Brookhaven Symp. Biology 32
Upton, NY June 1-4, 1982
(Publ) Plenum Press, B. Schoenborn (ed)
NY
NEUTRONS IN BIOLOGY

BIO 4040
BNL - 34169

Conf Art
Bk Chpt

119

BNL--34169

DE84 006757

A NEW NEUTRON SMALL-ANGLE DIFFRACTION INSTRUMENT AT THE BROOKHAVEN HIGH FLUX BEAM REACTOR

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INTRODUCTION

The construction of a reentrant cold source at the Brookhaven High Flux Beam Reactor over the past few years, and its successful operation since 1981, have made available an intense long wavelength neutron source that is ideally suited for the investigation of complex biological structures (1). This development and the more recent increase in reactor power to 60 MW encouraged the construction of a new small-angle neutron diffraction instrument.

The new instrument utilizes cold neutrons emerging from a series of straight neutron guides. A multilayered monochromator (2) is used in combination with a short collimator to obtain a monochromatized beam with a wavelength between 4 and 10 Å and a wavelength spread of about 10%. The flux at 5 Å exceeds 10^6 ns⁻¹ cm⁻² in a typical beam of 6-mm diameter at the sample. The spectrometer itself incorporates provisions for computer-controlled positioning of samples and a two-dimensional detector. At a sample-detector distance between 50 and 200 cm the detector can be centered at scattering angles of up to 45°. The beam-defining components, the monochromator, the collimator, and various slits, are easily accessible and exchangeable for alternative devices. These features make the instrument modular and give it flexibility approaching that of standard x-ray equipment.

The facility in its first year on line was used most of the time for solution scattering experiments. These included experiments to investigate the structure of plasma lipoproteins (3), to further the triangulation of the small ribosomal subunit (4), and to characterize micellar microemulsions (5). The spectrometer is

*Research carried out under the auspices of the U.S.D.O.E.

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equally suited to measuring diffraction at higher angles on oriented specimens (6).

In this paper, the components of the instrument are described, and its performance is discussed. Characteristic parameters are compiled in Table 1.

Table 1. Characteristics of H9B Small-Angle Spectrometer

Wavelength (λ)	4 to 10 Å
Wavelength resolution ($\Delta\lambda/\lambda$)	0.06 to 0.15 (FWHM)
Beam divergence	14' horizontal, 37' vertical
Beam diameter at sample	6 mm, typical (2 to 20 mm)
Flux at sample	1×10^6 to 2×10^6 ns ⁻¹ cm ⁻²
Best first-order resolution	~1000 Å
Best order-to-order resolution	~1500 Å
Detector	17 x 17 cm; 128 x 128 pixel

COLD SOURCE

The cold neutron source at the Brookhaven High Flux Beam Reactor (HFBR) is inserted into the tangential, horizontal beam thimble H9 to within 74 cm of the center of the reactor core.* At this distance, the thermal neutron flux in the surrounding D₂O reflector exceeds 2×10^{14} ns⁻¹ cm⁻² (7,8). The cold moderator itself, 1.4 liters of liquid hydrogen at 20°K and 5 atmospheres, is contained in an oblate ellipsoidal chamber with a diameter of 20 cm and a maximal thickness of 7 cm (9). This aluminum vessel is kept cold by helium gas circulating through veins on its surface. Thermal isolation from the surroundings is achieved by evacuation of a protective cap, which, in turn, is cooled by a helium blanket.

APPARENT SOURCE

Neutrons from the cold source are transferred to the monochromator by a series of straight neutron guides (10), as shown in Figure 1, having the dimensions listed in Table 2. The entrance window of the first neutron guide is located at a radius of 155 cm from the center of the core and is thus protected in a niche of the thermal neutron shield surrounding the reactor vessel. This window is separated from the cold source by 84 cm of evacuated

*Diagrams of the beam and spectrometer layout at the HFBR have been published (8,11-12).

Fig. 1. Floor plan of the H9B beamline. The reentrant cold source, CS, is part of the H9 plug, as are the inner Ni guide and the shutter, S. Be is the beryllium filter. MC is the monochromator cavity. A and C represent two other cold beam lines in the biological shield, BS. RC is the reactor core, the shaded area is its thermal neutron shield, and RW is the reactor wall.

Table 2. H9B Neutron Guides

Component	Cross section (mm) height x width	Length (cm)	Position* (cm)	
Inner Ni guide	44.5 x 19	198	84-282	N ₂ filled** 1 stainless window 1 Al window
Shutter Ni guide	45.3 x 19.3	61	283-344	Evacuated 2 Al windows
Be filter	51 x 32	20.3		At 78°K in Dewar Dewar in air 4 Al windows
Outer Ni guide	49.5 x 24	195.6	381-577	Evacuated 1 Al window
Ni-coated glass guide	49.5 x 24	2x100	577-777	Evacuated 1 Al window

*The origin of this scale is the cold source.

**This is a particularity of the current plug. The mark II plug, due to be inserted in 1983, will have an evacuated inner guide.

space. It subtends a solid angle of 0.0012 sterad. Taking into account the critical glancing angle in the first Ni guide ($\theta_c/\lambda = 5.8$ min) (12), the area of cold moderator that contributes neutrons to the raw beam is calculated as 20 cm^2 for 5-Å neutrons and as 34 cm^2 for 10-Å neutrons. Since these areas lie well within the source plane, the first neutron guide is fully illuminated. The neutron guides are interrupted at the reactor wall to accommodate a liquid nitrogen cooled polycrystalline beryllium filter which eliminates, by scattering, thermal neutrons with wavelengths $< 3.96 \text{ Å}$.

In the apparent source which is the exit aperture of the last neutron guide, the neutron intensity distribution is uniform along vertical lines. An intensity variation of 3% was measured in the central portion of a scan along a horizontal line. The divergence angle distribution in this apparent source was surveyed by recording neutrons passing through narrow horizontal or vertical slits with polaroid film at a distance from the slits. The gradual changes in angular characteristics across the source were those expected from geometrical considerations. Pinhole images of the apparent source (Figure 2) were recorded with the two-dimensional detector. The horizontal width of about 15 min, and the vertical width of about 36 min, of the central and most intense part in the pinhole image correspond to the angular apertures of the shutter. A dead angle due to the guideless filter gap and to the stepping in guide dimensions confines those neutrons emerging directly from the cold source and the inner neutron guides. Radiation with larger divergence angles emerges after multiple reflections out to the critical glancing angle of the wavelength considered. The guideless gap might manifest again when one is considering longer wavelength neutrons at high divergence angles. Even though this effect is partially annealed in the monochromatized beam, where several divergence angle distributions are superimposed with angular shifts, it is noted as a reason for a loss of 10% in source brilliance.

The neutron flux spectrum in the apparent source was measured by using a multilayer monochromator as an analyzer. A square 2-mm sample beam was emergent through the four-blade slit system (Figure 4) from the center of the apparent source. After passing through a further 2-mm aperture this beam was incident on the analyzing monochromator mounted on the sample axis. The peak intensities recorded at each rocking angle are shown in Figure 3 as a function of the wavelength. Superimposed on these data is a Maxwellian flux spectrum (13) calculated for an apparent temperature of 35°K . This non-physical distribution (T cold source $\approx 20^\circ\text{K}$) serves a descriptive purpose only. Since the wavelength distribution was measured with a multilayer monochromator, as is used in the standard configuration, it is a measure of the intensities actually obtainable at the various wavelengths.

By comparing the cold spectrum with one measured under identical experimental conditions on a beam through the evacuated moderator chamber, a gain between 10 ($\lambda = 5 \text{ \AA}$) and 18 ($\lambda \geq 10 \text{ \AA}$) was calculated, as shown in Figure 3. These values are in excellent agreement with similar measurements performed on the H9A cold

Fig. 2. The angular distribution of neutrons emerging from the center of the apparent source is mapped. A 2-mm-square beam was defined by a pair of slits at the exit of the neutron guides. The divergent beam was recorded with the two-dimensional detector 340 cm from the slits. Non-thermal contributions to the intensity distribution were subtracted by using a measurement taken with 2.5 cm of borated material in the beam. The beam is incident from underneath the image plane. The angular distribution is truncated on the right because of a slight misalignment of the outer neutron guides. The vertical (V) and horizontal (H) projections of the angle distribution were obtained by averaging a few profiles near the center of the peak. On the upper margin of these graphs a divergence angle scale is given. The solid bars associated with the scales mark the solid angles within which neutrons may emerge. These angular regions were calculated by ray tracing. The gaps between bars indicate dead angles due to the guideless filter gap and the stepping in guide dimensions.

Fig. 3. The neutron flux spectrum in the apparent source is plotted on a relative scale. The upper spectrum is that of the liquid hydrogen moderated neutron beam (H_2 20°K), and the lower spectrum was measured on a beam through the evacuated moderator chamber (empty). Intensities at each wavelength represent the peak intensity recorded with a multilayer monochromator ($d = 73 \text{ \AA}$) at a given rocking angle. The full line superimposed on the cold moderator spectrum is a Maxwellian spectrum calculated for an apparent temperature of 35°K. The shaded bar at 3.96 $\text{\AA}$ represents the wavelength cutoff of the Be filter. In the insert, the gain due to the cold moderator is plotted as a function of the wavelength.

moderator beam (14). At larger wavelengths the measured gain is smaller than values obtained on the geometrically very similar cold source at the EL3 reactor at Saclay, which is inserted into a radial beam hole (15). The large discrepancy between the measured gains and those calculated by comparing Maxwellian distributions shifted from the reflector temperature to the moderator temperature, as shown by Jacrot (15), are due mainly to the non-isotropic flux at the cold source, to the substantial absorption cross section of hydrogen, and to incomplete thermalization in the small hydrogen volume.

MONOCHROMATOR

The length of the H9B beam line between reactor face and outer walls is about 12 m. This spatial constraint makes it impossible to incorporate into the beam line a bent neutron guide (16) of sufficient cross-sectional area. [For an application of a bent guide, see Ibel (17).] The alternative straight guides transmit a beam poisoned by γ -radiation and a residual amount of fast neutrons that has not been eliminated by the beryllium filter. It is therefore imperative to choose a crystal monochromator for spatial separation of a primary beam from the incident radiation.

A multilayered monochromator (2) followed by a short collimator is used to obtain a well defined primary beam with a large wavelength spread. The monochromator plates are fabricated in this laboratory (18,25) by depositing alternate thin layers of Fe and Mn on a flat glass substrate. The parameters of a multilayer (FM 119) that has been used for more than a year as a 5-Å monochromator are examples of characteristic dimensions. It is built

Fig. 4. Horizontal cross section of monochromator cavity. Part of the last neutron guide (NG) is shown on the left. ES is a four-blade entrance slit. The multilayer monochromator (MM) is mounted on a rotation stage with vertical rotation axis. Its tilt and lateral position are also adjustable. The monochromator cavity is shielded with borated materials (shaded) and lined with lead as shown. The backstop (BS), the next unit downstream, is aligned parallel to the monochromatic beam with an adjustable opening to match the collimator apertures. The primary beam monitor (M) and the absorber wheel (AW) are part of the backstop assembly. The collimator (C) is shown to interlock with the shielded backstop cavity.

up of 180 bilayers with a thickness of 73 Å and has a uniform reflectivity of about 80% across its area of 5 x 46 cm.

The monochromator is positioned close to the apparent source with its diffracting planes parallel to a vertical axis (Figure 4). At this distance it is illuminated essentially on its entire surface by neutrons of all divergence angles within the limits of the critical glancing angles. Since the first-order Bragg angle of a multilayer with a repeat distance of 50 to 100 Å is small (2° to 5°) compared with the beam divergence, the range of scattering angles covered by the divergent rays gives rise to a wavelength band pass, $\Delta\lambda/\lambda$, of 35 to 70%. Monochromatization within narrower limits is achieved by selecting an appropriate bundle of diffracted rays with the collimator. This scheme offers various advantages:

1. The wavelength can be tuned, as with any crystal monochromator, by rotating the multilayer about its vertical axis. This changes the beam geometry, and collimator and sample axis have to be realigned. Since a number of alignments are involved and since all angles are small, the actual change in wavelength may differ from the target value. The new wavelength has to be measured with a calibration experiment. The wavelength spread ($\Delta\lambda/\lambda$) is essentially unaffected by changing the wavelength in this way.

2. Alternatively the wavelength can be changed quickly and at a fixed beam geometry by replacing the multilayer monochromator by one having a different repeat distance. In this protocol, the change in wavelength is exactly predictable. In addition, because of the change in multilayer repeat distance, there is a favorable increase in wavelength spread with increasing wavelength.

3. The wavelength spread can be adapted to experimental requirements by selecting a multilayer with an appropriate bilayer spacing. For instance, in a collimated beam of 5-Å wavelength a $\Delta\lambda/\lambda$ of 6% can be obtained from a multilayer with 50-Å bilayer spacing, and a $\Delta\lambda/\lambda$ of 11% is obtained from a monochromator with a bilayer spacing of 100 Å.

Stacking disorder in the multilayer monochromator, due to variation in the number of atomic layers per monolayer, affects the reflected intensity and contributes to the wavelength spread. A rigorous treatment of the diffraction by ordered and disordered lamellar systems with long-range order was given by Caspar and Phillips (19). Given the wide-angled illumination, and the limited analytical resolution of the instrument, however, effects due to disorders contribute little to the angular and chromatic characteristics of the beam. To rationalize experimental observations with this instrument it is sufficient to consider the

monochromator as a perfect one-dimensional crystal. Properties of the primary beam will be discussed below after the collimation arrangement has been described.

COLLIMATOR

The collimator consists of three exchangeable circular cadmium apertures, equally spaced by 68 cm in a helium-filled tube. In addition to defining a beam of the desired cross-sectional area it is an indispensable part of the monochromatization system, as discussed above. The four-blade slit system between apparent source and monochromator can be used as a further collimating aperture to reduce the divergence of narrow beams. The last collimating aperture, on the other hand, is part of a telescopic extension that can be brought into contact with the sample containments. Two adjustable four-blade slits may be inserted at the two ends of the collimator to produce a rectangular beam.

The collimator is separated from the monochromator by a massive vertical slit that serves as a beamstop to the non-reflected raw beam (Figure 4). At this location along the beam line there is also room to accommodate a low efficiency fission counter as a primary beam monitor, and an absorber wheel to facilitate transmission measurements. Also, provision is made for injecting a light or a laser beam into the collimator for accurate sample adjustments.

PRIMARY BEAM

A primary beam of 6-mm diameter at the sample is used for the majority of the experiments. The solid angle subtended at the apparent source by a straight 6-mm collimator is large enough to let neutrons pass with only minor truncation of their angular distribution. Given the width of this distribution and a sample-detector distance of 1 to 2 m, the size of a primary beam in the detector plane depends primarily on the beam divergence. Therefore, it is of great advantage to open up the entrance aperture of the collimator. The resulting convergently collimated beam has a divergence that is increased only insignificantly. The transmittance of the collimator, however, increases dramatically, as shown in Figure 5. For example, at 60-MW reactor power the flux in a 6-mm beam of 5-Å neutrons can be increased from 6×10^5 to 1.7×10^6 $\text{ns}^{-1} \text{cm}^{-2}$ by increasing the diameter of the entrance aperture from 6 mm to 12 mm. The full width of the primary beam at maximal sample-detector distance is thereby increased by 20%, as shown in Figure 6, compromising the first-order resolution and to a lesser extent the order-to-order resolution. At a shorter detector distance, the increase in beam diameter is proportionally less.

Fig. 5. The measured flux at 5 Å is plotted as a function of the collimation geometry. The solid vertical bars represent a series of flux measurements carried out by us. The superimposed thin-lined curves represent the calculated transmittance of the collimator-monochromator combination. They are scaled by eye to the experimental observations. The area of the exit aperture and the length of monochromator providing uniform illumination of the exit aperture are the important terms in this calculation. The flux scale on the right was obtained by averaging flux measurements carried out by the users of the facility over a 9-month period with reactor power at 40 MW. Compiled were data taken with wavelengths between 4.75 and 5.25 Å, a wavelength spread (FWHM) of 8%, and all 6-mm collimations shown in the graph. This flux scale was then expanded by 50% to reflect the recent increase in reactor power to 60 MW. All flux measurements were done with vented collimator and detector drift chamber. The usual helium filling can displace 3 to 4 m of air, resulting in an intensity gain of about 30%.

One aspect of the wavelength distribution in the collimated beam is the wavelength gradient along a horizontal section. This gradient is due to the use of a crystal monochromator. Its extent can be calculated from Bragg's law. Neglecting a small wavelength-dependent term, one obtains in good approximation $\Delta\lambda = 2d\Delta\theta$, where d is the multilayer spacing and $\Delta\theta$ the angle subtended at the monochromator by the exit diaphragm of the collimator. From a measured $\Delta\theta = 9$ min, in agreement with the geometrical value, the full bandwidth of the wavelength gradient in a 6-mm beam scattered

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from a 75-Å multilayer is calculated as 0.38 %, in excellent agreement with the experiment.

The largest contribution to the wavelength spread in the collimated beam arises from the variety of rays passing through a given point in the last collimating aperture. The wavelength spread in such a bunch of neutrons scattered under different angles from different portions of the monochromator is again defined by the above approximation. The smaller value of either the divergence of the apparent source or the angular aperture of the collimator is used for $\Delta\theta$. Neutrons emerging near the edges of the collimator may have angular distributions that are truncated by preceding collimating apertures. If, however, the entrance aperture of the collimator is opened sufficiently, its exit

Fig. 6. Intensity profiles across primary beams of 6-mm diameter at the sample as a function of collimation arrangement and sample-detector distance. The diameter of the collimator entrance aperture was increased from 6 mm (profiles on the left) to 14 mm (profiles on the right). The collimator exit aperture at 136 cm from the entrance had a fixed diameter of 6 mm. The front row of profiles was measured at a sample-detector distance of 95 cm, and the back row at 195 cm. The profiles were calculated from the two-dimensional detector data by integration over the vertical dimension of the beam trace. The profiles are normalized to arbitrary peak intensity to facilitate comparison of their widths. Data points are at intervals of 1 pixel = 1.6 mm.

aperture can be evenly illuminated. Under these conditions the wavelength spread due to the sampling of extended areas on the monochromator becomes independent of radial position in the beam. The convergently collimated 12 mm - 6 mm beam discussed above, monochromatized by a multilayer of 75-Å bilayer spacing, was measured to have a wavelength spread of 0.6 Å full width at any point across its area. From this wavelength spread the effective divergence of the apparent source is calculated as about 14'. This is in good agreement with the measured width of the most intense part of the divergence angle distribution in the apparent source (compare Figure 2).

Convergent collimation, as a drawback, increases the angles of extreme rays. This leads to a broadening of the wavelength distribution, mostly towards longer wavelengths. The resulting "tails" are weak and confined.

The wavelength distribution in the full primary beam is described by superimposing a series of local distributions on the wavelength gradient. In a cylindrical beam the extremes of the gradient are poorly populated, which leads to a reduction of the effective spread.

An experimental procedure is routinely used to protocol wavelength, its spread, and the beam divergence. The (002) reflection of a large Na-β-alumina crystal (d-spacing = 11.3 Å) is measured as a function of scattering angle, 2θ , for each angular setting, ω , of the analyzing crystal. The measured intensity distribution in an ω - 2θ space, as shown by Schoenborn (20), allows reconstruction of wavelength and angular distributions separately. In the example shown in Figure 7, the wavelength distribution was obtained by projecting the reflection onto the 2θ -axis. A wavelength spread (FWHM) of 8% is obtained at an average wavelength of 5.1 Å. In the ω - 2θ space the centerline of this reflection is inclined at an angle of 25°. This is within 1° of an angle of 26°, calculated from the above described chromatic properties of the beam.

The first-order resolution (minimum Q) of the diffraction instrument is calculated from the smallest scattering angles defined by intersecting with the detector plane extreme rays emerging from the collimator. The limited resolution of the detector is taken into account by radially shifting these rays by half a detector resolution element (see below). In Table 3, best first-order resolutions are listed for cylindrical collimators extended to the sample axis and for maximal sample-detector distance. The order-to-order resolution for the 6-mm beam is comparable with the first-order resolution under these conditions, and it is separately listed for the 4-mm beam. The order-to-order resolutions were calculated from measured beam profiles.

Fig. 7. An ω - 2θ intensity diagram of the (002) reflection from Na- β -alumina was used to analyze chromatic and angular properties of the primary beam. Each profile was measured at one ω angle and is the result of an integration over the vertical extent of the reflection. The profiles, when added at constant 2θ , are used to construct the wavelength distribution. This is possible since broadening of the reflection due to extended beam and limited counter resolution is small. The average wavelength is 5.1 Å, its spread FWHM = 8%. The monochromator spacing was 73 Å. The collimator was convergent with apertures of 10 mm and 6 mm. The analyzer-detector distance was 180 cm.

Table 3. First-Order Resolution

Wavelength, Å	Collimation:	First-order resolution (Å)		Order-to-order resolution (Å)
		6 mm	4 mm	4 mm
4		310	430	600
6		460	650	900
8		620	860	1200
10		770	1070	1500

Fig. 8. Schematic view of the H9B spectrometer.

SPECTROMETER

The H9B spectrometer (Figure 8) has a vertical sample axis (ω -axis). Oriented specimens are mounted on a goniometer head or supported from the 40-cm sample table 35 cm below the beam centerline. This table is sturdy enough to carry cryostats or magnetic equipment. For solution scattering experiments a rotary sample changer with provisions for 8 samples, or a linear changer with 11 positions, is placed on this axis. The sample axis is aligned into the beam by means of a manually operated lateral motion that comprises the forward support of the spectrometer.

The two-dimensional detector and its electronics ride on a carriage that moves on a radial beam. The maximal sample-detector distance is 195 cm and the minimal, 50 cm. The drift space between sample and detector is enclosed by segments of a shielded and helium-flushed snout. The detector can be raised and lowered along a vertical axis, and it can be translated manually along a horizontal slide. The radial beam is connected coaxially to the sample axis. Its other end is suspended by slip joints from a tangential beam. The radial beam with the detector pivots around the sample axis to cover a scattering angle of 45° . The volume element through which the detector can be moved under computer control is shown in Figure 9.

Fig. 9. Volume element within which the center of the two-dimensional detector can be positioned under computer control. The vertical sample axis is on the right. The beam centerline is shown as a horizontal axis. The heavy vertical bar centered on the beam line indicates the height of the 17x17-cm two-dimensional detector. The tangential support beam of the spectrometer can be wheeled easily to a new position. The volume element, therefore, can be centered at a scattering angle other than the current 5° .

DETECTOR

The two-dimensional ^3He -filled proportional detector has a sensitive area of 17 by 17 cm, which is sampled by 128 by 128 picture elements. Its construction and electronic design have been described (21,22). The discussion here is restricted to an eval-

Fig. 10. Measured x (a) and y coordinates (b) as a function of detector displacement. Displacement along an x -axis was replaced by a scan along the 2θ -angle (compare Figure 9) in order to automate the raster scanning protocol. The full lines represent the geometric displacement of the detector (target values), and the points (+) are the measured coordinates. The probing beam had a diameter of about 5 pixels.

uation of its performance. Since the detector was put into service in 1974, it has been refilled once. Deteriorating counting gas results in decreased γ -radiation discrimination, and at present the background counting rate has increased to 6 cpm cm^{-2} .

The linearity in position decoding as a function of actual displacement of a probing beam was monitored by moving the detector between the nodes of a rectangular grid (raster scanning). The raster of measured coordinates was then fitted with the accurately known displacement grid by varying the x and y size of the average picture element and by floating the coordinate origin (Figure 10). The average picture element calculated from the fit is a rectangular unit of about 1.6-mm side lengths (i.e., $\Delta x = 1.595$, $\Delta y = 1.609$ mm/pixel). The error accumulated across the detector by assuming a square picture element of average side length is about 1 picture element. This distortion is often too small to affect solution scattering data.

The integral nonlinearity is 0.8 pixel for the x coordinate and 1.5 pixel for the y. Comparison of linearity tests conducted at intervals over a year with various electronic adjustments showed that the systematic error in coordinate measurements remained invariant (Figure 11). The systematic error in the y coordinate, within 0.1 pixel, is a function of the y coordinate alone, and the systematic error in the x coordinate is a function of both coordinates. Given the correlations of systematic error and coordinate, it is possible to develop an efficient correction algorithm for very accurate data processing. The magnitude of the error is, however, small compared with the 4-mm spatial resolution of the detector.

The differential nonlinearity of the detector was measured by continuously sweeping its sensitive area with a very wide attenuated beam. A typical result is shown in Figure 12. Areas of higher counting probability differ by as much as 15% from areas of lower counting probability. The average intensity in successive columns oscillates by 2%. Comparison of counting probability measurements taken at intervals during a year showed that the overall patterns were essentially unchanged, while drifts were recorded within areas of comparable sensitivity. Comparison with similar data recorded since the last renewal of the counting gas indicated a gradual deterioration from an originally almost uniform counting efficiency. The uneven sensitivity profoundly distorts data taken at any counting rate. A matrix of correction factors was therefore constructed by locally averaging measured counting probabilities. When this was applied to the diffraction of water (23), the expected flat and featureless intensity distribution was obtained, and integration errors were within the limits of the statistical error. The correction matrix is readily available during experimentation, and it is routinely used in data reduction.

Fig. 11. The systematic error in the x coordinate (a) and the y coordinate (b) is plotted as a function of the coordinate. The data points are calculated as the difference of the average of the measured coordinates along a row (a) or line (b) of raster points and the geometrical target value. Data from 4 raster scans taken within a year are compiled in these correlations. The vertical bars through selected points in (a) represent the extent of the systematic variation of the x coordinate as a function of the y coordinate. This variation can be correlated with the y coordinate with an accuracy comparable with the graph shown.

Fig. 12. The intensity recorded in the 128 by 128 pixels of the uniformly illuminated detector is plotted on a quasi isometric grid. Each picture element accumulated about 1000 counts during continuous sweeps of the detector through an attenuated beam of 20-pixels diameter. An accurate counting-probability correction matrix was obtained from these data by averaging three next lines.

Fig. 13. The spatial resolution of the detector is measured by its response to a pair of 1-mm beams at a horizontal center-to-center distance of 3, 4, 5, and 6 mm. The solid dots represent the test beams drawn to scale. The x-axis of each intensity profile is indexed in units of picture elements.

The spatial resolution of the detector was evaluated with two 1-mm test beams at various distances from each other. The test beams were filtered from a wide primary beam through holes in a cadmium mask mounted close to the detector. A spatial resolution of about 4 mm, as shown in Figure 13, is representative for measurements carried out at various points across the sensitive area. The spatial resolution along the vertical dimension is marginally better. With the spatial resolution used as a measure of the independent resolution element, the number of independent picture elements on the detector was calculated as 42 by 42. Figure 13 also demonstrates that a very narrow beam gives rise to signals in an average of 4 next picture elements. High spatial resolution is a requirement antagonistic to high energy resolution (22). In fact, during monitoring of this slowly aging detector, the spatial resolution was seen to be the most vulnerable of the parameters discussed above to changes in gas multiplication.

SPECTROMETER AND EXPERIMENTAL CONTROL

The motions to position detector and sample are computer controlled, and they can also be "manually" adjusted from a remote control. Absolute encoders read the actual positions of moving parts. They provide a unique response for a given position independent of power or computer failure. The LSI-11 microprocessors of the spectrometer control unit manage all motor-related functions (24). In particular, new configurations of the spectrometer are assumed by driving motions iteratively at variable speeds until the requested encoder reading is achieved. This combination makes the instrument accurate and reliable. The spectrometer control unit is serially linked to the experiment control computer, a PDP 11-34 (Figure 14).

The experimenter interacts with the spectrometer through a keyboard of the experiment control computer. At his disposition are program sequences to facilitate the setup of new experiments. A library can be built with information to conduct experiments automatically. Such scans may involve all motions of the spectrometer, and a series of scans can be queued for sequential execution.

Events detected with the two-dimensional detector are accumulated into an external memory array. Upon completion of data accumulation, the data are stored on magnetic tape and simultaneously added into a storage area on disk that is unique either to the sample under investigation or to a step in a repeatedly executed scan.

A graphics terminal is available for on-line monitoring and preliminary analysis. Data stored in external memory or on disk

Fig. 14. Components of spectrometer and experiment control. Three independent systems are shown in the upper half of the flow chart. (i) The motor control unit updates the spectrometer configuration in response to requests from the experiment control computer. It continuously displays status and position of all motors on an auxiliary screen. (ii) The position-sensitive detector system feeds position decoded information into the multiport external memory. (iii) The counter control starts or stops the flow of information into external memory. At the same time, it updates single-channel counters containing the total detector and monitor counts. The experiment control computer (PDP 11-34) is shown in the lower half. The data pathway is accentuated. About 300 detector images can be stored on magnetic tape in integer format. The disk has a capacity of 76 images stored in floating-point format to provide accuracy for array manipulation. The keyboard is used to manage data collection and to receive control output during scanning. The graphics terminal, a Tektronix 4014, is used to obtain on-line displays and to manipulate disk stored data. External memory array 2 (A2) is set aside as a data base for the display routines.

are accessible for display. An array manipulator is provided to manipulate, combine, and correct various disk-stored data sets both for display and for permanent record. The display routines include elementary statistical functions, isometric and profile displays, and various mapping techniques. Data averaging, fast incrementing, and interactive readouts are supported. A further group of routines is used to integrate data in various ways and to display integrated intensities including Guinier plotting and related techniques.

To maintain and monitor the spectrometer a group of smart routines was developed that measure wavelength, counting probability, positional linearity, and resolution with minimal operator involvement.

ACKNOWLEDGMENTS

We wish to thank Ed Caruso and Jack Guthy for their expert professional help in operating the facility.

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DISCUSSION

ZACCAI: What advantages are there in moving the detector vertically?

SCHNEIDER: The vertical displacement of the detector is useful for measurements of the meridional diffraction on oriented specimens. Together with the lateral displacement this adjustment makes it possible to center the primary beam at any point on the detector and therefore to extend the range of simultaneously recorded scatter. In addition it is an essential movement for testing position decoding and spatial resolution of the two-dimensional detector.

ZACCAI: What is the short-term and long-term stability of the detector? If you measured the same sample repeatedly over a day what variation would you expect?

SCHNEIDER: From measurements of a 2-mm water sample over a 24-hr period we obtained a maximal variation of the integrated counts of about twice the statistical error. The average number of counts in two 24-hr periods with a 6-day interval was the same within the statistical error ($\sim 0.2\%$). In similar measurements on the very intense scatter from organic solids, both the short-term fluctuation (20 min) and the long-term fluctuation (6 days) were within the statistical error. We therefore consider the stability of the detector and the reproducibility of intensity measurements as satisfactory. The stability of the detector in position decoding was tested by repeated measurements of the center of an

attenuated primary beam over a 24-hr period. The maximal drifts were 0.2 pixel (= 0.3 mm). This value is smaller than the systematic error in position decoding (compare Figure 13).

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