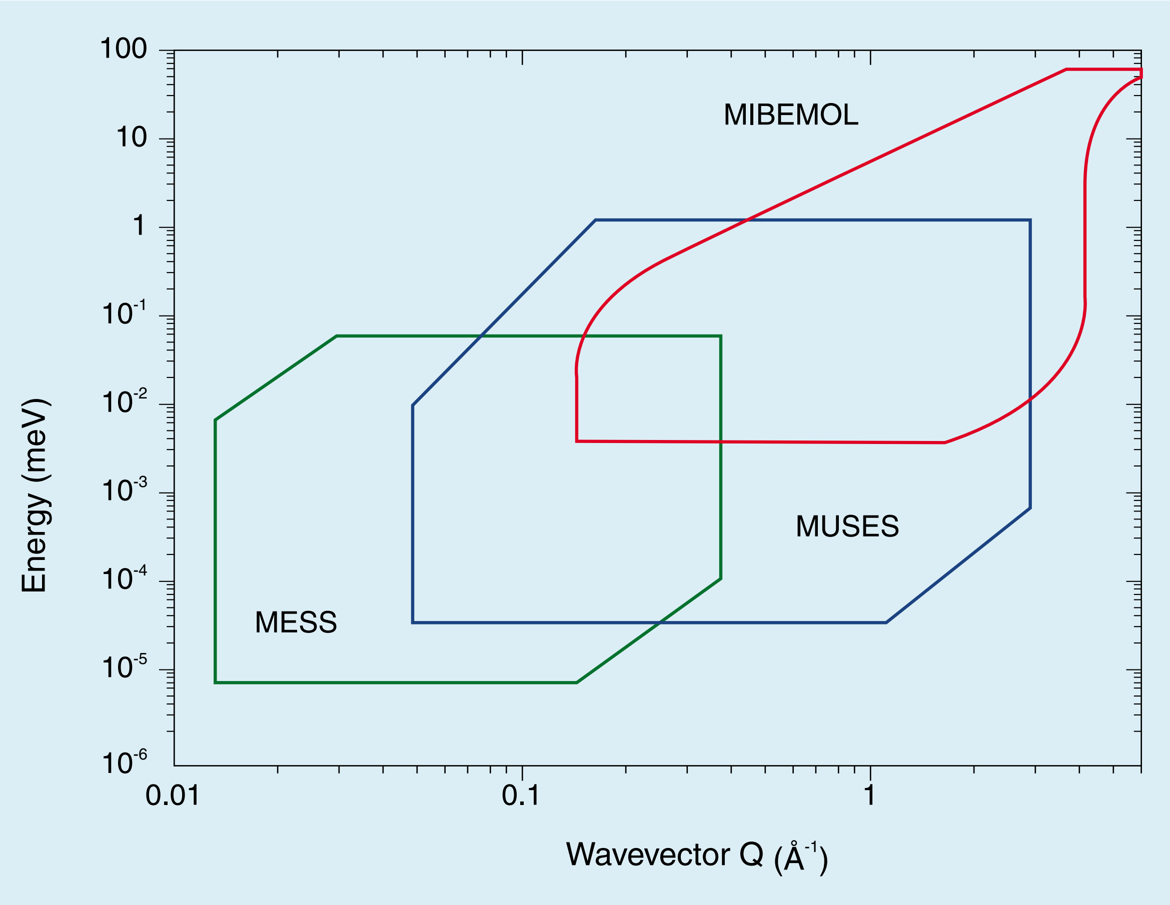
II

Quasi-elastic and high resolution instruments

HIGH RESOLUTION SPECTROMETERS FOR INELASTIC AND QUASIELASTIC NEUTRON SCATTERING

There are 3 different high resolution instruments at the LLB : a time-of-flight spectrometer (Mibémol) and 2 neutron spin-echo spectrometers (Mess and Muses). Each spectrometer cover domain in the energy-wavevector space which is complementary to the others (see figure). It is then possible to measure condensed matter dynamics from microscopic to mesoscopic length scales over more than 6 orders of magnitudes in time (0.1 - 50000 ps). The three spectrometers use cold neutron beams of the Orphée guide hall.



Energy - wavevector domain cover by each spectrometer

In time-of-flight spectroscopy, neutron energy changes are directly measured by differences between incident and scattered neutrons beam energies.

MIBEMOL uses a primary chopper cascade spectrometer to produce roughly monochromatic pulsed beam. Energy analysis is performed by measurement of the time-of-flight from sample to detector. The scattered neutron beam is recorded simultaneously over a wide range of angle. This instrument is very versatile with respect to wavelength and energy resolution which depend on chopper speed and respective phases. This spectrometer is generally used to measure dynamics of sample with a smooth wavevector dependent intensity like incoherent or coherent scattering of non long range ordered systems. The measurements are performed at constant angle, thus **Q** and ω are strongly correlated. Medium energy phenomena (E < 15 meV) can be measured in neutron energy gain and as a particular consequence of the instrument flexibility, measurements can be performed with everything constant but the energy resolution using different configurations.

In neutron spin-echo spectroscopy neutron energy changes are measured via neutron spins. This trick allows a decoupling of incident beam monochromatisation and energy analysis. Hence this technique can use a broad wavelength distribution and the measured quantity (scattered beam polarization) is, to the first order, proportional to the intermediate scattering function I (Q, t).

MESS is a small angle neutron spin echo spectrometer. It get advantage of the strong SANS intensity from coherent scattering length density differences (the use of H-D contrast is particularly important) to compensate for the flux decrease arising from the necessary beam collimation of the SANS measurements. The precession regions are very long (sample to detector distance ~ 6 m) thus the maximum Fourier time is ~ 40 ns.

MUSES is optimized for higher angle measurements with a very high intensity at the sample position and a strong flexibility. It can be used to measure coherent scattering and isotopic incoherent scattering processes over broad wavevector domain and high resolution. Spin incoherent scattering intensity can also be measured (in absence of significant coherent scattering) although the inherent P = -1/3 reduction of the scattered beam polarization. Both spectrometers MESS and MUSES can also be used for polarization analyzis.

	Mibémol	**MUSES**	**MESS**
λ (Å)	2 ... 12	3.5 ... 14	5 ... 10
2θ (°)	37 ... 142°	5 ... 110°	1.5 ... 25°
Max E gain (meV)	30	0.6	0.6
δ E (μev)	20 ... 500	0.030	0.016
Q max (Å^{-1})	4.0	2.75	0.5
Q min (Å^{-1})	0.2	0.05	0.016

Beam tube .... Cold G6. Neutron guide 2.5 x 5 cm^2
Incident wavelength .... $2 < \lambda < 12$ Å
Range of incident energies .... $0.6 < E < 20$ meV
Monochromator = counter rotating choppers .. 20 000 RPM (equivalent)

Elastic energy resolution ....$1\ \% < \frac{\Delta E}{E} < 8\ \%$

Distance from sample to detectors .... 3. 58 m
Horizontal divergence .... ±0.1° per Å on the sample
Vertical divergence .... ±0.1° per Å on the sample
Flux at specimen .... 1.2×10^4 n/cm^2/sec at 5.0 Å.
Beam size at specimen .... 2.5 x 5.0 cm^2

Detectors (size and scattering angle at specimen) :

★ 400 ^3He detectors (width = 32 mm, height = 370 mm) located at 67 positions ($\Delta\theta = 1.3°$, $\Delta\Omega = 5.6\ 10^{-3}$ sterrad) $35° < 2\theta < 147°$
★ 32 ^3He detectors (width = 32 mm, height = 250 mm) located at 4 positions between $12° < 2\theta < 32°$.

Ancillary equipments available

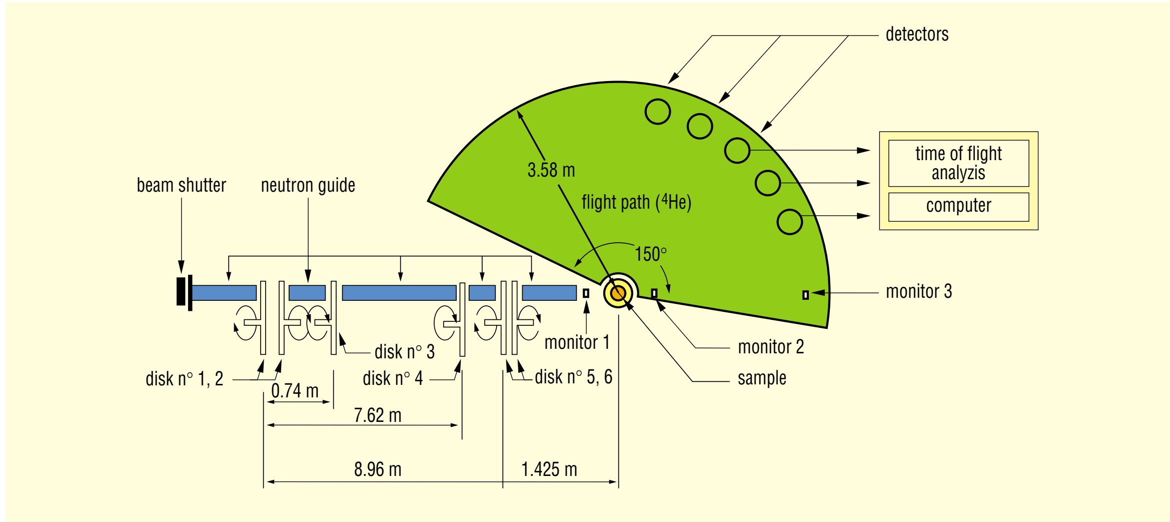
★ Cryostat 1.5 K < T < 300 K
★ Cryogenerator 10 K < T < 300 K
★ Furnace 50°C < T < 400°C
★ Cryofurnace 4 K < T < 600 K
★ Thermo regulated bath -40°C < T < 100°C
★ High Temperature furnace 200°C < T < 1200°C
★ Cryoloop 110 K < T < 700 K

MIBEMOL is an inelastic time-of-flight neutron spectrometer. It is designed to study soft non dispersive excitations in condensed matter between 0.01 and 100 meV (1 meV = 8 cm^{-1} = 0.25 Thz). The corresponding time-scale ranges from 10^{-13} up to 10^{-10} seconds.

Typical study performed on the instrument cover field as different as spin dynamics in high Tc superconductors, tunneling, dynamics of quantum liquids, dynamics of soft matter, biology, local and long range diffusion in disordered systems.

The spectrometer is settled at the end of the G 6 cold guide. The monochromatisation of the incident beam is achieved by a system of six choppers. The flight path from end of the guide to sample is under primary vacuum. To avoid scattering by atmospheric water the time-of-flight basis is filled with He.

As shown on Fig.1, flux at sample, energy resolution and accessible Q range (not shown) are strongly dependent of incident wavelength on sample. Mibémol is a very versatile instrument that makes possible to set-up those parameters so as to match with the best conditions needed to deal with the excitation under study. Some numerical examples showing large increase of flux upon spectrometer setting are given in table 1.



General layout of the time-of-flight spectrometer G 6-2.

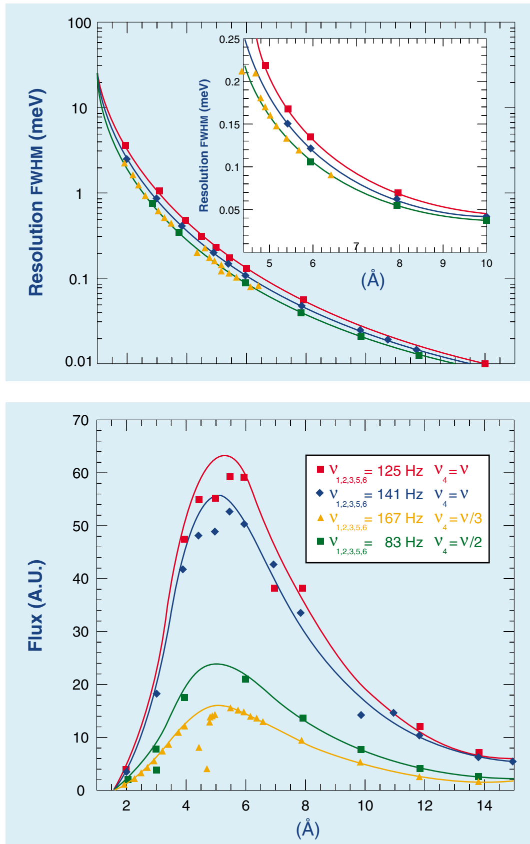


Fig. 1 : *Examples of some achievable instrumental conditions on Mibémol as function of neutron incident wavelength.*
For a given incident wavelength, while the resolution is a slowly varying function of the speed of the choppers, the flux is strongly dependent of this parameter.
Top : Corresponding energy resolution (FWHM). Symbols and colors are the same as for bottom plot. Resolutions achieved for usual wavelength are shown in the inset.
Bottom : Flux at sample as a function of the speed of the choppers. For all curves, frequencies of chopper 1, 2, 3, 5, 6 are equal. The frequency of chopper 4 (anti-overlap chopper is indicated).

		$\nu_2 = \nu_1$		$\nu_2 = 0$	
R (µe V)	$\nu_{1,3,5,6}$ (Hz)	λ (Å)	Flux (A.U.)	λ (Å)	Flux (A.U.)
	166	5.8	15.9	5.9	23.5
100	133	6.3	18.4	6.4	26.9
	83	7.3	22.8	7.5	32.7
	166	9.3	6.3	9.5	8.9
24	133	10.0	6.4	10.3	9.0
	83	11.8	6.4	12.0	9.0

Table 1 : *Selected examples showing the increase of flux obtained for two usual energy resolutions (R) by interplay of chopper frequencies (ν) and initial wavelength (λ). For each resolution, calculations have been made by considering $\nu_2 = \nu_1$ and $\nu_2 = \nu_1/3$.*

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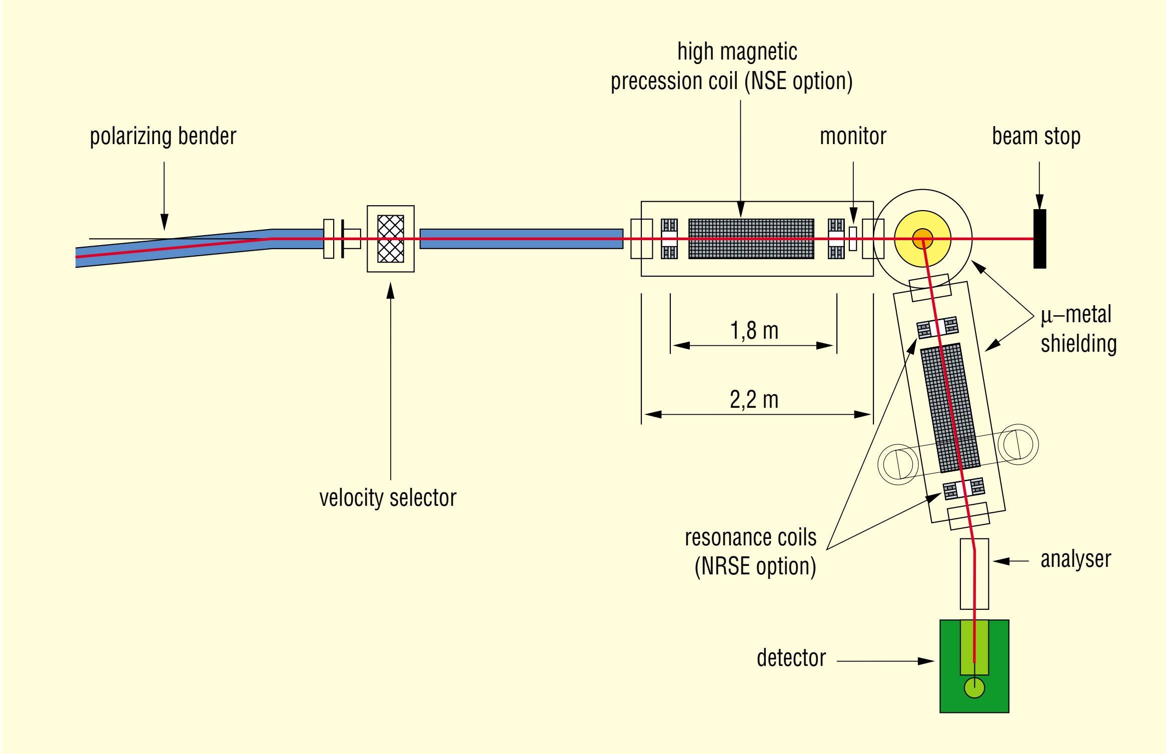
G 1 BIS Neutron resonance spin-echo Spectrometer MUSES

Beam tube	Cold G 1 bis. Neutron guide 2.5 x 5 cm^2, polarizing bender	
Incident wavelength	3.5 < λ < 14 Å	
Range of incident energies	0.4 < E < 6.7 meV	
Monochromator = Dornier velocity selector	max speed 28 000 RPM $\frac{\Delta\lambda}{\lambda}$ = 0.1 - 0.15	
Beam area at sample position	4 x 4 cm^2	
Flux at sample position (polarized)	10^7 n/cm^2/sec at 5.0 Å.	
Divergence	± 0.1° per Å on the sample	
Distance sample - Detector	3100 mm	
Field path integral with NSE Option	0.5 - 50 G.m	
Frequency range of the RF coils	40 - 640 kHz	
Distance between RF coils	1800 mm	
Time range	λ = 3.5 Å	1.2 ps ... 1.1 ns
	λ = 6.0 Å	6 ps ... 5.0 ns
	λ = 10 Å	29 ps ... 22.0 ns
Energy range	λ = 3.5 Å	6.0 10^{-4} ... 0.55 mev
	λ = 6.0 Å	1.3 10^{-4} ... 0.11 mev
	λ = 10 Å	3.0 10^{-5} ... 0.02 mev
Angular range	4 - 110°	
Polarizing/analysing devices	★ Polarizing bender R = 76 m, FeCo/TiNi supermirrors m = 2.5 on the concave side and m = 2 on the convex side, the 25 mm beam is divide into 3 channels of 8 mm ★ Analysing device : supermirrors R = 17 m, 5 x 5.6 x 500 mm^3	
Ancillary equipments available	★ 10 K < 800 K ★ 4 K < 600 K ★ 200°C < 1800 °C	

MUSES is a mixed resonance-conventional neutron spin echo spectrometer installed on the guide G 1 bis. The aim of this spectrometer is to study high resolution quasielastic scattering in the medium wavevector range (0.05 Å^{-1} < Q < 2.75 Å^{-1}) bridging the gap between Time-of-flight spectroscopy and SANS Neutron Spin-Echo at the LLB.

The spectrometer is divided into two distinct parts, a conventional NSE spectrometer for measurements at small Fourier times (typically τ < 200 ps for λ~10 Å) and an NRSE option for measurements at longer times. In resonance spin-echo spectrometry, the two high magnetic precession coils are replaced by four radio-frequency coils ; two in the first arm and two in the second. Only within these coils the spins are submitted to magnetic field and consequently the remaining neutron path has to be shielded from any magnetic contamination (earth magnetic field...). The field geometry in the coils is very similar to the one used in nuclear magnetic resonance : a static high field in the vertical direction B_0, and a horizontal radio-

Neutron resonance spin-echo Spectrometer MUSES G 1 BIS



General set-up of the spin-echo spectrometer G 1 BIS.

frequency field $B_1(t)$ rotating in the horizontal plane. Such a configuration allows measurements at high Fourier times without the need of high magnetic fields. It is particulary interesting for measurements at high angles, because of the difficulty of keeping the field line path in the sample position with conventional NSE option (needs of tunning devices). It allows a very high flexibility with respect to wavevector changes : the resolution function is negligibly angle dependent for a given wavelength.

The neutron beam is polarized by a bender of 4 m length and 76 m radius made out with NiTi super mirrors. A velocity selector roughly monochromizes the incident flux with a wavelength band of δλ/λ ~ 0.1 - 0.15. The polarized flux intensity at the wavelength maximum and at the sample position of the spectrometer MUSES is 10^7 n.cm^{-2} s^{-1} for λ ~ 5 Å, this total integrated flux of the 40 x 40 mm^2 beam at the sample position is ~ 1.6 10^8 n.s^{-1}. Due to the presence of μ-metal shielding, very small Fourier times can be measured (at low current) with NSE option because the depolarization of the beam due to the earth magnetic field or any environmental fields is absent.

Typical studies performed on the instrument are dynamics in liquids and supercooled liquids (in bulk or confined geometries), dynamical studies of soft condensed matter (polymers, colloids...), Biologically relevant systems, critical phenomena, molecular motions in crystals...

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Beam tube	G3 horizontally bent cold guide
Used wavelengths	5 Å - 10 Å (preferred wavelengths 6 - 8 Å)
Monochromation	Mechanical selector
	$\frac{\Delta\lambda}{\lambda} \cong 18\%$ FWHM
Polarizer, analyzer	Supermirrors
	Polarization $P_0 > 92\%$
Focusing guides of the incident collimation	^{65}Cu guides
	Length : 1.8 m and 2 m
Peak intensity at the sample	0.5×10^6 n cm^{-2} s^{-1}
	(Typical size : 27 x 27 mm^2)
Length of precession fields	L = 4 m
Total number of turns	2 478
Precession current	2 A - 140 A
Maximum field integral	0.4 T.m
Spectral resolution	At 8 Å : $h\omega_{min}$ = 1 neV
	Fourier time ~ 40 ns
Sample to analyzer distance	≈ 6 m
Momentum transfert range	$1.5° \leq 2\theta \leq 90°$
	At 6 Å : 0.0274 $Å^{-1}$ < Q < 1.5 $Å^{-1}$
	At 8 Å : 0.0205 $Å^{-1}$ < Q < 1.11 $Å^{-1}$
Detectors	5 ^3He detectors
Ancillary equipment	★ Sample box (3 sample positions) Either fluid heater (- 20°C < T < 80°C) or resistive heater (20°C < T < 120°C)
	★ Furnace (1 sample) (T < 500°C)
	★ Orange cryostat 1.5 K

Neutron Spin Echo (NSE) is a particular technique in inelastic neutron scattering : both the incoming and outgoing neutron velocity (rather given components of these) are measured by using the Larmor precession of the neutron's spin. This technique allows to directly determine the intermediate scattering function, S (Q, t) of the studied sample.
The accessible time range is a few ten nanoseconds (energy transfer of a few neV). This technique is peculiarly well suited to measurements of non-dispersive elementary excitations.

The neutron spin echo spectrometry is a method of wavelength focusing, allowing to use a large energy range of incident neutrons ($\frac{\Delta\lambda}{\lambda}$ ~20% FWHM). This advantage compared to the classical inelastic techniques partly compensates the loss of intensity due to the length of the instrument and to the polarization analysis of the neutron spins.

In the quasi-elastic approximation, the measured quantity, the echo amplitude is proportional to :

$$\int S(Q, \omega) \cos \omega t \, d\omega = \check{S}(Q, t)$$

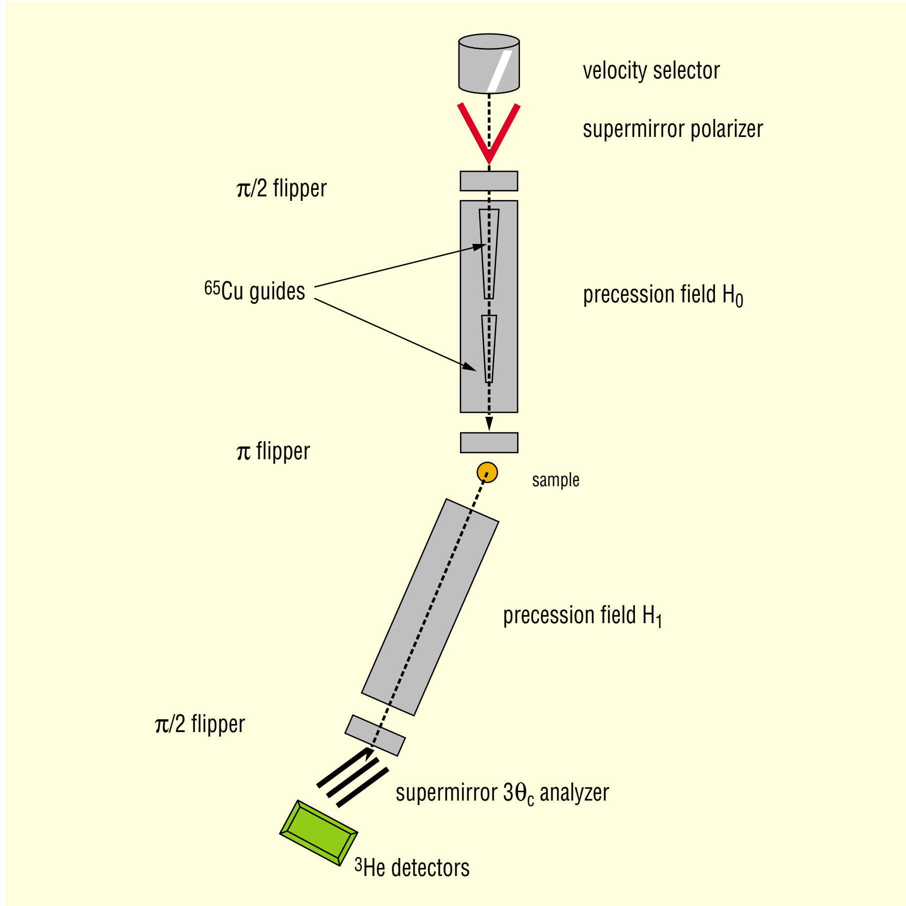
where t, the Fourier time, is expressed as :

$$t_{(sec)} = 1.863\ 10^{-16}. (\int_0^L H.dl). \lambda_0^3$$

H is the field in Oersted and λ_0 (in Å) is the mean incident wavelength. $\int_0^L$ H.dl is the field integral over the length L (in cm) inside the precession solenoids.

Besides the measurement of the echo amplitude, a classical polarisation analysis (three dimensional) can be performed in order to determine the coherent/incoherent contributions in S(Q, ω), to separate magnetic and nuclear signal…
Among the physical phenomena measured with MESS, we can mention :
- internal motion or diffusion of big molecules (biochemistry, polymers, membranes)
- magnetic scattering (paramagnetic, ferromagnetic, spin glass)..

This spectrometer has been built in collaboration with the KFKI (Science Academy Hungary).



General layout of the spectrometer G 3-2.

The neutron beam is roughly monochromatised by a velocity selector (Δλ/λ ~ 18%), then flipper turns the polarization perpendicular to the magnetic field H_0 of the first precession solenoid, so that the Larmor precession will start. The π flipper reverses the polarization so that the fields H_0 and H_1 (in the second precession arm) are in the same direction. After scattering by the sample, the neutron spin precess in the second precession field H1. At the end of the second solenoid, the neutron spin is turned again by the second π/2 flipper, parallel to the magnetic field in order to be analyzed. The spin-echo signal is recorded by several ^3He detectors.
Two guide elements coated with ^{65}Cu can be put in the first precession solenoid. This focusing device allows us to perform lower energy resolution measurements with higher neutron flux.
The whole length of the instrument and the high maximum field integral (0,4 T.m) lead to a high Q and ω resolution spectrometer.

On MESS, the Fourier time is expressed as :

$$t_{(ns)} = 2.341\ 10^{-7}. N_{sol}. I_p. \lambda^3$$

as function of the turn number (N_{sol}), the precession current (I_p) and the incident wavelength (λ).

Data acquisition and treatment are performed on PC computers working with Windows 98 or NT System.

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