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Developments in Experimental Neutron Physics at the Institut Laue-Langevin

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Developments in experimental neutron physics at the Institut Laue–Langevin

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A report and survey are given dealing with a variety of recent developments in neutron spectroscopy, diffraction and beam techniques, referring to work at the Institut Laue–Langevin. Subjects such as high resolution spectroscopy by 90° Bragg diffraction and spin echo analysis (leading to 10^{-7} to 10^{-8} eV energy resolution), spin polarizers, focusing crystals, modern multicounters, and methods dealing with neutrons of energies in the 10^{-7} eV region (ultra-cold neutrons), are dealt with.

Introduction

Besides the operation of the high flux reactor for the scientific users, a traditional and genuine task of the Institut Laue–Langevin (I.L.L.) is the innovation, development and improvement of the instruments in neutron physics. Regarding in particular diffractometry and spectroscopy, this task aims essentially at two aspects: first, the improvement of the statistical accuracy of the data and, secondly, the refinement of the instrumental resolution in energy and momentum space, both aspects being naturally related in most cases. This article presents a short review of recent methodical developments connected with these subjects, namely

high resolution spectroscopy;

methods of enhancing intensities and/or count rates, by increasing solid angles, and by multi-counters; and

production of high-intensity polarized neutrons.

The review also deals with the methods of achieving high fluxes or densities of ultra-cold neutrons.

Other means to enhance the accuracy of the results, namely speeding up the instrument control by computers and by sophisticated interaction between results and instrument operation, will not be treated. Nevertheless, we consider these aspects to be as important as those mentioned before. Many of the developments to be treated, e.g. multicounters, are so efficient that they also allow a low flux reactor to serve as a tool for many interesting applications.

High resolution spectroscopy

At present, we envisage four fundamental ways to define energies and energy transfers in neutron spectroscopy: (i) Bragg reflexion; (ii) measurement of flight-times with fast chopping devices; (iii) time-of-flight by using the 'inherent clock' of a neutron passing through a magnetic field and finally (iv) comparing the neutron's kinetic energy with interacting fields, such as gravity (Steyerl 1978), magnetic fields, and also the average potential of the nuclei

in solids, by using neutron mirrors or prisms. A possible fifth way is by neutron interferometry. This review will deal only with items (i), (ii) and (iii).

Time-of-flight methods

The new IN 6 time-of-flight spectrometer will use a new version of time focusing (Scherm *et al.* 1977*a*) to achieve improved resolution (20–100 μeV) at a chosen energy transfer and with increased neutron intensity. Three separate beams with different wavelengths fall upon the sample. The longest wavelength beam is chopped first and the shortest last in each cycle. Flight paths to the sample are arranged so that all pulses coincide at that point to give an effective short burst time.

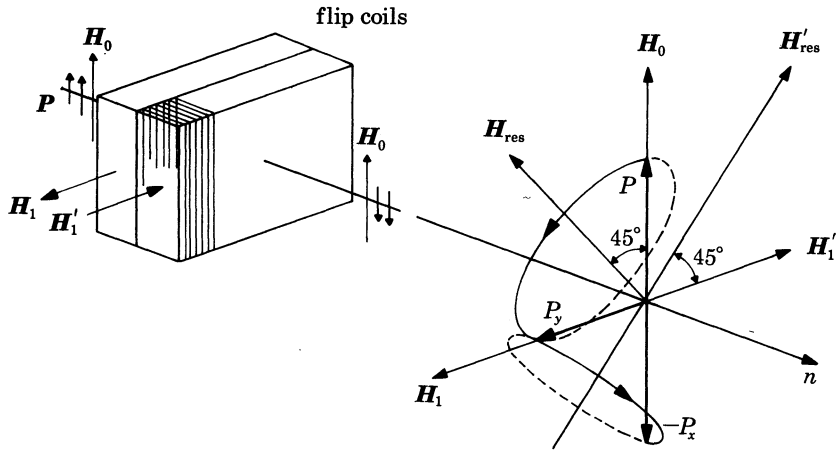

FIGURE 1. Continuous field spin flipper: the resulting H_{res} (or H_{res}') from the constant magnetic fields H_0 and H_1 (or H_1') is the axis inclined at 45° about which the spin $\boldsymbol{P}$ rotates when the neutrons pass through. By an appropriate choice of the time of flight in the field of the coils, a spin rotation from P_x to P_y (a single coil) or to $-P_x$ (the two coils) is obtained ($\frac{1}{2}\pi$ (or π-flipper); Badurek & Westphal 1975).

Another elegant time-of-flight method consists of bringing a polarized neutron beam to a spin analyser and inserting a spin flipper, as thin as possible, in front of the sample, which turns the spin through 180° when the electric current is switched on and off (Badurek & Westphal 1975) (figure 1). Such a non-mechanical device is limited by the inertia of the electrical switching process: rise times of *ca.* 4 μs were achieved with a beam of 5 cm × 5 cm. This method is already in use on the spectrometer D7. Shorter pulses would give shorter flight paths and thus greater intensity. Finally, the geometrical spread of the flight path due to sample and detector thickness sets a limit in time-of-flight experiments. The development of thin detectors such as the scintillation detectors under study for the Rutherford Laboratory Spallation Neutron Source promise further progress (Wroe 1978).

Backscattering spectroscopy

Diffraction at Bragg angles $\theta_B = 90°$ is well known as a method to achieve an extremely high resolution (Alefeld *et al.* 1969). In this backscattering situation, the neutron wavelength depends on θ_B only to second order. The remaining resolution width, caused by second-order effects in θ_B and by primary extinction in the crystal, is of the order of 0.2 μeV f.w.h.m. at best for an Si (111) reflexion (IN 10 spectrometer at the I.L.L.) and there is no way to pass far beyond this resolution limit.

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A certain drawback of the existing backscattering instruments is their lack of flexibility. The range of energy transfers to be covered by the Doppler drive at the monochromator (piston of a motorcycle) is limited to transfers $|\hbar\omega| \leqslant 20$ μeV. Furthermore, there is no way to adapt the energy resolution to the problem under investigation. In this connection, a new instrument is under construction at the I.L.L. called IN 13 (Buevoz & Heideman 1978), which covers a much larger range of energy transfers $\hbar\omega$, and of resolutions (figure 2). By changing θ_B from exact backscattering to $\theta_B = 70°$ and changing the lattice parameter of the CaF_2 (422) monochromator by heating it up to 500 °C, an energy scan from -300 to $+500$ μeV, and an energy resolution in the range between 3 and 20 μeV, can be achieved.

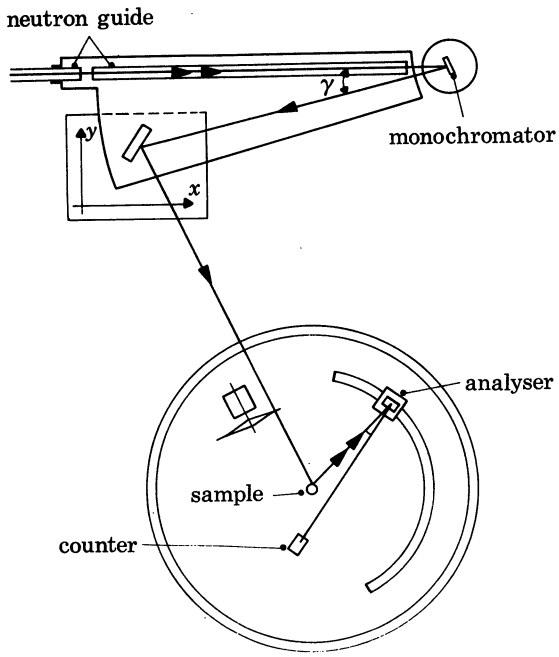

FIGURE 2. High-resolution crystal spectrometer IN 13. The Bragg angle $\theta_B = 90° - \gamma$ can be varied from 70° to 90° (backscattering), and the lattice plane distance of the monochromator by heating the crystal. Several analysers with associated detectors are provided. The energy scan is affected by varying γ and/or the monochromator temperature. The deflector crystal on the x–y plate ensures that the beam always strikes the sample (Buevoz & Heidemann 1978).

The broadening of the resolution width increases the intensity, provided the crystal is sufficiently imperfect. As a future development, a furnace is under consideration that applies a heat gradient over the crystal, thus providing a controlled increase of the resolution width. This spectrometer, installed at a thermal guide, reaches Q values as high as 5.5 $Å^{-1}$†. This is important for many experiments dealing with diffusion in solids and with molecular rotations in crystals, since information on the geometry of such motions is only obtained if $1/Q$ is of the same magnitude as the typical distances of these motions.

A further development, existing only in a preliminary state, aims at the highest possible resolution (i.e. 0.3 μeV) in connection with very large energy transfers, $\hbar\omega$. This can be achieved by combining monochromators with different analyser crystals. By using a sufficiently fast Doppler drive (Bauer 1972), the energy gaps for different pairs of crystals can be covered

† 1 Å = 10^{-10} m = 10^{-1} nm.

entirely. A spectrometer of this kind would allow the investigation of low energy librational states up to nearly 1 meV (e.g. CaF_2 (200) paired with Ge (111) yielding 700 μeV $\leqslant \hbar\omega \leqslant$ 940 μeV; A. Heidemann & Jenkins, 1979), unpublished).

Spin echo spectroscopy

An elegant time-of-flight method developed in the I.L.L. uses the 'clock' individually attached to the neutron, namely the Larmor precession in magnetic field sections before and after the scattering sample. The principle of this spectrometer, called 'spin echo', has been described in detail (Mezei 1972, 1978; Hayter 1978), and I deal only with several relevant aspects of the method (see figure 3). The flight-time difference before and after scattering is determined by the difference of the Larmor angles in the magnetic field sections before and after the sample,

$$\delta\phi = (\Omega_0 L_0/v_0) - (\Omega_1 L_1/v_1); \tag{1}$$

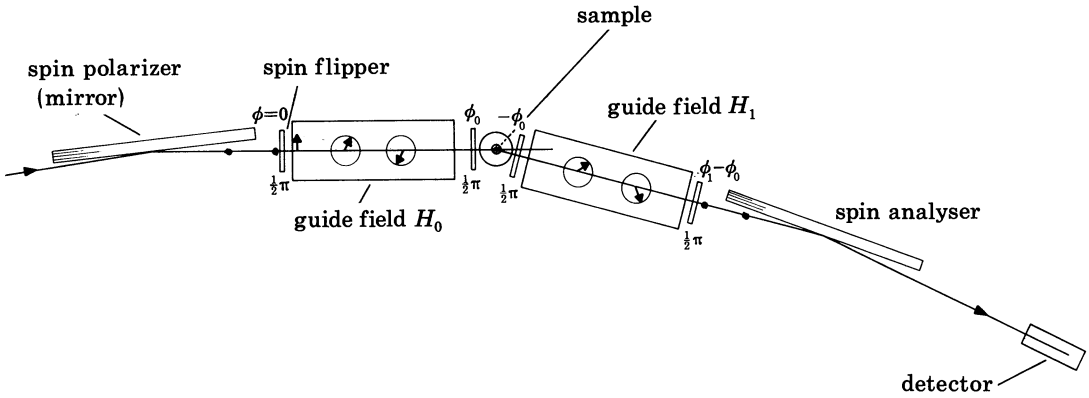

FIGURE 3. Principle of the spin echo spectrometer. A magnetized mirror or diffraction crystal can be envisaged as spin analyser or polarizer. The $\frac{1}{2}\pi$ coils operate on the principle shown in figure 2. The incident spin is perpendicular to the plane of the drawing. The difference of Larmor angles for the guide fields before and after scattering $\delta\phi$ is measured (Mezei 1972).

where $v_{0,1}$ and $L_{0,1}$ are the neutron velocity and the flight distance before and after scattering, respectively, and $\Omega = (4\pi\mu_n/h)\ H$ is the corresponding Larmor frequency. To avoid phase differences for neutrons entering or leaving the magnetic field sections at different positions in the beam, each neutron has to be rotated such that it starts with the same angle of precession within a well defined start (or arrival) plane (figure 3). This is achieved by a 90° spin flipper (action of only one of the two coils shown in figure 1). To obtain the flight-time *difference* according to (1), a spin rotation has to be applied near the sample position by a π-coil.

For purely elastic scattering ($\hbar\omega = 0$) and for an exactly symmetric arrangement ($H_0 L_0 = H_1 L_1$), one obviously has $\delta\phi = 0$. For a very small energy transfer,

$$\hbar\omega = \tfrac{1}{2}m/(v_0^2 - v_1^2) \ll \tfrac{1}{2}mv_0^2, \tag{2}$$

a change of the phase angle is observed which depends on $\hbar\omega$, and which is measured by means of the polarization behind the field section H_1. Differentiation of (1) and (2) with respect to v_1 and v_0 leads to the condition that $\delta\phi$ is *linear* in $\hbar\omega$, namely

$$H_0/v_0^3 = H_1/v_1^3. \tag{3}$$

This yields

$$\delta\phi = \phi_0(\hbar\omega/2E_0). \tag{4}$$

Since the polarization after the second spin flipper is proportional to cos $\delta\phi$, a quasi-elastic energy distribution of the scattered neutrons, $S(\omega)$, produces an average polarization

$$\langle P(\phi_0)\rangle = P_0 \int S(\omega) \cos(\phi_0 \hbar\omega/2E_0)\, d\omega, \quad (5)$$

where P_0 is the polarization for purely elastic scattering. Obviously, the average polarization as a function of ϕ_0 or of the magnetic field $H_1 = H_0$ directly yields the Fourier transform of $S(\omega)$. Equation (5) holds for incident spectra of a width below, say, 10%.

The spin echo spectrometer IN 11 based on this principle is now working in routine operation. To define its resolution, we assume that $S(\omega)$ is a Lorentzian with a half width 2Γ. Then the polarization ratio is given by $\langle P\rangle/P_0 = \exp(-\pi\Gamma N/E_0)$, where N is the total number of Larmor precessions (*ca.* 10^4) and E_0 is the incident energy. We define the resolution by a drop of $\langle P\rangle/P_0$ from 1.0 to 0.95 for the magnetic field range covered by the instrument (this value is considered as an easily measurable polarization change). This definition leads to an accuracy for the width of about 3×10^{-9} eV with 8 Å neutrons.

Each neutron obviously measures its velocity change individually (independent of v_0 to first order). Consequently, this very high resolution can be achieved with a broad incident spectrum, with the spectrometer IN 11, at present with a width of about 5×10^{-4} eV. This is 10^5 times larger than the resolution in energy transfer. In other words, the spin echo principle decouples the ω-resolution from the width of the incident spectrum. (This should be compared with the fact that the backscattering spectrometer decouples the ω resolution from the *angular width* of the incident beam.) Up to now, experience has demonstrated that the spin echo instrument IN 11 is certainly superior to the backscattering instrument IN 10 for high resolution quasi-elastic scattering experiments as long as they also require a good resolution in momentum (less than 10^{-2} Å^{-1}), in particular for critical scattering, or coherent polymer scattering. On the other hand, the backscattering spectrometer is normally equally good or superior for most of the incoherent scattering experiments.

At present, two further applications of the spin echo principle are under investigation, and will be tested at the D 10 spectrometer in the near future.

(i) The high resolution of the spin echo method allows a reduction of the thermal diffuse scattering (t.d.s.) occurring in the vicinity of Bragg reflexions (Hayter *et al.* 1979). This is feasible by separating the elastic part of the scattering, where the Larmor angle difference $\delta\phi$ is zero, from the phonon scattering, which leads to dephasing and therefore to a decay of the average polarization. A separation of phonons with energies not more than 50 μeV appears possible, which may reduce the t.d.s. contribution by a factor of more than 10. This will be important for accurate structure determinations.

(ii) The high resolution of the spin echo method can be applied to investigate the shape of phonon lines. Since the phonon peaks occur at relatively large energy transfers, this requires the asymmetric operation of a spin echo instrument, i.e. $v_0 \neq v_1$ and $H_0 L_0 \neq H_1 L_1$ in (1). Calculations have demonstrated that a triple-axis spectrometer with polarized neutrons applying this asymmetric spin echo principle will achieve a resolution which is about ten times better than the values achieved with triple-axis spectrometers like IN 3 with focusing, at a flux 30 times lower. The resolution with respect to the wavevector of the phonons, q, compatible with such an energy resolution had to be 10^{-4} to 10^{-5} Å^{-1}. To avoid the intensity loss caused by such a q-resolution, $q\omega$-focusing has to be applied. It can be achieved if the axis of the

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magnet is inclined against the beam direction so that a linear relation is obtained between the inclination of a neutron path in the magnet against the axis, and the phase angle $\delta\phi$ (Mezei 1978; Pynn 1978).

Multidetectors and focusing crystals to increase count rates

Multicell counters

The conventional method of investigating crystal diffraction patterns, or diffuse scattering from disordered crystals, is an intensity measurement point by point. Multicell counters yield an enormous gain in experimental time and/or statistical accuracy. This would hold, in particular, for crystals with large lattice cells, where many reflexions have to be studied. For the first time, multicounters were introduced in the I.L.L. for the small-angle cameras D 11 and D 17, using BF_3 (Allemand *et al.* 1975). More recently, a multicounter 'slice' for a single crystal diffractometer is under construction (D 19) filled with He^3 at 10 bar†. The counter has 16×512 cells for neutron localization, with a resolution of about $0.1° \times 0.2°$. This device is considered as a prototype for a bigger and advanced cylindrical multicounter for single crystal work (in particular for proteins) with a size of $70° \times 120°$, and about 10^5 cells. Such a counter would create data fluxes of 10^5 words per second, and, apart from mechanical difficulties with the counter itself, the on-line data reduction would be a serious problem.

A different application of multicell counters is the simultaneous registration of diffraction patterns as a function of time. This has many applications for kinetic experiments, as concerns problems of chemisorption, solid state reactions (as intercalation), or H–D exchange in macromolecules. A banana-shaped counter with 400 cells, covering an angular range of 80°, is operated with the powder diffractometer D1B. The development of such detectors obviously aims at very high count rates. The best expected for the near future (detector D 20, ^3He, with 1800 wires) would be a count of about 10^5/s per cell. If 10^3 events per reflexion are considered sufficient in terms of statistics, a pattern could be measured within 10 ms. The use of conversion electrons for multicounters from neutron capture in ^{157}Gd is an alternative under development (Jeavons *et al.* 1978).

Increasing solid angles

In the past, it has been repeatedly argued that the virtue of thermal neutron guides is diminished by the small critical angle γ_c accepted by total reflexion. In particular, for the vertical direction, relatively large angles are tolerable and desirable in many instances (in particular for diffractometers). In this respect, considerable progress has been achieved by *vertical focusing*. This means 'transforming' the great height of a neutron guide into angular width. A typical *monochromator* for vertical focusing has a height of 10–15 cm, consisting of 10–30 lamellae made of Cu, Si, Ge or pyrolytic graphite, on an elastic metal sheet with variable radius of curvature. At a focal distance of say 2 m, the resulting intensity spot may have a height of about 3 cm, thus increasing the intensity by a factor of 3. The vertical height of the focal spot is essentially caused by the mosaic spread of the monochromator and the divergence of the incident beam. Careful cold working of ideal crystals with selected glide planes of dislocations leads to the desirable increase of spread in the horizontal plane, but, at the same time, leaves the mosaic spread small in the vertical direction (Freund 1975; Freund & Forsyth 1978).

† 1 bar = 10^5 Pa.

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Such devices are used at the I.L.L. for triple-axis spectrometers and diffractometers. Recent developments of focusing in space *and* in energy by horizontally curved crystals, applied in triple axis spectrometry will not be treated here (Scherm *et al.* 1977*b*).

An interesting way to improve neutron mirrors was developed recently by Mezei (1976, 1977). A multilayer sheet can be produced by repetitive evaporation of two materials with a different refractive index. Consequently, in addition to normal total reflexion below the

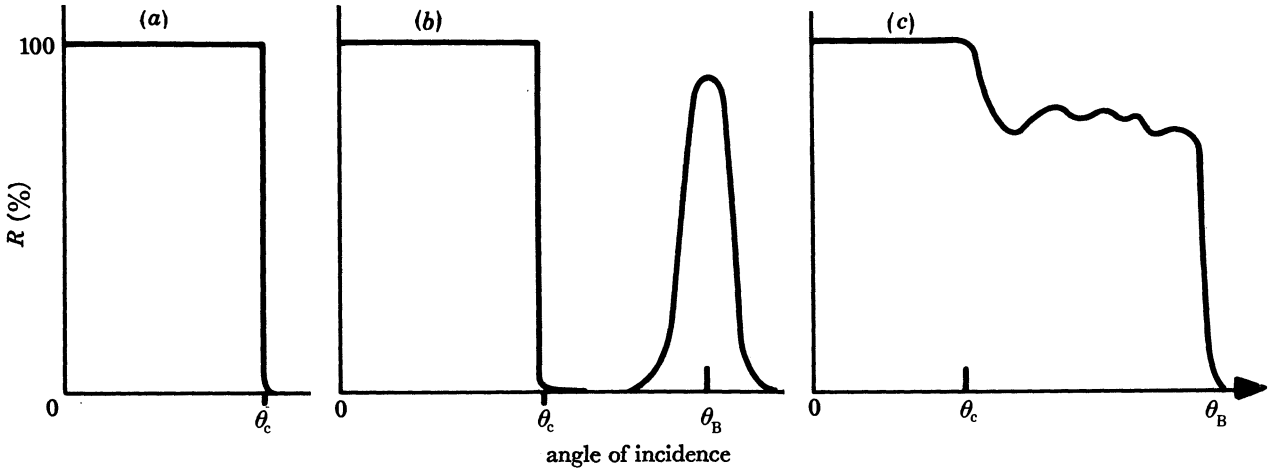

FIGURE 4. Neutron reflectivity, R (schematic): (*a*) a simple total reflecting mirror, (*b*) a multilayer mirror (equidistant double layers of different refractive index) and (*c*) variable distance between layers. θ_c = critical angle, θ_B = Bragg or Bragg cut-off angle (from Mezei 1976, 1977). For a Ni-coated guide at 2 Å, $2\theta_c = 0.40°$ (for ^{58}Ni, $2\theta_c = 0.47°$). For the multilayer mirror, θ_c corresponds to the average refractive index.

critical angle $\overline{\gamma}_c$ of the mixture of the two materials, a Bragg reflexion due to the periodic structure appears at an angle beyond $\overline{\gamma}_c$. By introducing a gradient with respect to the layer distance, and by a proper choice of this gradient, it is possible to broaden the Bragg peak asymmetrically until it merges with the cut-off angle $\overline{\gamma}_c$ ('supermirrors', figure 4). By using an alternation between magnetic and non-magnetic layers, this type of mirror also serves as a spin polarizer. So far, the following characteristics have been achieved by F. Mezei: 75 double layers Fe/Ag with a gradient of distance (average 300 Å) yields a reflectivity of nearly 100 % up to $\overline{\gamma}_c$, which decreases to about 70 % between $\overline{\gamma}_c$ and the Bragg cut-off angle, which occurs at about 2.4 $\overline{\gamma}_c$. Above $\overline{\gamma}_c$, the polarization of a reflected neutron beam is 99 %; below $\overline{\gamma}_c$ it decreases to 97 %. Obviously these mirrors are excellent polarizers and are already used in several instances. As concerns their application for neutron guides, the gain by the large cut-off angle is only effective if the number of reflexions is small, because of the poor reflectivity above $\overline{\gamma}_c$. An evaporation unit for routine production of such mirrors will start operating at the end of 1979.

3. PRODUCTION OF ULTRA-COLD NEUTRONS

This topic, being somewhat outside the main scope of the review, is related to experiments with ultra-cold neutrons (u.c.n.), where 'ultra-cold' means kinetic energies of the order of 10^{-7} eV ($\lambda = 1000$ Å, $V = 4$ m/s). This is below the average nuclear interaction potential of most solids (also below the interaction potential of the neutron dipole moment with high magnetic fields). Consequently, total reflexion can be achieved even at perpendicular incidence,

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which is the basis of many interesting experiments, for instance the measurement of the electric dipole moment of the neutron (or of its lifetime against β decay). This paper does not deal with such experiments and I restrict myself to the problem of the 'production' of such neutrons (for reviews see Lushikov 1977; Steyerl 1977; Golub & Pendlebury 1979).

The neutron current $I(E)$ at energy E which leaves a neutron guide at the reactor is described by a Maxwellian at the moderator temperature T_M. This is a good approximation for a D_2O moderator, even at very low energies. One gets

$$I(E)/\mathrm{d}E = \phi_0(\Delta\Omega/4\pi)\exp(-E/kT_M)\,E\mathrm{d}E/(kT)^2\,\mathrm{d}E, \tag{6}$$

where $\Delta\Omega/4\pi \approx \frac{1}{4}$ is the solid angle for u.c.n. accepted by the guide, and ϕ_0 is the total thermal flux.

Integration of (6) up to, for our example, $E = 1.9\times10^{-7}$ eV (6.2 m/s) yields a total extracted u.c.n. current of $I_{\mathrm{u.c.n.}}$ ($\leqslant$ 6.7 m/s) $= (\frac{1}{8})\,\phi_0(E_{\mathrm{u.c.n.}}/kT)^2 \approx 3\times10^3\ \mathrm{s^{-1}\,cm^{-2}}$ with $\phi_0 = 0.4\times10^{15}\ \mathrm{s^{-1}\,cm^{-2}}$ for the disturbed thermal flux, as obtained at the inclined hole IH 3 (Ageron 1978).

Owing to high absorption in the wall of the beam-hole thimble, only a small fraction of the u.c.n. could escape. To improve this situation, a special installation has been constructed in the IH 3 beam hole, called 'PN 5'. The front wall of the tube has been covered on its inside by a *ca.* 2 mm layer of streaming water, with a 100 μm aluminium window to prevent absorption. The H_2O scatters the thermal neutrons into the u.c.n. region (originally depleted by absorption); thus the spectrum in this range recovers. Nevertheless, owing to reflexion losses in the u.c.n. neutron guide (10 m long, 7 cm × 7 cm cross section, Ni-coated glass), and owing to air gap and window losses, only about 80 u.c.n. $\mathrm{s^{-1}\,cm^{-2}}$ reach the detector (Ageron 1978).

A method of improving the extraction losses is a neutron turbine (Steyerl 1975) sitting outside the reactor at the end of a neutron guide. In momentum space, it shifts the neutrons from high energies down to u.c.n. energies, thus avoiding the extraction losses at u.c.n. energies (of course, Liouville's theorem says that one certainly cannot get more than the density corresponding to the equilibrium value in momentum space, $\rho = \mathrm{d}^6N/\mathrm{d}x\,\mathrm{d}y\,\mathrm{d}z\,\mathrm{d}p_x\mathrm{d}p_y\mathrm{d}p_z = \{\phi_0/8\pi m_N(kT)^2\}\exp(-E/kT)$; see, for example, Maier-Leibintz 1966). This method has been worked out at the Munich reactor and its application at the I.L.L. is being considered.

In many experiments, a high u.c.n. *density*, $\rho_{\mathrm{u.c.n.}}$, is needed instead of a high current. Outside the 'PN 5' hole, the u.c.n. current quoted above would lead to a value

$$\rho_{\mathrm{u.c.n.}} = 4I_{\mathrm{u.c.n.}}/\bar{V}_{\mathrm{u.c.n.}} = 0.8/\mathrm{cm^3} \tag{7}$$

for an ideal and empty bottle, with $\bar{V}_{\mathrm{u.c.n.}} = 4$ m/s.

Some time ago a very interesting proposal was developed to increase considerably the u.c.n. density outside the reactor (Golub & Pendlebury 1975, 1979). A beam of 'medium' energy (*ca.* 10^{-3} eV), with no appreciable extraction losses, penetrates the wall of a cryostat filled with superfluid ^{4}He. There the neutrons interact with the phonon-like elementary excitations of the helium. A large proportion of the neutrons lose almost all of their energy and momentum in these processes, with the simultaneous production of quanta of helium elementary excitations. (For a *solid* at extremely low temperature this would not apply: although there exist phonon excitations, at low temperature the majority of the scattering processes are purely elastic and therefore not effective.) For a 3 m long closed helium 'neutron bottle', a stationary density can be produced, determined by the equilibrium between the u.c.n. production rate per

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volume unit, $P_{\text{u.c.n.}}$, and the unavoidable losses. The losses are described by an effective lifetime τ_{eff}. This results primarily from the temperature-dependent energy gain or up-scattering processes in superfluid helium (τ_{g}), from the walls (τ_{w}), from the absorption in the isotope ^{3}He (τ_{a}, avoidable by isotope purification), and of course from the β-decay of the neutron $\tau_\beta = 907$ s. For the equilibrium density in the superfluid helium one obtains

$$\rho_{\text{u.c.n.}} = P_{\text{u.c.n.}}/\tau_{\text{eff}} = P_{\text{u.c.n.}}(1/\tau_{\text{g}} + 1/\tau_{\text{w}} + 1/\tau_{\text{a}} + 1/\tau_\beta + \ldots). \quad (10)$$

Under favourable conditions a lifetime is expected that is governed by the wall losses and the helium up-scattering; with $\tau_{\text{eff}} = 30$–100 s and with $P_{\text{u.c.n.}}$ from the approximately calculated helium up-scattering processes (Golub 1979), we obtain a density $\rho_{\text{u.c.n.}}$ for this bottle, which is almost 100 times greater than the figure obtained by using the existing 'PN 5' beam.

A preliminary test with a stationary beam, with incident neutrons of 10^{-3} eV in helium at 1.1 K, showed that the estimated production rate $P_{\text{u.c.n.}}$ is, in fact, achieved (Ageron *et al.* 1978). The loss processes due to up-scattering in helium (Golub 1979) and particularly in hydrogen absorbed on the walls (Stoika *et al.* 1978) can be estimated theoretically. It will certainly be possible to achieve improvements as regards the wall losses. Such a He facility is planned and it will essentially allow a repetition of the search for the neutron's electric dipole moment, and other experiments.

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