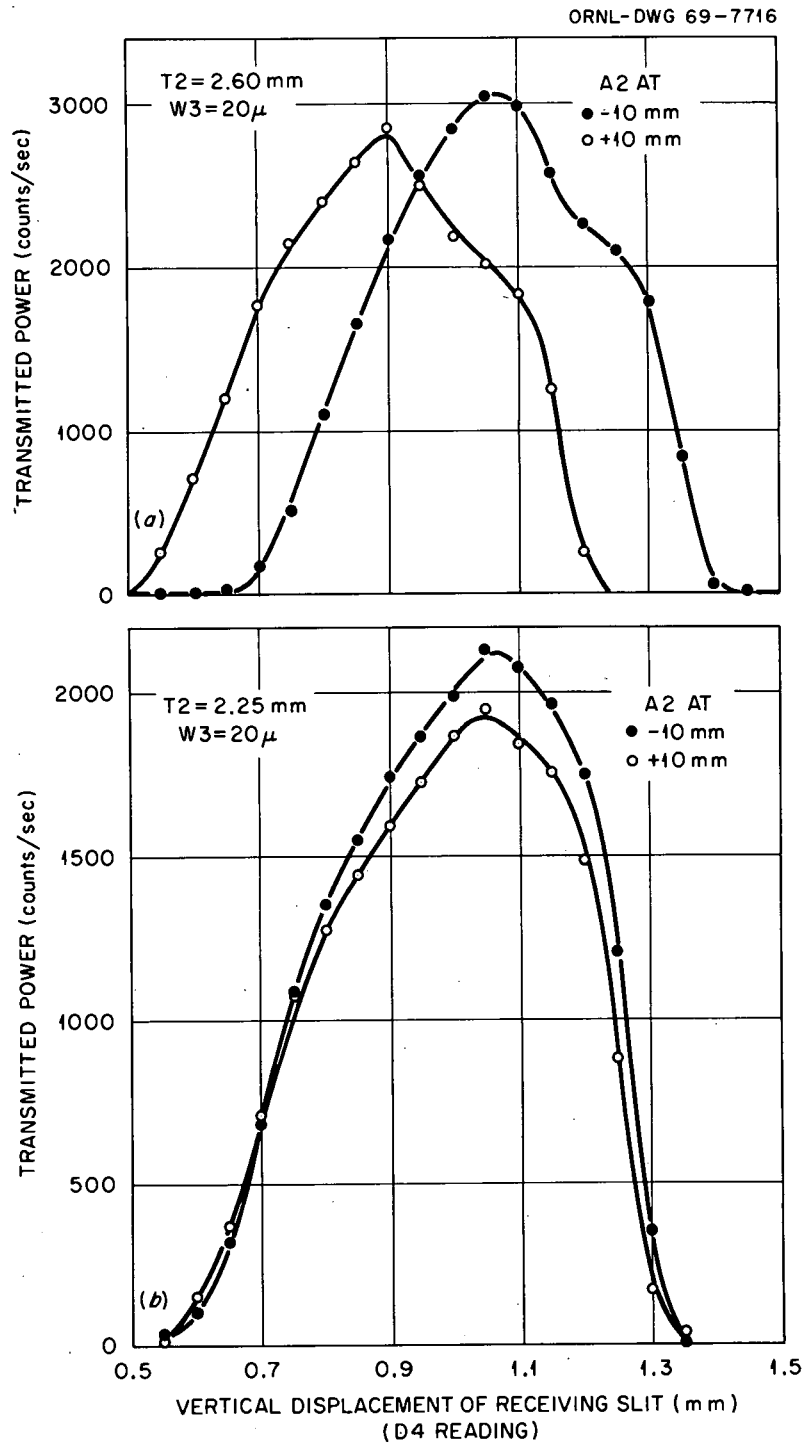

Fig. 9. Receiving Slit W3 Tilt Alignment. (a) Rocking curves obtained with auxiliary alignment slit A2 at ±10 mm for a misaligned receiving slit. (b) Same as (a), except tilt of receiving slit is now aligned parallel to x-ray beam.

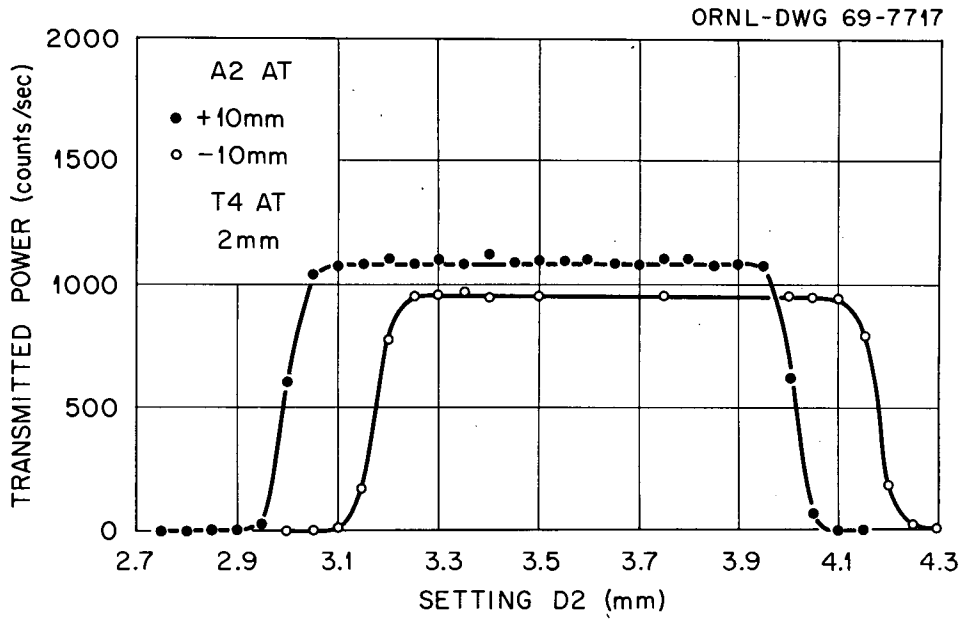

Fig. 10. Sample Chamber Tilt Alignment Curves. Transmitted power through each end of a 1-mm-high liquid sample cell as a function of vertical displacement of sample chamber.

may now be rechecked. The only one that is likely to need readjusting is step E. Tightening the retaining rings on the bellows tends to twist slit W3. It can generally be readjusted to its correct setting by simply grasping the yoke and twisting until the peaks of the rocking curves coincide once more.

H. Setting the Entrance and Receiving Slits and Repointing the Collimator at the Focal Spot

Remove the 20-μ entrance slit and replace it with one of the desired width, consistent with incident beam power and angular resolution requirements. Open the receiving slit W3 to full width and set the 2θ displacement micrometer D4 to 0.000 mm. Remount the auxiliary alignment slits A1 and A2. Repeat the horizontal scans across the ends of the focal spot as described in step C, p. 12, to ensure that it is still centered. Usually no realignment will be required. Remove both auxiliary slits A1 and A2 and using V1 make a vertical scan across the focal spot. A typical curve is shown in Fig. 11. Set V1 at the position which gave maximum intensity (denoted by the arrow in Fig. 11).

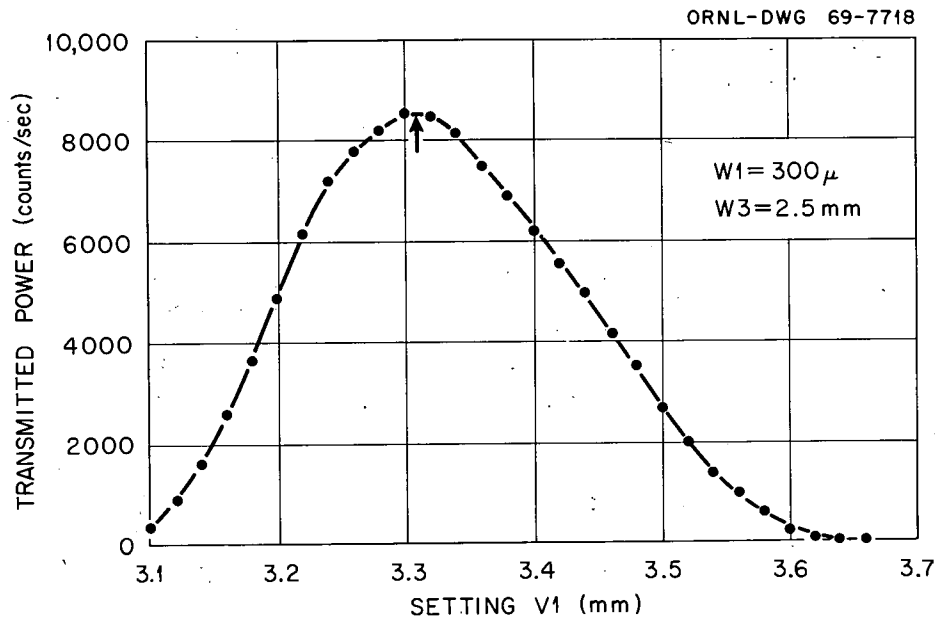

Fig. 11. Vertical Scan of the Focal Spot with 300-μ Entrance Slit. For maximum transmitted power, camera vertical height V1 should be set at 3.31 mm.

Using the micrometer D1 and the zero position determined from the "closing curves" described in step D, p. 12, set receiving slit W3 to the desired width. Set slit L3 to the desired length.

I. Mounting the Monochromator

Mount the monochromating crystal in the box such that the axis of monochromator rotation passes through the exposed surface of the crystal. Rotate the crystal with R1 to the Bragg angle (26.6° 2θ for hot-pressed pyrolytic graphite). Mount the box (without the detector) behind the receiving slit W3. Position W3 in an intense region of the direct beam and lower the assembly with micrometer D3 until the x-ray beam just passes over the top of the crystal as seen on a fluorescent screen. Raise the box until the beam is centered on the crystal. Adjust the monochromator Bragg angle $2\theta_M$ until a diffracted beam is observed on the fluorescent screen. Mount a lead beam stop behind the crystal, and lower the radiation shield D5 but do not intercept any of the incident or diffracted beam. Mount the detector bracket, detector, and preamplifier

such that the diffracted beam enters the center of the proportional counter. Fine-tune the monochromator Bragg angle with R1 to achieve a maximum diffracted intensity.

J. Measuring the Rocking Curve and Computing the Weighting Functions

At this point the collimation system should be completely aligned. To complete the procedure, the experimental and theoretical rocking curves are to be compared. The theoretical weighting functions have been computed with the program described by Buchanan and Hendricks[12] for the collimation system dimensions taken from Fig. 4(a) and (b), p. 7. The results are shown in Fig. 12. The experimental slit-width weighting function is, in fact, the rocking curve obtained by scanning the receiving slit W3 through the direct beam. The results are shown in Fig. 12(a); the agreement is considered to be good.

K. Measuring the Focal Spot Intensity Distribution

Although not essential, it is instructive to measure the intensity distribution along the length of the focal spot. This is done by setting A1 at 0.0 mm and scanning the length of the direct beam with a narrow vertical slit at the back of the camera. Depending on the entrance slit to focal spot distance [81 mm in Fig. 4(a)], the length of the direct beam at the receiving slit is often longer than the maximum length of L2. In this case, the special Kratky device (slit A3, Fig. 3, p. 6) for measuring the length of the beam is needed. After aligning the tilt of the collimator (step B), the specimen chamber and vacuum tank are removed, and A3 is mounted as in Fig. 3. Slit A1 now becomes an effective pinhole, and by scanning A3 across the full length of the direct beam, the intensity distribution along the focal spot is obtained. A typical result is shown in Fig. 13. The maxima and minima are a result of the helical coil construction of the filament. It is

[12]Mary G. Buchanan and R. W. Hendricks, Program Weight: A FORTRAN IV Program for Evaluation of Weighting Functions Used in Small-Angle X-Ray Scattering, ORNL-TM-1950 (September 1967).

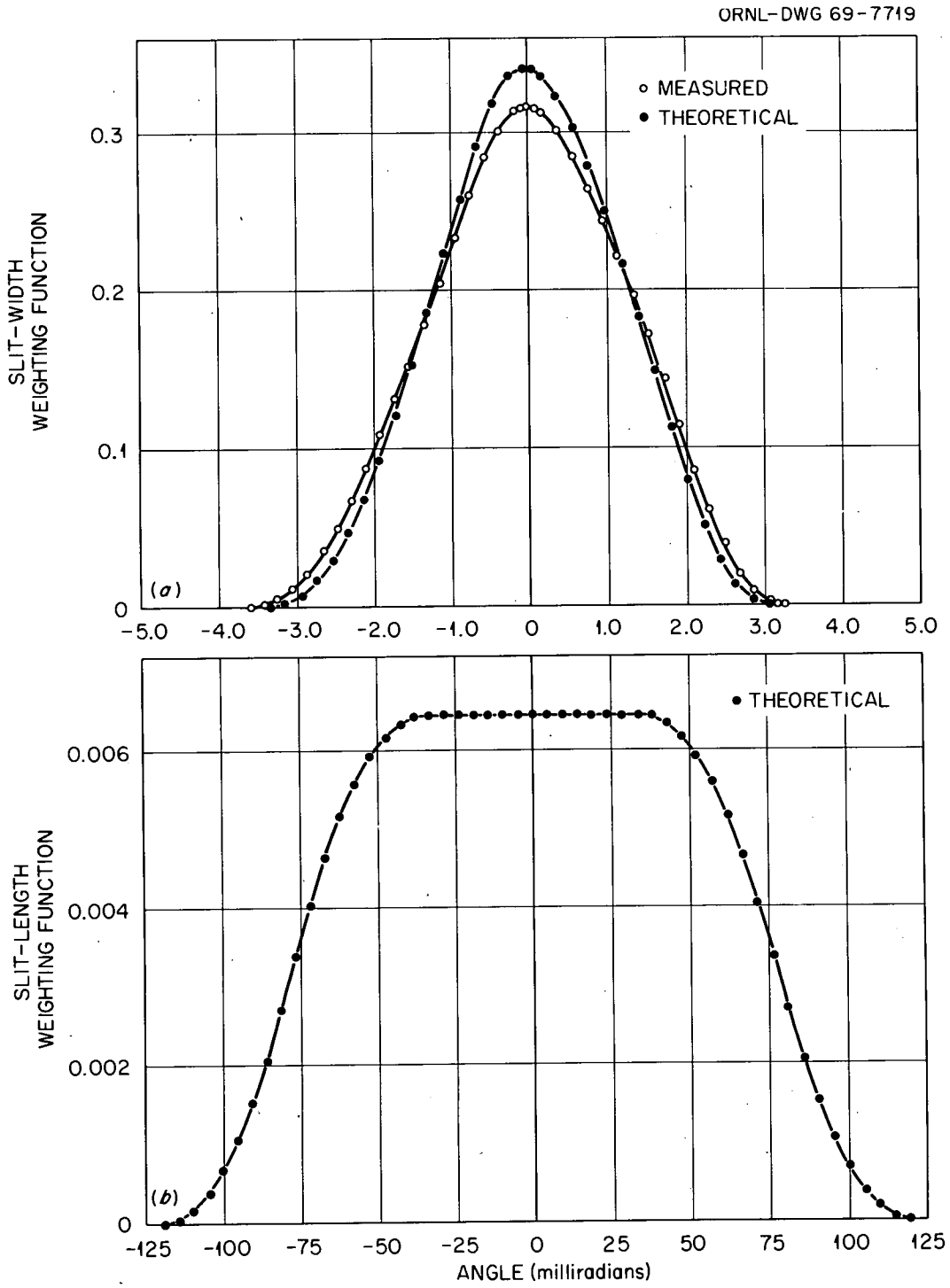

Fig. 12. Weighting Functions for the Collimation System of Fig. 4(a) and (b). (a) Experimentally observed and theoretical slit-width weighting functions. (b) Theoretical slit-length weighting function.

Fig. 13. X-Ray Intensity Along the Length of the Focal Spot. Maxima and minima are due to the coils in the filament.

these maxima and minima which cause the imperfect symmetry of the vertical scans of the focal spot observed in Fig. 6(b), p. 11. If, in the procedure of step B, the slit A2 was moved slightly in either direction from the plus or minus setting, the shapes of the peaks in Fig. 6(b) changed, consistent with the above arguments. The slit-length weighting function has been computed using this focal spot intensity distribution and the camera geometry of Fig. 4(a), p. 7. The slit-length weighting function computed for a camera of the same geometry but assuming a uniform focal spot intensity has been shown in Fig. 12(b), p. 20. It was found that these weighting functions are different only in their outer regions, and there they differ only by a few percent. In the flat central region, they agree to within 0.1%.

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