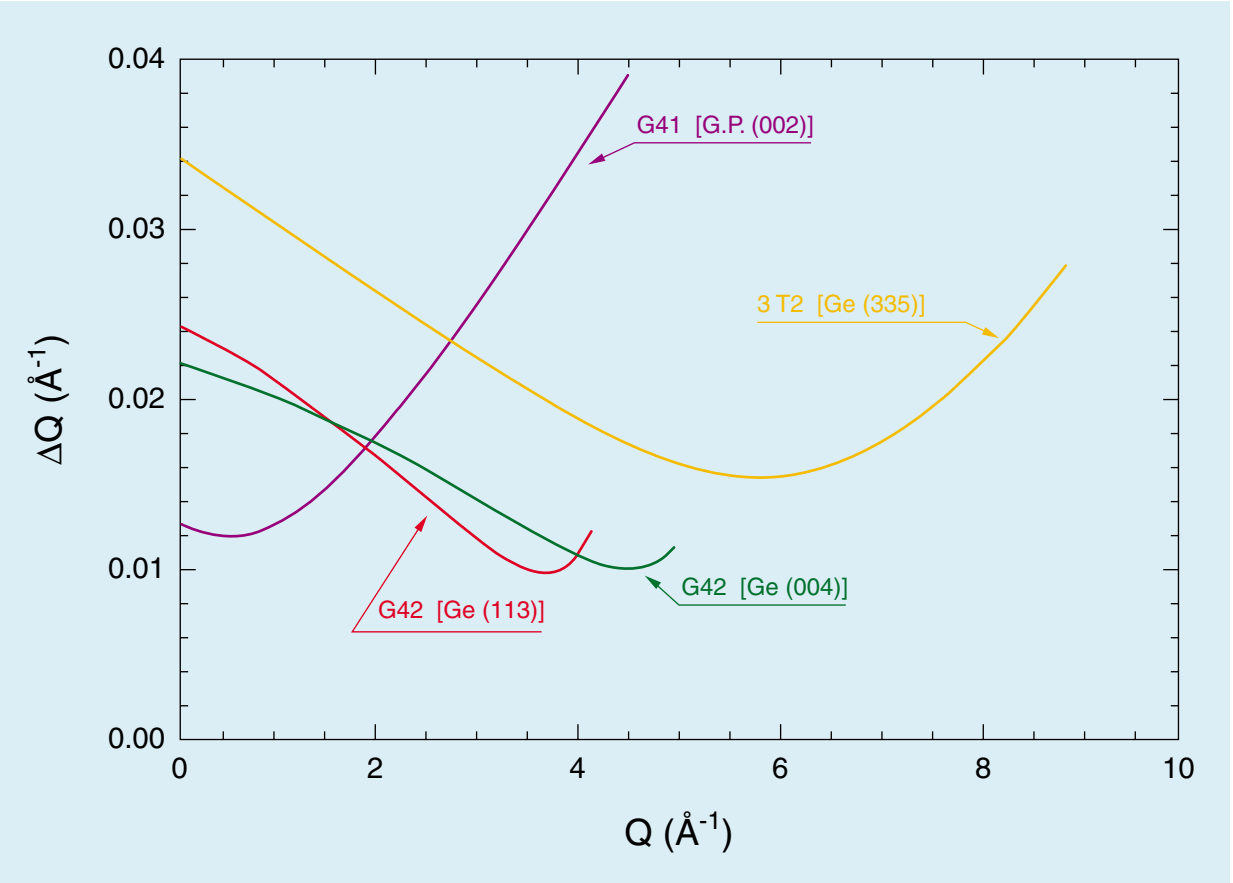
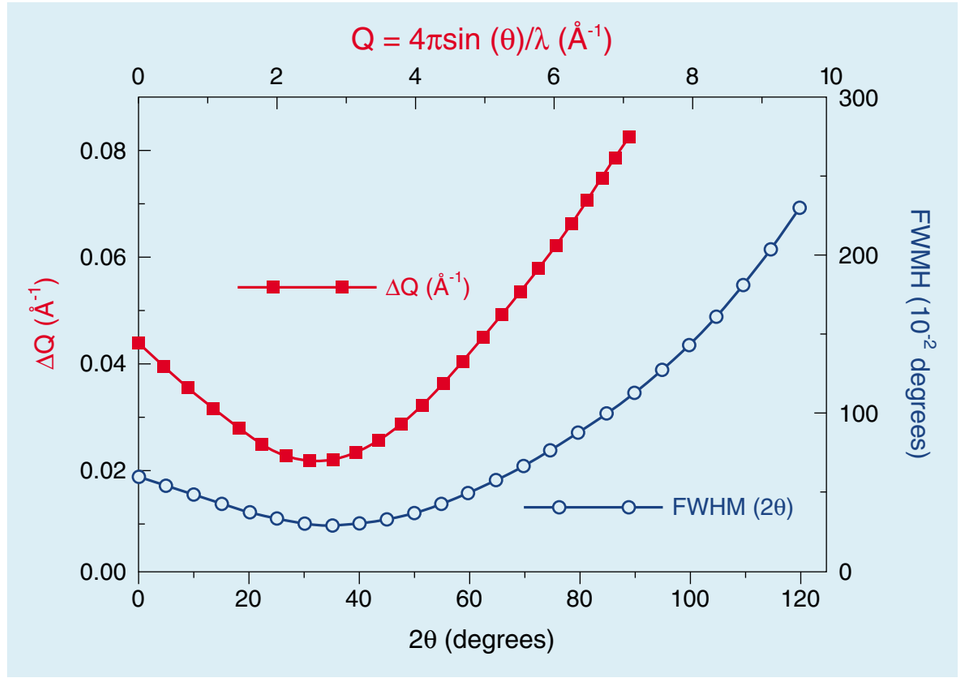
POWDER DIFFRACTION SPECTROMETERS

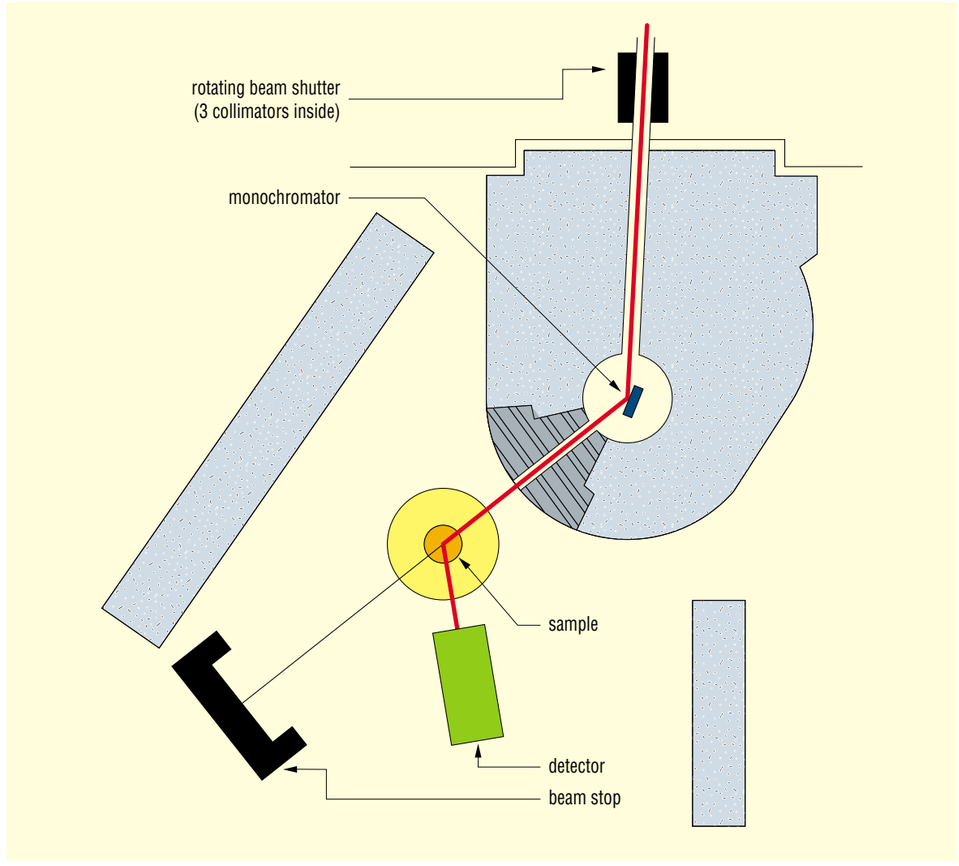


Q domain and resolution for three diffractometer.

Type of instrument	Two-axis diffractometer
Beam tube	Thermal (30 x 80 mm^2)
Monochromators	Cu (111), PG (002),
Maximum flux at specimen	1.5 10^7 n cm^{-2} s^{-1}
Maximum beam size at specimen	20 x 80 mm^2
Incident wavelength	1.5 Å < λ < 2.4 Å λ depends on the choices of monochromator and $2\theta_M$ ($10° \leq 2\theta_M \leq 50°$)
Angular resolution	See figure
Angular range	0° - 120° (2θ)
Collimation	(α_1) : 15', 30', 2°
Detector	^{3}He (α_3= 10' ; 1°)
Minimum step size scan	0.02° (2θ)
Data collection and Instrument control system	PC / SUN
Ancillary equipment	★ Cryostat 1.5 K → 550 K ★ Furnace 100°C → 1000°C



Resolution curves : (○) *Full width at half maximum (FWHM) versus 2θ ;*
(■) *Q variation of the resolution ΔQ (λ_0 = 0.1525 nm).*



General layout of the diffractometer 3 T1.

3 T1 is a two-axis spectrometer dedicated to neutron diffraction studies either on powders or single crystals (a ± 20° rotation of the sample around two orthogonal horizontal axis is available).

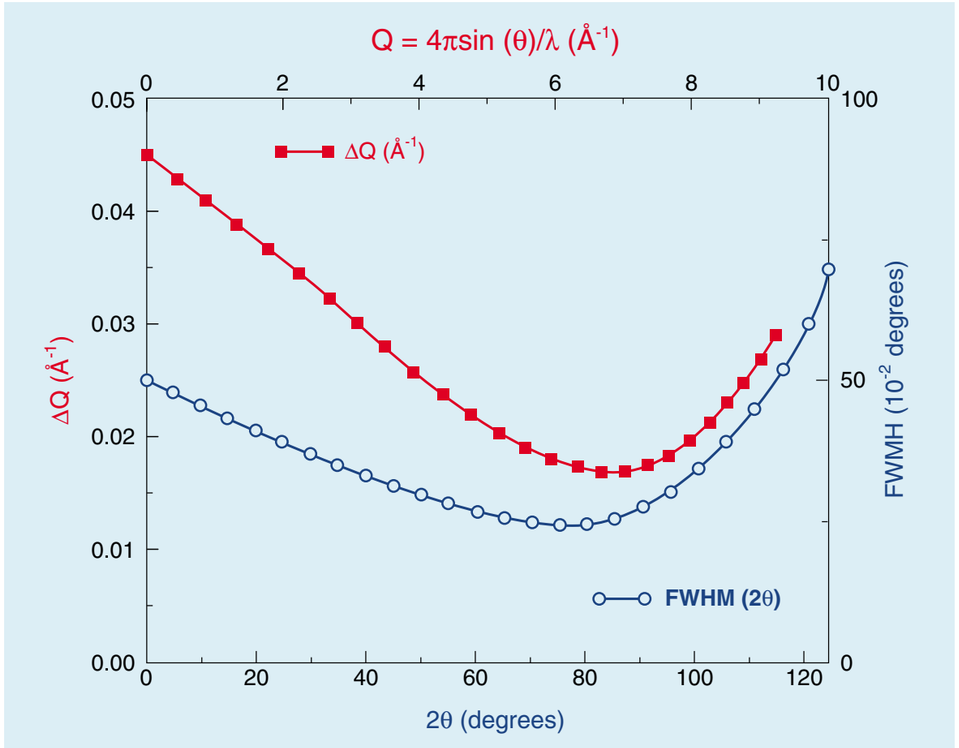
The incident monochromatic wavelength is obtained via two reflecting monochromators :
either : Copper (111)
or : Focusing (002) graphite (β = 24')

Responsible : F. Bourée **email : bouree@llb.saclay.cea.fr**
Co-responsible : B. Rieu **email : rieu@llb.saclay.cea.fr**

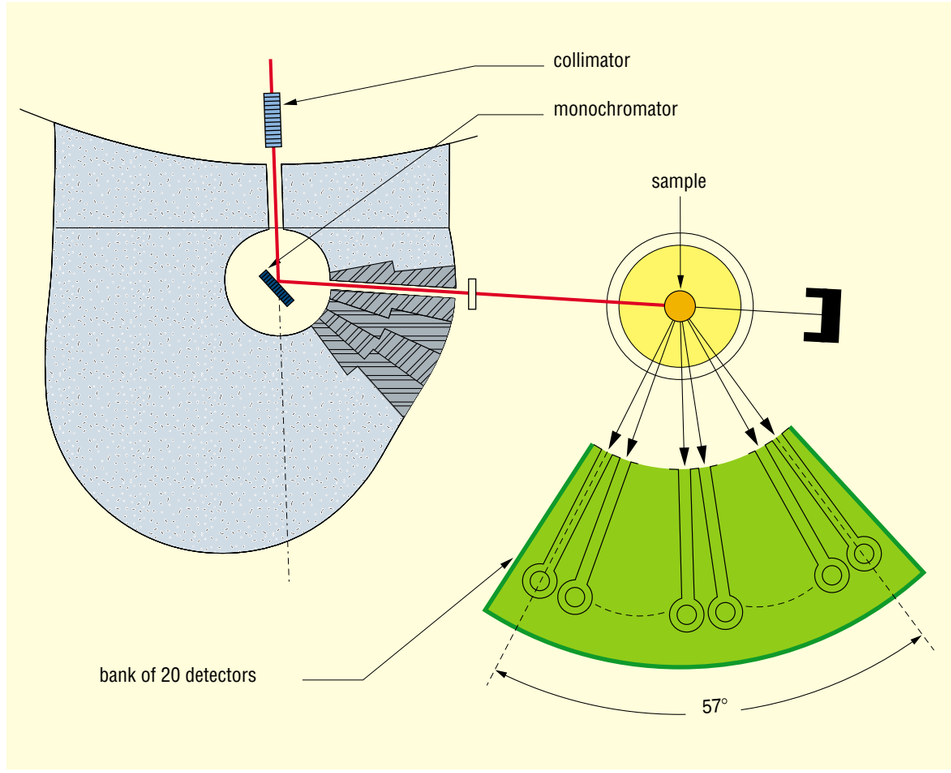
Type of instrument	Two-axis diffractometer
Beam tube	Thermal (30 x 80 mm^2)
Monochromator	Vertically focusing Ge (335)
Maximum flux at specimen	10^6 n cm^{-2} s^{-1} (λ = 1.2 Å ; α_1 = 10')
Maximum beam size at specimen	20 x 50 mm^2
Incident wavelength	1.225 Å ($2\theta_M \approx 90°$)
Angular resolution	See Figure (α_1 = 10')
Angular range	$2\theta < 125°$
Collimation	α_1 variable (10', 14', 21')
	α_3 fixed (10')
Detectors	20 ^{3}He detectors, 3° apart
Minimum step size scan	0.02° (2θ)
Data collection and Instrument control system	PC

Ancillary equipment

★ Cryofurnace (1.5 K - 550 K)
★ Furnace (T ≤ 1000°C)



Resolution curves : (O) *Full width at half maximum (FWHM) versus 2θ ;*
(■) *Q variation of the resolution ΔQ (λ_0 = 0.1225 nm).*



General layout of the diffractometer 3 T2.

3 T2 is a high resolution two-axis diffractometer dedicated to neutron powder diffraction studies in the fields of crystallography, solid state physics, chemistry and material science.

Due to the value of the incident monochromatic wavelength (1.225 Å, thermal neutrons) crystallographic unit cell volumes of the studied samples have to be ≤ 1000 $Å^3$)

Responsible : F. Bourée **email : bouree@llb.saclay.cea.fr**
Co-responsible : B. Rieu **email : rieu@llb.saclay.cea.fr**

G 4-1 Cold Neutron Two-Axis Diffractometer PYRRHIAS,

Type of instrument	Two-axis diffractometer
Beam tube	Cold neutron guide G 4
Monochromator	Pyrolytic graphite (002), vertical focusing
Take-off-angle	$42 < 2\theta_M (°) < 110$
Incident wavelength	$2.43 < \lambda (Å) < 5.5$
Max. flux at specimen	4.10^6 n cm^{-2} s^{-1} (λ = 2.43 Å)
Max. beam size at specimen	10 x 50 mm^2
Detectors	Linear multidetector 800 cells (BF_3)
Minimum step size scan	0.02° (2θ)
Angular range	$3 < 2\theta (°) < 105$
Angular resolution	See figure
Data collection and Instrument control system	PC computer

Ancillary equipment

★ Cryostat 1.5 K < T < 300 K
★ Cryofurnace 1.5 K < T < 550 K
★ Furnace T < 1000°C
★ High (hydrostatic) pressure cell : P < 23 Kbar
★ Vertical magnetic field : H < 1.5 T

G 4-1 is a two-axis powder diffractometer equipped with a vertical focusing pyrolytic graphite monochromator and a 800-cells multidetector covering a 80° - 2θ range (step 0.1° between 2 cells).

The most frequently used wavelength is 2.43 Å but can occasionaly be varied between 2.43 and 5.5 Å. The accessible 2θ diffusion angle covers the range 3° - 105° ; in that range it is possible to perform diagrams with 0.02° step (2θ).

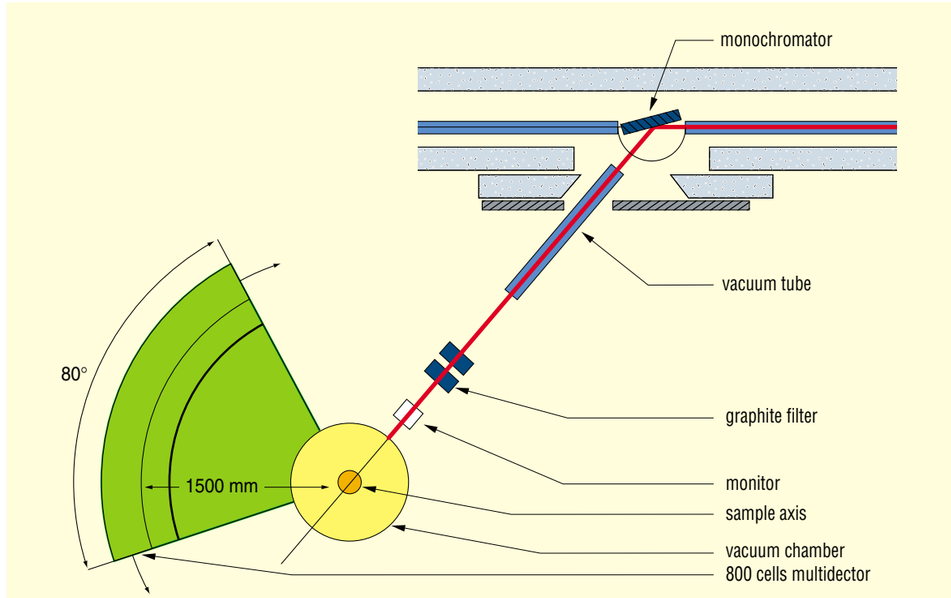
The instrumental resolution of the spectrometer being minimal at low 2θ diffusion angle ($2\theta < 60°$), G 4-1 is particularly well adapted for magnetic structure determination.

The high acquisition rate of the multidetector allows to perform diffraction studies (structural or magnetic) as a function of external parameters (temperature, pressure…) and to follow in situ cinetic reactions or phase transitions ; the minimal acquisition time is of the order of one minute. With longer acquisition time (a few hours) it becomes possible to detect and quantify minority phases present in a multiphase compound, generally down to 0.1% (weight percentage).

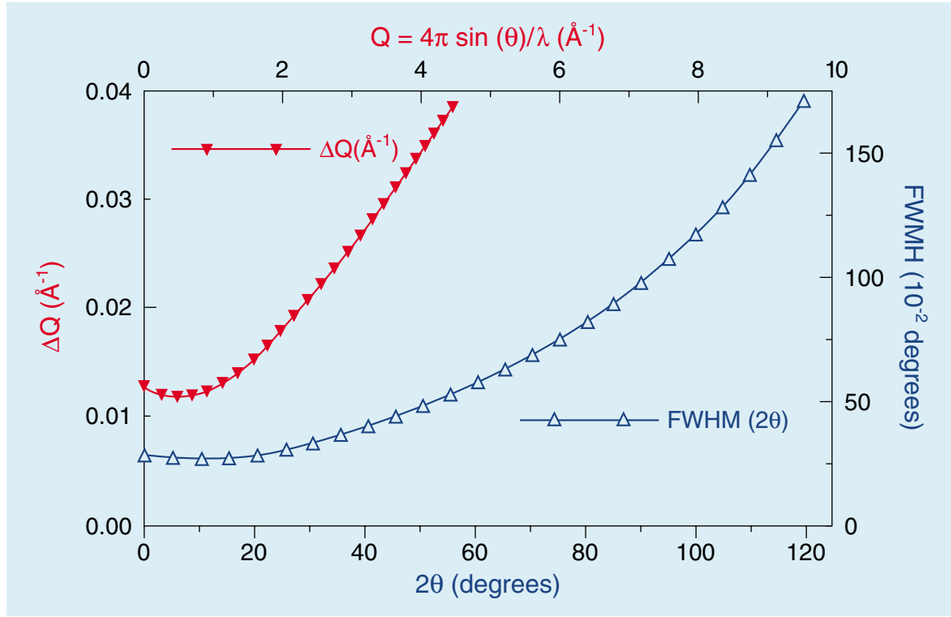
Soon available :

• dilution cryostat down to 50 mK.

Cold Neutron Two-Axis Diffractometer PYRRHIAS, G 4-1



General layout of the cold neutron two axis diffractometer G 4-1.



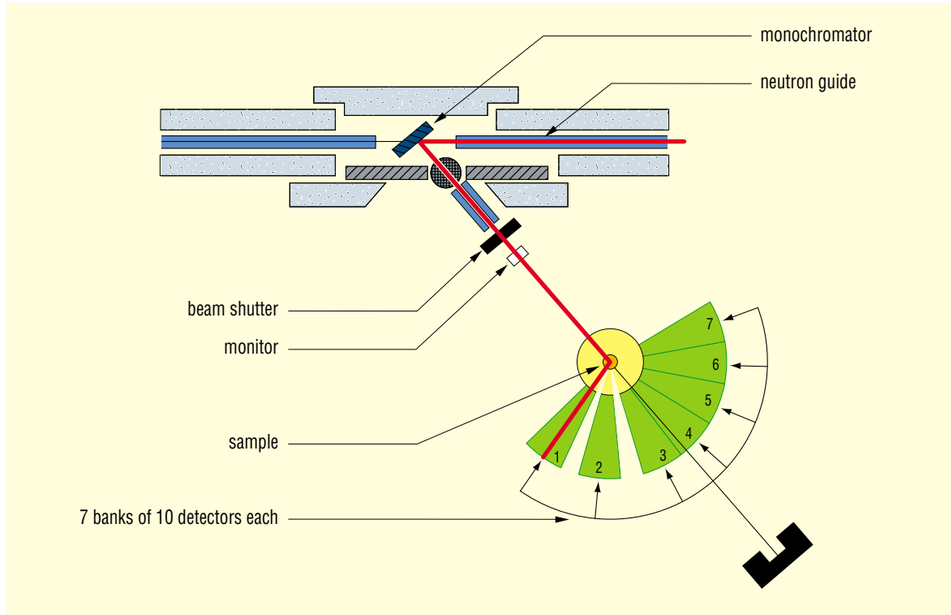
Resolution curves : △ *Full width at half maximum (FWHM) versus 2θ ;*
▼ *Q variation of the resolution ΔQ (λ = 0.245 nm).*

Responsible : G. André **e-mail : gandre@llb.saclay.cea.fr**

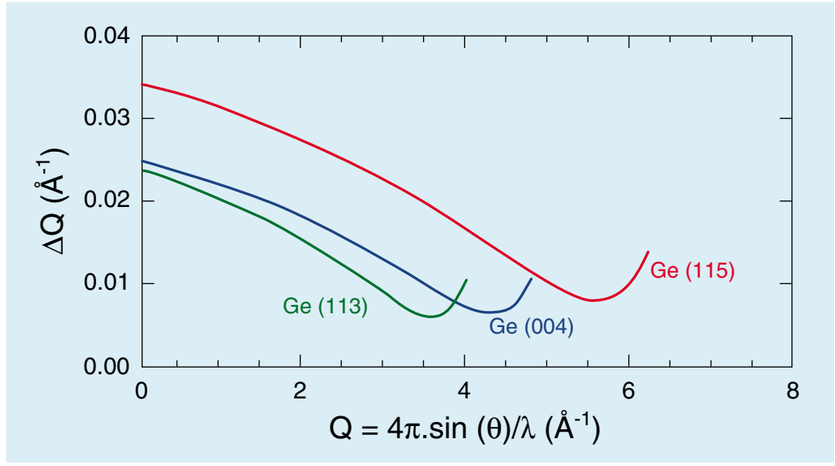
G 4-2 High Resolution Powder Diffractometer

Type of instrument	Two-axis diffractometer
Position	Cold neutron guide G 4, position G 4-2
Monochromator	Focusing Ge ; vertical axis [1 $\bar{1}$ 0] II (115) plane
Take-off angle	$2\theta_M = 112°$
Wavelength	1.80 Å, 2.34 Å, 2.82 Å
Detector system	70 ^{3}He detectors enclosed in 7 sections each containing 10 detectors with a Soller collimator in front of each detector. The angular divergence of the collimators is 13'.
Minimal scanning step (2θ)	0.01°
Working scanning step (2θ)	0.1°
Angular range	$6° < 2\theta < 174°$ ($Q_{max} \approx 7.0$ Å^{-1})
Accessible range of lattice spacing	0.9 Å < d < 27 Å ; 0.12 Å^{-1}< Q < 6.95 Å^{-1}
Best resolution	$\Delta d/d \approx 2.0 \times 10^{-3}$
Data collection and instrument control	PC + CAMAC
Ancillary equipment	★ Cryofurnace : 1.5 K < T < 520 K

This diffractometer was designed and constructed in the Materials Science Research Laboratory of PNPI, Russia. It is installed at the G 4-2 site and has been in operation since january 1997.
The diffractometer is designed for determination and refinement of the crystal and/or magnetic structure of powder materials especially with an elementary cell volume up to 1500 Å^3.
The original feature of the diffractometer consists of the way in which its 70 detectors are arranged, i.e., inside 7 dependent sections (each with a step motor and an absolute position encoder). Each section contains ten neutron detectors with a Soller collimator in front of each detector. The encoder of each section measures the angular position of the first detector in the section with a precision of 18" in the range of 0 - 360°. The other detectors in the section are positioned at 2° from each other. In the stage of preliminary data processing their positions are refined by the angular position of the transmission neutron beam measured by each detector. The sections are set in motion by step motors with the help of three air cushions lifting them over the base surface.
The diffractometer mainly operates in the superposition mode : each part of the neutron diffraction pattern is measured successively by all detectors and the results are added up. The procedure is as follows : the detectors assembly as a whole (all seven sections) starts motion from the initial position at a given step and has the specified monitor counts (or specified exposition time) for each position. At the same time, the smallest technological gap between the sections is preserved. As soon as the first section reaches the specified final position, all sections stop and the transport velocity is switched on, at which the first section moves to the end of the detector assembly. The measuring procedure continues till the second section reaches the final position, then the third, etc… As a result, each detector measures the entire diffraction pattern. In further measurements of the same sample, the movement of the detector assembly can be accomplished in the backward direction. This determines a high luminosity of the diffractometer and considerably simplifies the correction for the efficiency of counting channels and the relative positions of the detectors. Simpler algorithms of operation in the non-superposition mode are also possible. In this case, for calibrating the counting channels and relative positions of the detectors, the efficiency file obtained during measurements in superposition mode is used.
The collimator window is 11 x 110 mm^2, the working length is 200 mm, and the distance between the films is 1 mm ; the collimators are made of a thin polymer film with a gadolinium oxide-based absorber. The mean transmission coefficient measured for all 70 collimators is 85% and the beam divergence is 12 - 13'. The focusing Ge monochromator provides the possibility of using three wavelengths. This allows the diffractometer to be easily adjusted for many physical problems. The neutron monochromator is a set of 11 plates made from a plastically deformed Ge single crystal. The vertical axis is the rotation axis of the monochromator and coincides with the crystallographic direction [1$\bar{1}$0] within an accuracy of several minutes of arc. The plates are arranged one over another in a computer-controlled vertical focusing device which enables precision turning of the plates around the horizontal axis for the monochromator to have the form of a degenerated parabola. Turning of the monochromator as a whole around the vertical axis allows us to use the Ge (115), (004) and (113) reflections in the experiment. At the same time the resolution of the diffractometer changes and the neutron wavelength range changes from 1.8 Å to 2.8 Å. This allows us to optimise the conditions of a particular neutron diffraction experiment.



General layout of the cold diffractometer G 4-2.



Resolution curves.

Responsibles : A. Kurbakov **e-mail : kurbakov@llb.saclay.cea.fr**
J. Rodriguez-Carvajal **e-mail : juan@llb.saclay.cea.fr**

Cold Neutron Two-Axis Diffractometer MICRO

Beam tube ..... Cold neutron guide G 6
Monochromator ..... Pyrolytic graphite, vertical focusing
Type of instrument ..... Two-axis diffractometer (“Tanzboden” type)
Maximum flux at the sample ..... 3 x 10^6 neutrons cm^{-2} sec^{-1} (ambient pressure version)
2 x 10^7 neutrons cm^{-2} sec^{-1} (high pressure version)
Maximum beam size at the sample ..... 10 x 30 mm
Incident wavelength ..... Between 4 and 6 Å
Removal of the λ/2 contamination ..... Beryllium filter
Angular range ..... $0 < 2\theta < 147°$
Detector ..... Linear (BF_3) multidetector (“Banana” 0 type, 80°) with 400 cells

Detector and sample rotations and the data collection are made using a PC computer linked to the central SUN computer for the storage of data. On-line date treatments can be performed by a second PC computer installed near the first one.

Sample environment

Low temperatures ..... Cryostat (ILL type) : $1.5 < T < 300$ K
Closed circle cryogenerator : $10 < T < 300$ K
High temperatures ..... Small furnace which replaces the inner part of the cryogenerator : $T < 300$ C
High temperature furnace $T < 1300$ C
Magnetic fields ..... No

High pressure ..... Sapphire anvil cells $P < 10$ GPa
Diamond anvil cells $P < 50$ GPa

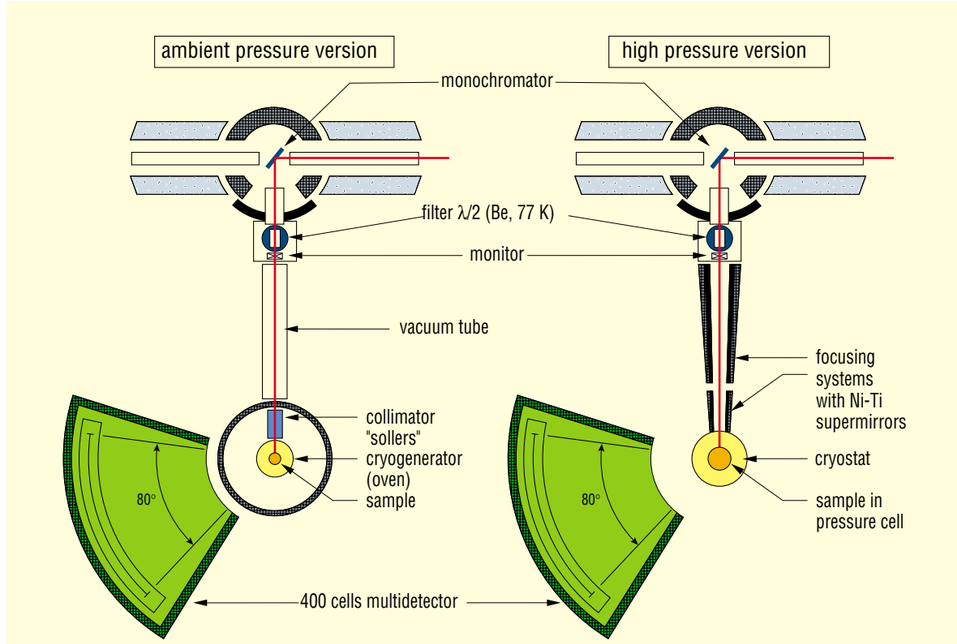
Note : all the pressure cells are compatible with the ILL-type cryostat. Pressure is applied outside of the spectrometer in the neighboring building. Pressure is measured by a ruby fluorescence at room temperature and kept constant during cooling.

G 6-1 is a two-axis powder diffractometer installed on a cold neutron guide. It is a high-intensity long-wavelength ($4 < \lambda < 6$ Å) instrument used to study magnetic structures, long-periodicity mesoscopic structures and diffuse scattering at ambient and high pressures. A monochromatic neutron beam is selected by a graphite monochromator. The typical incident wavelength is 4.74 Å and can be exceptionally changed in the above range. The contamination of the second harmonics (λ/2, λ/3,…) is suppressed by inserting a beryllium filter (cooled down to liquid nitrogen temperature) in the incident beam path. The diffractometer is equipped with a linear (banana-type) 400-cells multidetector covering 80 degrees of scattering angle. The multidetector and its protection can rotate around the sample axis, covering a total angle of 150 degrees.

The spectrometer operates in two different versions :

1) ***ambient pressure version***
In this version, the incident neutron path is under vacuum. The sample, collimator and beam catcher are placed inside a large vacuum chamber. In this vacuum chamber, several types of sample environment may be insered. Namely a cryogenerator ($10 < T < 300$ K), an ILL type cryostat ($1.5 < T < 300$ K), a small furnace ($T < 300$ C) easy to install with the same environment as the cryogenerator, or a more powerful furnace ($T < 1300$ C) similar to that used on 7 C2 diffractometer. An electromagnet could be also installed ($H < 1.5$ Tesla) but the instrument is not currently equipped with it. The option of polarized neutrons is now suppressed.

Cold Neutron Two-Axis Diffractometer MICRO
G 6-1



General layout of the diffractometer G 6-1.

Due to its high flux and resolution, but limited scattering vector range ($0.15 < q < 2.5$ $Å^{-1}$ in the typical configuration), this diffractometer is well adapted to the study of structural and magnetic phase transitions, diffuse scattering in disordered systems, specific features at low q's in amorphous and liquid states, adsorbed layers, etc…

2) ***High pressure version***
In this version the diffractometer is used to study magnetic order, phase transitions, mesoscopic structures (nanomaterials, polymers,…) in wide range of pressures $0 < P < 50$ GPa.
To study small samples at very high pressures a special double-stage focusing system with Ni-Ti supermirrors is installed along the incident beam path. The focusing system allows to increase intensity at the sample place by a factor of 7 (with some reduction of angular resolution). The focusing angle in the horizontal plane can be varied allowing to choose an optimal ratio between intensity and resolution in each experiment. Additional protection is used to decrease background in experiments with small samples.
The diffractometer is equipped with various high-pressure cells with sapphire and diamond anvils. Available sample volume : from several mm^3 (sapphire anvils) down to 0.01 mm^3 (diamond anvils). The maximal pressures are up to 50 GPa (500 Kbar) for strongly scattering samples and 7 - 10 GPa (70 - 100 Kbar) for normal scatterers. A specially modified cryostat generates temperatures in the range $1.5 < T < 300$ K. Currently, there is no high-pressure high-temperature option.

Responsibles :
Ambient pressure version : I. Mirebeau **e-mail : mirebea@llb.saclay.cea.fr**
High pressure version : I. Goncharenko **e-mail : gonch@llb.saclay.cea.fr**