The Neutron Small-Angle Camera D11 at the High-Flux Reactor, Grenoble

BY K. IBEL

Institut Laue-Langevin, 156X Centre de Tri, 38042 Grenoble Cédex, France

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The neutron small-angle scattering system at the high-flux reactor in Grenoble consists of three major parts: the supply of cold neutrons via bent neutron guides; the small-angle camera D11; and the data handling facilities. The camera D11 has an overall length of 80 m. The effective length of the camera is variable. The full length of the collimator before the fixed sample position can be reduced by movable neutron guides; the secondary flight path of 40 m full length contains detector sites in various positions. Thus, a large range of momentum transfers can be used with the same relative resolution. Scattering angles between $5 \times 10^{-4}$ and 0.5 rad and neutron wavelengths from 0.2 to 2.0 nm are available. A large-area position-sensitive detector is used which allows simultaneous recording of intensities scattered at different angles; it is a multiwire proportional chamber. 3808 elements of 1 cm$^2$ are arranged in a two-dimensional matrix.

1. Introduction

Neutron small-angle scattering is a specialized crystallographic technique for investigating long-range fluctuations (Schmatz, Springer, Schelten & Ibel, 1974). It consists of the analysis of the intensity distribution of long wavelength neutrons scattered within small angles.

Examples of such structures, which have been investigated with the D11 camera, are:

1. Structure of biopolymers (Baldwin, Boseley, Bradbury & Ibel, 1975; Baldwin, Bradbury, Carpenter, Hjelm, Hancock & Ibel, 1976; Miller, White & Ibel, 1974; Bram, Butler-Browne, Bradbury, Baldwin, Reiss & Ibel, 1974; Bram, Baudy, Butler-Browne & Ibel, 1974; Bram, Butler-Browne, Baudy & Ibel, 1975; Chabre, Saibil & Worcester, 1976; Doyle, Haas, Hulmes, Ibel, Jenkin, Miller, Timmins & White, 1976).
2. Precipitation in alloys and in glasses (Roth & Zarzycki, 1974; Roth & Raynal, 1974; Raynal & Roth, 1975).
5. Long-range fluctuations at the metal–non-metal transition in Li in ND$_4$ (Chieux, 1974).

The neutron small-angle camera D11 (Fig. 1) at the high-flux reactor in Grenoble is the realization of a theoretical concept which has been published previously (Ibel, Schmatz & Springer, 1971). A similar instrument of 40 m length has been built at the FRJ-2 reactor in Jülich (Schmatz & Schelten, 1971; Schelten, 1972). This paper, based on an contribution to the Neutron Diffraction Conference in Petten (Ibel, 1975),

Table 1. The most important parameters of the neutron small-angle camera D11

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Incident wavelength</td>
<td>$0.2 &lt; \lambda &lt; 2.0$ nm</td>
</tr>
<tr>
<td>Monochromator</td>
<td>Helical slot selectors</td>
</tr>
<tr>
<td>Wavelength resolution</td>
<td>$\Delta \lambda / \lambda = 45%$, FWHM</td>
</tr>
<tr>
<td>Maximum flux at specimen</td>
<td>$10^3 - 10^6$ s$^{-1}$ cm$^{-2}$</td>
</tr>
<tr>
<td>Typical beam size at specimen</td>
<td>25 mm high; 15 mm wide</td>
</tr>
<tr>
<td>$Q$ range, $Q = 4\pi \sin (\theta/2)/\lambda$ (full scattering angle)</td>
<td>$10^{-3} &lt; Q &lt; 10^{-1}$ nm$^{-1}$</td>
</tr>
<tr>
<td>Angular resolution</td>
<td>$\pm 0.0, \pm 8%$ full width at a mean $Q$ value independent of detector position</td>
</tr>
<tr>
<td>Detector</td>
<td>64 x 64 channels</td>
</tr>
<tr>
<td>Two dimensional set of</td>
<td>BF$_3$ containing multi-electrode chamber with gas amplification</td>
</tr>
<tr>
<td>Cross section of one element</td>
<td>$10 \times 10$ mm</td>
</tr>
<tr>
<td>Maximum counting rate</td>
<td>$10^4$ c.p.s.</td>
</tr>
<tr>
<td>Installation of detector possible at 40, 20, 10, 5 and 2 m distance from the sample, corresponding to collimation length</td>
<td></td>
</tr>
<tr>
<td>Further sample positions</td>
<td>170 and 100 cm in front of the detector</td>
</tr>
</tbody>
</table>
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deals with the properties of the camera as a part of the overall system. The most important parameters of the instrument are summarized in Table 1. A few examples for typical applications of the instrument have been selected (§ 3) which show the capacity and versatility of the instrument.

2. Neutron small-angle scattering system at the HFR

The overall system may be divided into three parts: (i) the supply of cold neutrons, (ii) the instrument, and (iii) the handling of data.

2.1. Supply of cold neutrons

Neutrons released in the fuel element are thermalized in heavy water. They are further cooled down by liquid deuterium, contained in a spherical vessel of $22 \times 10^{-3}$ m$^3$ volume. The diameter of the vessel (about 38 cm) permits a height of 20 cm for the neutron guides delivering the neutrons to the external instruments. The width of the guides is 3 cm. The first 28 m of the guides up to the wall of the reactor hall are bent with a radius of 2700 m to eliminate $\gamma$-radiation and fast neutrons. The instrument has been installed far from the core at good space and overall background conditions. The brightness of the source is preserved within a solid angle which is determined by the limiting angle of total reflexion inside the tubes. Loss of useful neutrons (Fig. 2) may be attributed to a guideless section at the entrance and to aluminum windows (Ageron, 1974). The limits imposed on the system by the source and the neutron guides are essentially the flux of cold neutrons in the cold source and the cross section of the guides. The width of 3 cm of the guides determines all other dimensions of the instrument, including the size of the unit cell of the multidetector.

Fig. 1. Perspective of the small-angle camera D11, full length 80 m, in the external hall of the high-flux reactor (HFR). The camera is connected to the cold source near the core of the reactor via neutron guides. The primary parts comprise mechanical monochromator (M) and collimator system (C). The sample-containing vessel (S) is fixed in its position between the primary and secondary flight paths. The detector (D) containing tube is partly housed in an appendix to the hall. The transfer of the detector to the different positions 2; 5; 10; 20 and 40 m is done with a movable crane.

Fig. 2. Neutron flux in neutrons per nm per s, at the exit of the guide H15 as calculated and measured.
The total height of 20 cm is shared by three spectrometers (Fig. 3), the small angle camera using the lower part (5 cm height).

2.2. Camera D11

The different parts of the camera (Fig. 4) are: the primary flight path for selecting an appropriate wavelength and collimation; the sample and its environment; the secondary flight path for analysing the angular distribution of the scattered neutrons, including on-line data acquisition and visualization. A part of the mechanical arrangement has been described in an earlier publication (Degenkolbe & Greiss, 1973).

2.2.1. Pumping system

The neutron flight paths (total volume about 35 m³) are evacuated to below 10⁻¹ torr in less than 2 h; the velocity selector at the entrance of the camera is running in air and the detector is mounted in air in an insert, separated by a partition wall from the evacuated secondary flight path.

2.2.2. Primary flight path

2.2.2.1. Unmonochromatized 'white' beam. The unmonochromatized ('white') incoming beam could be used to observe the intensity of scattered neutrons extrapolated to zero momentum transfer. Fig. 5 shows its wavelength distribution measured by time-of-flight techniques within a small fixed solid angle around the beam axis. A maximum has been observed at about 0.33 nm. However, regarding the flexibility in camera length, the highest intensity of scattered neutrons at a given resolution in momentum transfer is obtained

![Fig. 5. Wavelength distribution in counts per channel per unit time of the incoming unmonochromated collimated beam, as measured by time-of-flight. Not corrected for efficiency of detector. The horizontal bar indicates the time-of-flight resolution. Collimation length / = 5 m; size of sample diaphragm: Δxₜ₄, Δyₜ₄ = 1 mm². The step at 0.37 nm is probably due to the onset of a Bragg reflection in the aluminum walls of the entrance.](image)

![Fig. 3. Top view of the three spectrometers at the guide H15. The diffuse scattering spectrometer (D7) uses the middle part of the beam. The upper part hits the Doppler drive of the inelastic backscattering spectrometer (IN 10), and the lower part of 5 cm height is directly fed into the monochromator (M) of the small-angle camera D11. W, the wall of the reactor hall; G20, G10, G5, G3, the movable neutron guide sections; S, sample vessels; D2, D5, D10, D20, D40, detector sites.](image)

![Fig. 4. The main parts of the neutron small-angle camera D11 are: the helical velocity selector (M); the movable neutron guides (G20, G10; the guides G5 and G3 in the standby position). The collimator / between the exit of the guides and the sample diaphragm; the sample (S); the secondary flight path L; the multidetector D, including on-line computer. The centre of the detector is fixed in the direction of the primary beam. The highest relative angular resolution (about 4°) is the ratio of the diameter (d₀ = 1 cm) of an element at the edge of the detector to its distance from the primary beam, independent from the sample detector distance L. Δxₛ, Δyₛ size of the apparent source, normally the exit of the neutron guides with Δxₛ = 30 and Δyₛ = 50 mm; Δxₛ, Δyₛ size of sample diaphragm, normally with Δxₛ = 15 and Δyₛ = 25 mm.](image)
between 0.6 and 1.2 nm. The intensity $\Delta J_0$ which hits the sample within the cross section $d_2^2 = \Delta x_s \Delta y_s$ is given by (Maier-Leibnitz, 1966; Fig. 6)

$$\Delta J_0 = f_0(\varphi_{th}/4\pi) \exp \left( -E/k_B T \right) (E/k_B T)^2 (2\Delta \lambda/\lambda) d_2^2 d_2^2 / l^2$$

assuming that the neutrons are distributed according to a Maxwellian with a temperature $T$. The total thermal flux is called $\varphi_{th}$ and $d_2^2 = \Delta x_E \Delta y_E$ is the cross section of the source, at the distance $l$ from the sample. $f_0$ is an attenuation factor. The number of counts within the detector unit cell is then

$$\Delta J_D = f_1 \Delta J_0 d_2^2 / L^2$$

where $d_2^2$ is the cross section of the detector unit cell, and $L$ the sample–detector distance. The scattering probability $w$ is

$$w = D_S n_S (d\sigma/d\Omega) \exp \left( -D_S n_S \sigma_t \right)$$

where $D_S$ is the sample thickness and $n_S$ the density of the scattering particles. $\sigma_t$ is the total cross section of the sample material, including absorption, incoherent and coherent scattering. $f_1$ takes into account the attenuation of scattered neutrons in the secondary flight path and the response of the detector.

If we assume that the total cross section $\sigma_t$ and the attenuation factors $f_0$ and $f_1$ do not depend on the wavelength, we find, at constant sample thickness and constant particle density,

$$\Delta J_D = \text{const.} \Delta J_0 (d\sigma/d\Omega) d_2^2 / L^2.$$

We choose a symmetric arrangement of primary and secondary part, $l = L$, and the sizes of the source, the sample and the detector unit cell such that $d_2 = 2d_S = d_P$. We express the resolution in momentum transfer by

$$r = \text{const.} (d_P / L \lambda)$$

and find at large wavelengths, where the Boltzmann factor in the Maxwellian becomes negligible:

$$\Delta J_D = \text{const.} (1/\lambda^4) (2\Delta \lambda/\lambda) d_2^2 (d\sigma/d\Omega) r^2 \lambda^4$$,

i.e. the scattered intensity is independent of the wavelength at constant relative wavelength resolution, constant beam cross section as well as constant resolu-

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Fig. 6. Intensity, in counts per second, of the primary beam as a function of resolution in momentum transfer:

$$2\pi \Delta h / \lambda = (2\pi / \lambda)(\Delta x_E \Delta y_E + \Delta x_S \Delta y_S)^{1/2} / l$$

for three different wavelengths. The wavelength interval $\Delta \lambda / \lambda$ was at 9% FWHM. Above the points $A$ and below the points $B$ we have chosen a fixed size of the apparent source, i.e. the neutron guide cross section $\Delta x_S \Delta y_S = 15$ cm$^2$. A fixed size of the sample area $\Delta x_E \Delta y_E = (15/4)$ cm$^2$ has been taken; the resolution in momentum transfer depends in this domain on the collimation length $l$, the distance between the source and the sample. Hence, between $A$ and $B$, a quadratic law holds for the intensity resolution relation due to the quadratic increase in solid angle with diminishing collimation length. Above $B$, the intensity is limited by the maximum angle of total reflexion within the neutron guides. Below the points $A$, the full length of collimation of 40 m has been reached and the beam size of the source as well as the sample area has to be diminished for improvement of resolution; hence the intensity drops with the fourth power of the resolution. The curves have been normalized to an experimental value taken for $\lambda = 10$ nm. No correction has been made for the detector response.

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Fig. 7. Position of a velocity selector in the beam. The middle part of the main beam of 20 cm height had been reflected into the diffuse scattering instrument ($D7$) via a graphite monochromator mounted on a turntable behind the leadshield ($LSh$). The upper part is guided to the inelastic backscattering spectrometer ($IN10$). The drum ($Dr$) and motor ($Mo$) of the selector are mounted on a heavy concrete block ($CBI$). The beam can be attenuated by a boral plate with a hole of 2 mm diameter ($Att$). A neutron monitor ($Mort$) and a diaphragm ($Dia$) are attached to the entrance of the collimating system ($Col$). $gg$ is the guideless cap, creating a half-shade of neutrons of higher angle of incidence onto the totally reflecting inner surfaces of the neutron guides. All distances in mm.
tion $r$. This means, we may choose any arrangement of camera length and wavelength, as long as we restrict ourselves to the long-wavelength tail of the Maxwellian. However, the optimization of the wavelength, hence of the camera length, has to be done for each sample separately, taking into account the wavelength dependence of its total cross section. Furthermore the factor for attenuation before the sample increases with wavelength, and large solid angles suffer from losses due to imperfect reflections at the neutron guide surfaces and from half-shades due to guideless sections. This leads to the optimized conditions at wavelengths between $\sim 0.6$ and $\sim 1.2$ nm, as mentioned above.

2.2.2.2. Monochromatization. The first device intrinsically belonging to the instrument is a mechanical velocity selector. Two selectors with 45°, FWHM (Adele) and 9°, FWHM (Brunhilde) are available.

The two selectors are easily exchangeable. They are mounted on heavy concrete blocks. The blocks may be displaced using air pads. Fig. 7 shows the mechanical arrangement of the selector Brunhilde at the beam. Both selectors are constructed using the same principle: they are drums with their axes parallel to the beam. Helical slots are engraved at the periphery; these slots are fixed in the frame of reference of the neutrons of the required velocity which allows them to pass through the drum within the same time needed, at a given rotatory speed of the drum, to bring the exit of a reference slot onto the beam axis.

The drums are connected with synchronous motors, driven by an alternating current of variable frequency. The deviations in the rotatory speed from a preselected value are about 1 part in 3000, even during extended periods. The power supply unit transforms a variable direct voltage statically into the alternating current.

The length of the drum of the low-resolution selector Adele is 400 mm, with 72 equidistant slots; the other geometrical dimensions are indicated in Fig. 8. The wavelength distributions obtained at different rotatory speeds are shown in Fig. 9. There is no simple relationship between the most probable wavelength and the rotatory speed; furthermore, the wavelength distribution is quite sensitive to small deviations in the alignment of the drum axis parallel to the beam axis.

The drum length of Brunhilde is 1000 mm with 115 equidistant slots; the geometry of the slots is shown in Fig. 10. Due to the higher resolution, a simple formula holds for the relationship between rotatory speed $\Omega$ and the $a priori$ most probable wavelength:

$$\lambda \Omega = 1876 \text{ nm(r.p.m.)}$$

using the neutron wavelength–velocity ($v$) relationship

$$\lambda v = 395.7 \text{ nm(m/s)}$$

and the pitch $P = 12.560 \text{ m/revolution}$ of a helix in the drum. The experimental value of the wavelength–rotatory speed relationship (Fig. 11) is $\lambda \Omega = 1845 \pm 20 \text{ nm(r.p.m.)}$. The discrepancy with the theoretical value is due to the Maxwellian fall-off in wavelength. The magnified curve shows the excellent cut-off of the wings of the distribution, which is important for applications in metal physics, where a contamination...
of the beam by short-wavelength neutrons has to be avoided.

The use of the selector is limited by the maximum speed of rotation; wavelengths down to 0.41 nm are available.

Fig. 10. The medium-resolution velocity selector Brunhilde. Projections of the cross sections of the entrance and the exit of one of the 115 helical slots on a plane perpendicular to the beam axis. $R=170$ mm, distance of the centres of the slots from the drum axis. Drum length: 1000 mm. The distances are indicated in mm. The transmission is about 75%.

Fig. 11. Wavelength distribution in counts per time-of-flight channel of Brunhilde for two different rotatory speeds. The ten times magnified curve shows the sharp cut-off of the monochromatized beam towards lower and higher wavelengths.

Fig. 12. Neutron guides and collimation. The use of movable guides in the collimator makes it possible to put the exit of the guides, i.e. the apparent source of the beam, at different distances from the sample (S). Above: neutron guides (G) in the beam; high intensity, large divergence. Below: neutron guides removed; weak intensity, limited divergence. D, diaphragm.

The reproducibility in the mechanical alignment of the selector is better than 1% giving rise to the same relative uncertainty in wavelength. The broadening of Debye-Scherrer rings, which is due to the wavelength spread, leads with Brunhilde to rings with a full width of at least 30 cm x 18% ≈ 5.4 cm at the edge of the detector.

2.2.2.3. Incident flux monitor. The full cross section of the monochromatized primary beam is monitored by a fission chamber of low response; the monitor is attached to the entrance window of the collimation tube (Fig. 7).

2.2.2.4. Collimation. The flexibility in the choice of an appropriate solid angle is achieved by movable neutron guides of 30 x 50 mm inner cross section. The lengths of the sections are: 20, 10, 5, and 3 m. The guides are made from borated glass with highly polished inner surfaces, which are not nickel coated. The deviation of the orientation of the inner surfaces from an ideal flat plane is less than $2 \times 10^{-5}$ rad. Between the sections, ports are available for mounting supplementary diaphragms.

The exit of the guides is the apparent source of neutrons; the system of movable guides makes it possible to put this apparent source at different distances from the sample (Fig. 12).

In practice a decrease in intensity of neutrons hitting the sample at larger angles has been observed (Fig. 13). This is mostly due to the half-shade arising from the guideless gap at the monochromator site.

A thorough shielding of the walls surrounding the incoming beam within the collimator is essential for
suppressing the background. Fig. 14 shows a perspective view of the neutron absorbing wings within the tube, with the neutron guide in the standby position.

2.2.3. Sample
The essential problem concerning the sample is its cross-sectional area. With regard to the size of the source and optimizing the resolution versus intensity, sample cross sections with dimensions of half the dimensions of the source would be the best choice, for D11 samples of 15 mm width and 25 mm height. With regard to the size of one detector element of 10 × 10 mm, sample cross sections of less than 5 × 5 mm would not lead to an appreciable improvement in the angular resolution.

Fig. 15 shows the standard sample environment. The sample vessels are separated by valves from the collimation tube and the detector-containing tube.

Fig. 16. Cross section and top view of the heads of the fingers A and B. Quartz windows of 2 mm thickness allow the entrance of the primary beam and the exit of the scattered beam. All inner and outer surfaces are covered with Cd foils. Distances in mm.
They allow the sample to be put into the beam without any separating windows. The small vessel can be removed and replaced by some auxiliary equipment, such as cryostats, magnets, stop-flow equipment, furnaces, etc. An auxiliary accessory for the sample vessel is a computer-controlled goniometer head.

The fingers (Figs. 15 and 16) allow the samples to be put rapidly into the beam. The tips of the fingers A and B are equipped with quartz windows of 26 mm useful diameter; that of the nearby finger C is an aluminum cylinder which allows the scattering up to angles of 30° to be observed.

2.2.4. Secondary flight path

This part of the instrument comprises: the detector and the attached electronics including the data acquisition system.

The detector has been placed outside the vacuum in a container (Fig. 17), separated from the evacuated flight path by a partition wall of aluminum of 6 mm thickness. For changing the detector position, the whole detector-containing tube of 40 m length (volume: 32 m³) has to be filled with dry gas, e.g. nitrogen, and then pumped out again.

The primary beam is suppressed by a cadmium beam catcher of 1 mm thickness. In order to measure the position, size and intensity of the primary beam the opaque catcher can be replaced by a thin foil of cadmium. A thickness of 0.3 mm is sufficiently transmissive for monitoring neutrons of 0.6 nm wavelength and sufficiently opaque even with a short camera length (all guides on the instrument axis). The beam stop is exchangeable without breaking the vacuum by withdrawing it into the air lock. Vertical adjustment of the position of the primary beam stop is possible by pulling the handle, horizontal adjustment by displacing the cover of the air lock.

The technological methods used for the construction of a similar detector operating by direct charge collection, which was used up to November 1974, are described elsewhere (Allemand, Bourdel, Roudault, Convert, Ibel, Jacobé, Cotton & Farnoux, 1975). Since this date, a detector operating by gas amplification has been in use. It consists of \(64 \times 64 = 4096\) elements. The four corners of the square comprising 72 elements are not sensitive; thus \(3808\) elements each with a cross section of \(10 \times 10\) mm are available, with a maximum distance of 72 cm along the diagonal. This was the maximum area which could be realized in practice at the time of building the detector.

The essential reason for designing the instrument was for analysis at very small angles. Given a certain size of sample (which should make the best use of a source of given size), high resolution at very small angles can only be achieved by a very long instrument. For the D11 system, the maximum distance of the detector from the sample is 40 m.

In order to simplify the mechanical lay-out, an arrangement has been chosen where the centre of the detector coincides with the axis of the instrument.

The number of elements was determined by the core size of the data acquisition system at the time of the design of the instrument. A unique core size of 4 K was available for storing the data into 12-bit addresses. With the development of large core memories it is possible to design detectors with a higher number of independent elements. Given a constant element size this would lead to larger sensitive areas.

The sensitive volume of the detector contains \(^{10}\text{BF}_3\) gas at a pressure of 900 torr. The thickness of the sensitive volume is 2 cm, which would lead to an efficiency for 0.6 nm neutrons of 60%. In practice, 50% has been achieved.

The sensitive volume is enclosed in glass plates, each bearing 64 band-like electrodes. At one of the glass plates, the electrodes are arranged horizontally, at the other vertically. In the middle plane of the sensitive volume, proportional wires are spanned out. The addresses are created by observing the coincidence of pulses at the vertical and horizontal electrodes. The pulses on the wires serve to open the gate of the coincidence unit.

An increase in the thickness of the sensitive volume
would not proportionally increase the sensitivity of the
detector. Hence the best use of a larger sensitive volume
would be to increase the total area of the detector.

The response across a single element is a continuous
function of the distance from its centre (Fig. 18). Thus,
the position of a highly collimated beam can be deter-
mined within an accuracy of around 1 mm at the limit
of the element.

Small-angle scattering at the transparent aluminum
partition walls immediately in front of the detector
leads to the spreading out of a highly collimated tiny
beam. Centred in the midst of one detector element,
this leads to a contamination of the neighbouring cells.
The total number of counts in the four nearest neigh-
bour cells is about 3\% of that in the reference cell, with
neutrons of 6 Å wavelength.

The detector response turned out to be stable. The
difference in efficiency of different cells can be corrected
for with high accuracy. With measuring times of about
12 h, peaks which are 1\% above the incoherent back-
ground can be observed. With radial symmetric
distributions, signal-to-noise ratios of $2 \times 10^{-3}$ are
measurable.

The noise exhibited by detector and electronics as
measured during the reactor shutdown with applied
high voltage is around 1 count/h cm$^2$. It is randomly
distributed over the whole area of the detector. The
noise due to neutrons from sources outside the
instrument is about ten times higher. This neutron
background determines the limit for the investigation
of very low scattering samples.

2.2.5. Electronics

The main parts of the electronics are the units for
pulse forming and address creation (multidetector
electronics), and the electronic modules for the transfer
of pulses to the on-line computer, which are standard-
ized corresponding to CAMAC specifications (Fig.
19).

The highest counting rates are limited by the speed
of address creation in the logical unit attached to the
detector. Counting rates exceeding about 400 counts/s
per single element or 2000 counts/s per single electrode
lead to dead time losses of around 20\%. The upper
limit in the counting rate of the whole detector is
50 000 counts/s. By too high count rates faulty
addresses are created at the addresses (0;0), (0;1),
(1;0), (1;1) and at the addresses 0 and 1 of the corre-
sponding single electrodes (Fig. 25).

Each of the 128 electrodes is connected with a
preamplifier, an amplifier and a discriminator, near the
multidetector. This pulse-forming and analog-to-
digital converting electronics is followed by the en-
coder, creating 12-bit words. They consist of 6 bits for
the horizontal location and 6 bits for the vertical location of an event.

A time-of-flight unit is incorporated in the electronics which allows for the measurements of the time-of-flight spectrum of the incident neutrons. It supplies the time information of an event with respect to the time of opening of a chopper. The number of bits for the time-of-flight analysis can be chosen between 3 and 7 bits. Inelastic measurements may be performed using a single or double chopper system (Ibel & Seeger, 1976).

A regrouping unit adds the location and time bit pattern. It makes it possible to choose a certain number of horizontal location bits, vertical bits and time bits. The maximum number of bits is limited to 12. The choice can normally be made to suppress the least significant bits of the 6 horizontal location bits, the 6 vertical location bits or the 7 time information bits independently of the others. Data can be accepted or rejected by an adjustable window.

The 12 bits of the data words are derandomized by a CAMAC input module, which incorporates a 16-word 'last in first out' memory stack. This input module transmits the statistically arriving event to the computer via 50 000 word/s modem.

The monitoring system consists of some CAMAC scalers for time and beam monitoring. The scalers are manually and computer controlled. The data collection is hardware-controlled by a gate signal from the monitorscaler (Master).

2.2.6. On-line computer

The main functions of the Telefunken TR 86 on-line computer and the Nicole system are threefold. (1) The storage of the rough data on magnetic tape for transfer to the PDP 10 data handling system. (2) Visualization of the intensities on a display unit for supervision of the data acquisition. (3) The automatic control of the instrument.

The display enables one to see, even while the experiment is running, the number of counts registered in each channel of the multidetector. One can select either the whole area of the $64 \times 64$ channels or a rectangular part of the whole area, or a single $x$ line or a single $y$ column.

Fig. 20 shows an example of the information obtained at the display. Each point corresponds to one detection element; the number of counts within one channel is visualized by the distance of the points from the $xy$ plane. The ring in the pattern is due to the interparticle effect of low density lipoproteins, exhibiting a spherical shape, in a high-concentration gel (Stuhrmann, Tardieu, Mateu, Sardet, Luzzati, Aggerbeck & Scanu, 1975).

It is also possible to visualize all channels containing a number of counts in the range between $2^{n-1}$ and $2^n (19 > n > 4)$. Fig. 21 presents in this way the diffraction pattern obtained with fibres of chromatin. The regular arrangement of globular protein subunits forming beads on a DNA string gives rise to meridional reflexions above and below an equatorial line (Baldwin, Boseley, Bradbury & Ibel, 1975).

2.3. Data reduction

The data reduction is most conveniently done off-line on a big computer, the task of the on-line computer being restricted to a convenient supervision of data acquisition and the storage of the data in a form which simplifies the transfer to the big computer.

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Fig. 20. Perspective of a pattern on the visualization unit. The primary beam has been suppressed by a rectangular beam catcher; its shadow is clearly seen in the centre. Deviations from a smooth arrangement of the points reflect differences in the response of different electrodes, which might be corrected for by normalization with the isotropic incoherent scattering of water or of a vanadium single crystal. The low-resolution selector Adele has been used (Stuhrmann, Tardieu, Mateu, Sardet, Luzzati, Aggerbeck & Scanu, 1975).

Fig. 21. Map of a diffraction pattern obtained with chromatin fibres. Elements containing between $2^{10}$ and $2^{11}$ counts are visualized. The layer lines above and below the middle equatorial line $(E - E)$ are due to the subunit distance of 11 nm of the bead-like histone particles on a DNA string (oriented in the direction $S - S$).
Fig. 22. Example of a continuous scattering curve of low resolution. The scattering curve of fibrinogen (concentration 2% w/w) in heavy water was obtained at three different camera settings at sample detector distances of 2.2, at 5.2 and at 20 m, using the low-resolution selector with an effective wavelength of 0.36 nm. The sample volume was about 0.2 cm$^3$, and the measuring times 10 min. The angular distribution of scattered neutrons is largely unaffected by the low-wavelength resolution of about 45°, FWHM (After Marguerie & Stuhrmann, 1976). Units of intensity: counts channel/10 min.

Fig. 23. Scattering pattern due to the dislocation structure in Fe single crystals. A horizontal magnetic field (M) has been applied across the sample. The primary beam (P) was not attenuated. Logarithmic scale of neighbouring contours from 32 to 32768 with 20 increments of $2^{10}$. For evaluating the pattern, the radial distribution within 12 sectors of 15° has been determined (Goeltz & Scheuer, 1974) using magnetic small-angle scattering. A horizontal magnetic field has been applied, perpendicular to the incoming beam. In order to separate the magnetic scattering, one measures at two different field strengths. The difference contains only the magnetic part. For evaluating the azimuthal distribution the pattern is divided into radial sectors. A low resolution in wavelength would be sufficient as in the previous case. However, the spreading out of the primary beam in the vertical direction, which is due to the dispersion of the trajectories due to gravity, would not allow a straightforward evaluation of the pattern. Hence a moderate wavelength spread had to be used.

3. Applications of neutron small-angle scattering

The following examples show that we may classify the different types of experiments with respect to the relative resolution in momentum transfer they need:

1) Scattering from isolated, randomly distributed particles leads to a more or less continuous pattern ('diffuse scattering'). The main parameters usually evaluated are the intensity at zero angle and the radius of gyration. Fig. 22 gives an example: The complete scattering curve of 2% fibrinogen in D$_2$O is shown on a double logarithmic scale (Marguerie & Stuhrmann, 1976). It was obtained by matching the experimental curves observed at three different camera settings. The scattering curve is not very sensitive to the width of the wavelength distribution of the monochromatized beam. The Maxwellian-like distribution of the incoming beam allows the use of a fairly broad band of wavelengths, and it is not necessary to use sophisticated mathematical procedures for deconvoluting the data. This can be seen in the following example: consider the influence of a normalized triangular shaped wavelength distribution $f(\lambda)$ with a full width of 100°, reaching from $\lambda_0/2$ to $3\lambda_0/2$, with the most probable wavelength $\lambda_0$ on a Gaussian scattering curve,

$$I = I_0(1 - R^2_0 4\pi^2 \theta^2/\lambda^2)$$

with $R_0$, the radius of gyration. The observed scattering curve is then:

$$I_{obs} = I_0 \left\{ 1 - R^2_0 4\pi^2 \theta^2 \int \left[ f(\lambda)/\lambda^2 \right] d\lambda \right\}.$$

One may correct the observed intensity distribution by taking an effective wavelength $\lambda_{eff} = \lambda_0 [\ln (4/3)]^{1/2}$ = 0.935 $\lambda_0$.

2) The information obtained in anisotropic patterns is more detailed (Fig. 23). With the D11 system, the dislocation structure in iron and nickel single crystals has been studied (Goeltz & Scheuer, 1974) using magnetic small-angle scattering. A horizontal magnetic field has been applied, perpendicular to the incoming beam. In order to separate the magnetic scattering, one measures at two different field strengths. The difference contains only the magnetic part. For evaluating the azimuthal distribution the pattern is divided into radial sectors. A low resolution in wavelength would be sufficient as in the previous case. However, the spreading out of the primary beam in the vertical direction, which is due to the dispersion of the trajectories due to gravity, would not allow a straightforward evaluation of the pattern. Hence a moderate wavelength spread had to be used.

3) Fig. 24 shows the equatorial layer line obtained with an oriented gel of the tobacco mosaic virus. This is a rod-shaped virus of length 300 nm, diameter 18 nm, consisting of a single, helically wound RNA string coated with protein subunits. The relative resolution has to allow for an unambiguous location of the maxima and minima of the equatorial line (Holmes,
Fig. 24. Medium-resolution diffraction pattern of oriented gels of tobacco mosaic virus in D₂O. The orientation has been achieved by preparing the rod-like virus particles in quartz capillary tubes of 0.8 mm inner diameter. About nine tubes have been stacked together forming a total cross section of about 10 × 10 mm. The orientation of the helix axis is indicated by A-A. The first three maxima in the equatorial layer line (E-E) (zero order) are clearly detected on both sides of the beam stop. The experiment should finally give some indication about the RNA conformation in the virus (Holmes, Fuess, Ibel, Jacrot, Mandelkow & Warren, 1974). A medium resolution in wavelength is only needed in order to locate the maxima and minima of the layer line unambiguously. Sample-detector distance: 236 cm; sample-source distance: 500 cm; wavelength: 0.64 nm at 9% FWHM; increment of neighbouring contours by 2½ (720-524000 in 19 increments); uncorrected rough data. The momentum transfer at the position of one corner is at 2πθ/λ = 1.88 nm⁻¹.

Fuess, Ibel, Jacrot, Mandelkow & Warren, 1974). The purpose of the neutron diffraction study is to determine whether the bases of the RNA are situated inside or outside of the helix formed by the sugar–phosphate backbone.

Fig. 25. Diffraction pattern of stacked membranes in solution. Retinal rods of frogs’ eyes have been aligned by a magnetic field (M → M). The rods were suspended in 200 µl Ringer solution in a 2 mm thick, 8 mm wide quartz cell. Logarithmic scale of contours from 1722 to 623487 with 34 increments of neighbouring lines by 2½. The high counting rate near the limit of the maximum transmission rate to the data acquisition system leads to the creation of faulty addresses (F) along the limiting rows and columns and at the zero point (upper left corner). The kinks in the contours near the primary beam reflect the finite size of the detection elements. Sample-detector distance: 224 cm; wavelength: 0.51 nm at 9% FWHM; measuring time: 2990 s; uncorrected rough data. The momentum transfer at the corner is at 2πθ/λ = 2.49 nm⁻¹.

Table 2. Typical measuring times for D11 at a momentum transfer resolution £k = 0.01 nm⁻¹ [k = 4π sin (θ/2)/λ] at FWHM

<table>
<thead>
<tr>
<th>Parameter</th>
<th>δk = 0.01 nm⁻¹</th>
<th>0.5 s</th>
<th>1</th>
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<td>Zero angle scattering</td>
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<tr>
<td>Radius of gyration</td>
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<td>1</td>
<td></td>
</tr>
<tr>
<td>Intensity distribution of continuously</td>
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<td>10²</td>
<td></td>
</tr>
<tr>
<td>scattered neutrons</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Central symmetric sectors of 15%</td>
<td></td>
<td>10³</td>
<td></td>
</tr>
<tr>
<td>Layer lines in fibre diffraction</td>
<td></td>
<td>10³</td>
<td></td>
</tr>
<tr>
<td>Diffraction pattern with nine orders</td>
<td></td>
<td>10⁴</td>
<td></td>
</tr>
</tbody>
</table>

A medium resolution in wavelength is only needed in order to locate the maxima and minima of the layer line unambiguously. Sample-detector distance: 236 cm; sample-source distance: 500 cm; wavelength: 0.64 nm at 9% FWHM; increment of neighbouring contours by 2½ (720-524000 in 19 increments); uncorrected rough data. The momentum transfer at the position of one corner is at 2πθ/λ = 1.88 nm⁻¹.
4. Conclusions

The specifications and the performance of the different parts of the D11 system allow long-range fluctuations to be studied by observing the scattering of cold neutrons within small angles. For a further development, a larger area of the detector with an enhanced number of elements could be useful. For the improvement of the resolution and for studies in a larger momentum space, an enhanced flexibility in positioning of the detector would be necessary combined with a correspondingly higher wavelength resolution.

The actual limit of the system for the study of hydrogen-containing samples with their inherent high scattering cross section is the limit in the transmission rate of counts to the on-line data acquisition facility. With the fluxes available at the high-flux reactor, this transmission rate could be increased by one order of magnitude.

The author is indebted to Professor Maier-Leibnitz who enabled such an instrument to be built at the high-flux reactor. Professor Schmatz and Professor Springer helped to determine the specifications and supported actively the construction. The main mechanical parts were built in the Centralinstitut für Reaktorexperimente in the KFA Jülich. The multidetector was constructed in the Centre d'Études Nucléaires de Grenoble (CEN-G). The work was supported by the technical services of the Institute. J.F. Barthelemy has helped to mount the instrument and to improve it.

References


Fig. 26. Diffraction pattern of collagen, showing along the diagonal (D-D; lower left to upper right) nine orders of the 670 Å meridional repeat unit. Logarithmic scale of contours from 152 to 131072 with 39 increments of neighbouring lines by 214. The primary beam and the very strong first order are attenuated by the semitransparent beam stop. The third and sixth order reflections are dominating; the weak seventh and eight order reflections are not visible, partly because of the statistical noise of the incoherent background. Sample-detector distance: 437 cm; wavelength: 0.57 nm at 9°; FWHM: measuring time: 1800 s; uncorrected rough data. The momentum transfer at the corner is at 2π/λ = 1.14 nm⁻¹. The sample cross section was 7 mm along the fibres and 14 mm perpendicular to them. Collimation length: 20 m.