1.1 Neutron Scattering Science in Australia

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Abstract

Neutron scattering science in Australia is making an impact on a number of fields in the scientific and industrial research communities. The unique properties of the neutron are being used to investigate problems in chemistry, materials science, physics, engineering and biology. The reactor HIFAR at the Australian Nuclear Science and Technology Organisation research laboratories is the only neutron source in Australia suitable for neutron scattering science. A suite of instruments provides a wide range of opportunities for the neutron scattering community that extends throughout universities, government and industrial research laboratories.

Plans are in progress to replace the present research reactor with a modern multi-purpose research reactor to offer the most advanced neutron scattering facilities. The experimental and analysis equipment associated with a modern research reactor will permit the establishment of a national centre for world class neutron science research focussed on the structure and functioning of materials, industrial irradiations and analyses in support of Australian manufacturing, minerals, petrochemical, pharmaceuticals and information science industries.

The Instruments

The present supported instruments located on the HIFAR reactor diffractometers (2tanA, 2tanB), a polarised beam facility (LONGPOL), two powder diffractometers (HRPD, MRPD) and a small angle neutron scattering (SANS) instrument. Except for 2tanA and 2tanB, instruments are located on radial beam tubes. The beam tubes have a diameter up to 25cm, and terminate either in the D_2O moderator or the graphite reflector regions of the reactor core. This has an impact on beam quality and has influenced instrument design. Table 1 is a summary of instrument characteristics and Figure 1 illustrates their disposition on the experimental floor.

The two single crystal diffractometers share one end of a 'tangential' beamline that passes under the reactor core. The High Resolution Single Crystal Diffractometer (2tanA) has recently undergone an upgrade. The goniometer has been replaced with a four circle device of standard design with all motions interfaced to locally developed instrument control software on an IBM-PC. A small 2D position sensitive detector (PSD) with single reflection capability has been installed, and the primary shielding and optics will be upgraded. A copper crystal at a take off angle of 58° produces the 1.235Å neutron beam. The 10mm diameter neutron beam has a flux of $\sim 5 \times 10^5 \mathrm{cm}^{-2}\mathrm{sec}^{-1}$ at the sample position with good signal to background characteristics. The maximum 2θ is ~118° and over this range the reflection widths are typically ~0.4° (fwhm) (2θ) at 5° (2θ) increasing to ~0.75° at 105°. The major purpose of the instrument is high resolution structure determination, particularly organic molecules with a modest number of atoms.

The Medium Resolution Single Crystal Diffractometer (2tanB) is a four circle instrument with a single BF_3 detector. A pyrolytic graphite monochromator provides a 10mm diameter 1.239Å neutron beam at a take off angle of 21°, with a flux of $\sim 10^6 \mathrm{cm}^{-2}\mathrm{sec}^{-1}$ at the sample position. The reflection widths are typically ~0.4° (fwhm) (2θ) at 5° (2θ) increasing to about 2° at 90° with a maximum 2θ of ~120°. The major purpose of the instrument is structure determination particularly where diffraction intensity is limited and/or where a small 2θ range provides the required information eg magnetic and larger molecule structures. The instrument can also be used to investigate texture and diffuse scattering from disorder in metals, alloys, ceramics, polymers etc.

The Long Wavelength Polarised Neutron Spectrometer (LONGPOL) (Figure 2), the most novel instrument on HIFAR, is a diffractometer/spectrometer for diffuse and inelastic scattering measurements incorporating both neutron polarisation analysis and energy analysis[1]. The instrument operates at a wavelength of 3.6Å and is equipped with 8 ^{3}He detectors. Saturated iron filters are used to polarise the neutron beam before the sample (polarising efficiency ~40%) and to analyse the polarisation after the sample. Polarisation analysis allows separation of magnetic scattering events from nuclear scattering events, and also allows isolation of nuclear-spin-incoherent scattering, which can be used to determine hydrogen concentration, for example. The neutron flux at the sample position is $\sim 3 \times 10^4$ $\text{cm}^{-2}\text{sec}^{-1}$ in a low resolution configuration that makes the instrument ideally suited to neutron diffuse scattering studies. Main applications include determination of atomic and magnetic distributions in magnetic materials, measurement of hydrogen concentration in bulk materials, measurements of paramagnetic scattering, magnon measurements, neutron depolarisation studies, and flux creep in superconductors[2]. The spectrometer is under constant development with recent improvements including installation of a new time-of-flight data acquisition and instrument control system developed at ANSTO, and a rapid response neutron spin flipper developed at Monash University.

Installation of high efficiency polarising supermirrors developed in collaboration with the Hahn-Meitner Institute is now complete. The incident polarisation of the neutron beam is now 96% - improved from 40% by the installation of a supermirror bender, made to ANSTO's design at the Hahn-Meitner Institute (Berlin), and supported by Australian Research Council Research Infrastructure Funds. The supermirror benders on single crystal silicon substrates are the latest in neutron polarisation technology. The next stage in this development is to install the polarising supermirror benders that are required to analyse the neutron spin orientation after scattering from a sample. Possible configuration options for the analyser benders have recently been investigated and the favoured prototype mount has been manufactured. This stage will unfold over the next twelve months, delivering a further factor of >4 in performance. These improved characteristics increase the quality and quantity of data available from this instrument, which has special applications in the areas of magnetic order, flux dynamics in high temperature superconductors and crystal field studies.

The two powder diffractometers (Figure 3) were designed specifically to accommodate the conflicting requirements of neutron intensity and resolution[3]. The neutron flux at the sample position on the Medium Resolution Powder Diffractometer (MRPD) is typically five times that of the High Resolution Powder Diffractometer (HRPD) and the resolution is about half. This enables rapid phase transition type patterns to be collected on the MRPD and high resolution structure patterns on the HRPD. The HRPD has a selection of two Soller collimators before the monochromator (0.16 and 0.25°), a take-off angle of 120° and uses a germanium monochromator. Routine wavelengths are 1.371, 1.493 and 1.8834Å. The beam has dimensions 20mm wide by 50mm high and the neutron flux at 1.8834Å is approximately $8 \times 10^4 cm^{-2} sec^{-1}$. Patterns are collected from 5 - 156° (2θ) in a bank of 24 detectors (5° apart), with each detector having its own high efficiency collimator (0.17°). The minimum peak width is 0.25° and the width is less than 0.4° over most of the pattern. Data collected on this instrument is primarily for structure determination and quantitative phase analysis by multiphase Rietveld refinement of known components.

The collimation of the primary neutron beam on the MRPD can be either 0.25 or 0.5°. The monochromator consists of an array of 8 germanium single crystals with total height 80mm and width of 50mm, with vertical focussing to increase the neutron flux at the sample position to $\sim 10^6 cm^{-2} sec^{-1}$. Currently, 32 ^3He neutron detectors are mounted at 4° spacings at a distance of 0.7m on the 2θ drive. Each detector is mounted down-beam from a Soller collimator that has an acceptance angle of 0.35°. The instrument is designed for neutron powder diffractometry, magnetic structure determination, phase transition and residual stress measurements. *In-situ* kinetic studies of structure transformations, hydration mechanisms etc down to time resolution of ~15 minutes are also possible.

The Small Angle Neutron Scattering (SANS) instrument (Figure 4) has a 5m collimation length and sample-to-detector distance range of 1.5 - 5m. The monochromator and first section of the collimator are located within the reactor containment building, and the second section of the collimator, the sample position and the detector system are located in an external laboratory. The monochromator is a double multilayer system based on a design developed at the Brookhaven National Laboratory (BNL)[4,5]. At present the multilayer monochromators are single d-spacing, planar geometry used in reflection mode, and d-spacing in the range 40 - 120Å will permit the selection of λ and $\Delta\lambda/\lambda$ over a considerable range. Geometric focussing and planned developments in multilayer technology will improve the neutron flux at the sample position.

The large 64 × 64cm^2 active area PSD (Figure 5) has been designed in collaboration with BNL and the ILL, and constructed at ANSTO[6]. The detector chamber has been developed along principles established at BNL, the event readout system is based on the wire-by-wire method and the Proportional Chamber Operating System (PCOS) (LeCroy Inc USA) is used for event encoding. The detector is mounted in a vacuum tank that can be rotated +5 to -30° in 2θ to expand the accessible q range. The sample position provides a range of environments including a computer controlled sample changer that can operate *in vacuo*, or in the laboratory atmosphere. Since the instrument is located on a thermal neutron source, the flux is modest particularly at longer wavelengths. Nevertheless, considerable useful work can be undertaken on samples with high contrast and large physical dimensions.

Of course, no suite of neutron scattering instruments is complete without a comprehensive range of ancillary devices to supply special sample environments. These devices include closed cycle helium refrigerators that allow sample temperatures in the range from 3.6K to room temperature, and furnaces that operate between room temperature and 1700C. Magnetic fields of up to 1T, a range of specialised gas environments and a range of thin walled chambers of aluminium or vanadium are also available. In addition, for the powder diffractometers there is a 20 tonne axial compression/tension rig, and a 30 position automatic sample changer. All ancillary devices are computer controlled, allowing a flexible data collection regime without user intervention.

In addition to this hardware, there is a considerable investment in software development partly to optimise the performance of the unique combination of instrument and computer hardware on HIFAR. All instruments are controlled by IBM-PC's except for a UNIX workstation for data acquisition, display and analysis on the SANS instrument. All control computers are attached to a local site network (ANSTOnet) to facilitate data transfer and to connect to remote users through an extended computer network. A number of medium to large UNIX computers are also available on ANSTOnet for data analysis and storage.

For neutron scattering instruments at ANSTO, the immediate future is expected to mirror the immediate past with upgrading of existing instruments on a priority basis. In terms of expansion, perhaps an instrument that would best complement the present suite is a dedicated reflectometer. Plans for such a device are being considered.

The Science

International scientific assessments rate the neutrons produced by research reactors as a unique and broadly applicable scientific tool for leading edge, basic and applied investigations across a wide range of scientific and technological disciplines in physics, chemistry, biology and medicine. Because neutrons probe in a non-destructive way, they are particularly suited for investigating the microstructure and properties of existing solid and liquid materials and of emerging advanced materials in the aerospace, automotive, biotechnology, petrochemical and telecommunications fields. As each new class of materials (eg, high-temperature superconductors, carbon-cage "fullerene" molecules) has been developed, neutrons have been the primary tool for studying the properties and understanding the behaviour.

Whilst opportunities exist to pursue scientific ideas of a fundamental nature, in general, the neutron scattering science in Australia is problem driven which has led to a diverse array of activities involving multi-disciplinary teams. Consistent with ANSTO's mission to contribute to the vitality and competitiveness of Australian industrial research and development, the neutron scattering effort has a focus on science that support this mission. Recent studies of practical significance include investigation of the hydrogen storage capacity of metal hydrides; quantitative phase analysis of structural ceramics; studies of rare-earth and transition metal ferromagnets; residual stress studies of welded steels; structural and magnetic studies of superconductors; studies of magnetic materials; and structural studies of fullerenes. The following are indicative of recent activity on HIFAR.

- The study of magnetic structure and magnetic moment distributions in antiferromagnetic, ferrimagnetic, ferromagnetic, superparamagnetic and spin glass metals, alloys, mixed oxides etc continues to be a productive research theme on HIFAR[7]. Recent time-of-flight measurements on LONGPOL of polycrystalline $PrAl_3$ sample, have highlighted the utility of polarisation analysis to distinguish crystal field transitions from other low energy inelastic scattering events[8].

- The interstitial distribution and site occupancy of hydrogen/deuterium in metals and alloys. is important in the understanding of the application as a hydrogen storage media, and in rechargeable battery construction. For example, time resolved powder patterns of the phase transition induced in $LaNi_5$ by the passive diffusion of D_2 gas (at a pressure of 1MPa) into the lattice structure, have been obtained. The analysis has provided information on anisotropic strains in the lattice structure[9]. Extensive studies on palladium hydride have also been undertaken[10].
- Residual strain measurements in metals, alloys, ceramics and composite materials are of direct interest to industry for examining welds and fabrications to improve quality and durability of end products. As an example, the width and position of peaks in powder patterns from zirconia-toughened aluminas were used to investigate (i) the strain broadening of the alumina matrix as a function of zirconia content, and (ii) the tensile strains in tetragonal zirconia induced by thermal contraction mismatch[11].
- The detailed structure and phase composition of transformation toughening ceramics, particularly zirconia ceramics, have been intensively investigated[12]. There have been a number of highlights. The characterisation of the orthorhombic phase of zirconia in magnesia-partially stabilised zirconia (Mg-PSZ) is one notable result[13,14]. Further, analysis of powder diffraction data supported the model of substantial quantity of δ-phase ($Mg_2Zr_5O_{12}$) contributed to the observed properties of high toughness Mg-PSZ[15].
- There have been a number of studies of high T_c superconductor materials. For example, inelastic neutron scattering has been used to study the lattice dynamics of $YBa_2Cu_3O_6$[16] and obtain the free parameters of shell model based calculations and the results extended to $YBa_2Cu_3O_7$. The magnetic superconducting properties have been investigated on a number of systems using the neutron depolarisation method on LONGPOL.
- Neutron diffraction has been used to study, at atomic resolution, the incorporation of various radioactive waste elements into the crystal structures of the components of the synthetic rock, synroc[17]. Synroc could have a major impact on the storage of high level radioactive waste on a global scale[18]. In another study, SANS contrast-variation

techniques were used to investigate the local oxide ultrastructure in TiO_2/ZrO_2 synroc precursor sols[19].

- A more detailed understanding of the microstructure of cement paste will lead to a more detailed understanding of the durability of cement paste in concrete. The SANS spectra of hydrating cement paste has been measured *in situ* and studied as a function of hydration time, and correlated to the quantity of heat generated[20,21]. Further, *in-situ* changes to the composition of hydrating cement have been monitored by neutron powder diffraction. The rate of composition change is used as input to models for the hydrating cement process that give a greater understanding of the pore structure of cement paste. The three-dimensional microstructure controls the rate at which water can penetrate the paste, and it dominates the rate at which radioactive ions leach from cements in conditioned low level nuclear wasteforms.
- A number of studies in structural biology have been attempted and two instruments were modified primarily for this purpose. Highlights of this work include the study of ion channels and anaesthetics in reconstituted membranes; the structure of membrane fragments containing NaK/ATPase; the high resolution structure of selected drugs and of the protein plastocyanin; and general studies on biomolecular hydration[22].

As an illustration that science has little regard for geographical boundaries, considerable science is being undertaken by Australian scientists at neutron sources around the world. Over recent years surfaces, surfactants, ceramics, cements, hydrides, superconductors, polymers, catalysts, and neutron optics have been undertaken in collaboration with colleagues in almost every neutron scattering centre in the world. In a recent review of the international projects, almost 60% of the funded projects required access to SANS and reflectometry instruments, and about 10% each to inelastic, single crystal, powder and polarised neutron facilities.

The Neutron Scattering Community

The neutron scattering community in Australia is distributed throughout universities and government research organisations, but understandably the focus is on ANSTO. Within this context, neutron scattering is identified as a national facility and this environment influence funding for its continued use and expansion. ANSTO is a vital component of the neutron scattering enterprise in Australia and makes a major contribution to the infrastructure in terms of the efficient operation and maintenance of the neutron source HIFAR; the staff to support active scientific programs; the staff to develop and exploit neutron scattering instruments and; the capital funds for equipment development and maintenance; and the access on a priority basis to a comprehensive range of support facilities.

ANSTO's capability in neutron scattering science brings direct benefit to a number of internal programs as well as to programs driven by external partners. The externally driven science programs are accomplished, in part, by collaborating with ANSTO scientists and, as a result of a competitive review process, are allocated beamtime on a particular instrument. The competitive review process for beamtime involves representatives of all users groups including the Australian Institute for Nuclear Science and Engineering (AINSE). AINSE is a consortium of 28 Australian and New Zealand universities in partnership with ANSTO, and was established by the Australian Government in 1958 to provide a mechanism for access to all the special facilities at ANSTO by universities and other tertiary institutions.

In general, the neutron scattering instruments have been developed with input from ANSTO and AINSE, and certain instruments have been developed in close collaboration with user groups from universities, with financial assistance from the Australian Research Council. In addition, through a program of grants, studentships and fellowships, AINSE provides research students with opportunities to use neutron scattering techniques, thereby ensuring an expanding future for the field. More than 8,000 hours of research time are provided to university researchers each year. Approximately 15% of PhD candidates in Australia in the physical sciences and engineering use HIFAR for research.

Many Australian neutron scattering scientists are integrated into the international community through long term collaborations involving science and instrument development; active membership of learned societies and science/policy committees; and regular attendance at international conferences.

The Future

The Australian Government has decided that HIFAR will be replaced to ensure that Australia retains the capabilities to produce its own medical and industrial radioisotopes, to conduct nuclear based research and to maintain the first hand ability to remain abreast of international and regional nuclear developments and regulation. The decision to replace the HIFAR research reactor by the year 2005 will opened up exciting new opportunities for Australia's capabilities in neutron scattering science, nuclear medicine, environmental science, education and industrial support. The advanced neutron source will facilitate research and development relating to, for example, polymers, ceramics and other new materials, life sciences and biotechnology, understanding complex industrial processes, advanced therapeutic treatment strategies with radiopharmaceuticals, and advanced environmental management processes.

The total project will including reactor island, all associated infrastructure and buildings. It will incorporate modern instrumentation and enhanced experimental access, high intensity neutron beams, cold and hot neutron sources. Since cold neutron sources provide the basis for many of the current advances in neutron science and technology, the replacement reactor with the most advanced cold neutron source will enable Australia's basic and applied research scientists to enter new areas of endeavour. An example is the field of nanotechnology, which requires multidisciplinary application of knowledge in physics, chemistry, mathematics, biology and electronics and where science and engineering converge at the level of individual atoms. The developments in nanotechnology will require access to intense sources of neutrons to probe the most minute structures of materials.

In order to ensure that the neutron scattering instruments will be the most appropriate for the Australian scientific and industrial research community, a consultative group was formed to identify present and future research priorities. As a consequence of extensive deliberations a suite of instruments was formulated. Table 3 lists the range of instruments considered most likely to facilitate the priority research science areas, and Figure 6 is an appropriate schematic layout of the suite of neutron scattering instruments located on a replacement research reactor. The neutron scattering facilities will be built up from the existing expertise and equipment base. When the replacement reactor commences operation in 2005 it is anticipated that four newly developed instruments will be relocated from HIFAR and four new instruments will have been built. Some of these instruments will be located on cold neutron beam guides. Three more instruments will be built in the first 5 years' of operation.

Summary

Neutron scattering science is an important, integral component of the scientific and industrial research community in Australia. The present neutron source, HIFAR, and the attached suite of neutron scattering instruments facilitate research programs of a very high standard. The replacement research reactor due to be commissioned in 2005 will build on the strengths developed on HIFAR as well as offer new opportunities particularly in cold neutron scattering science. The combination of four decades of experience and a modern research reactor will assure ANSTO's place as a national and regional centre for neutron scattering science.

Acknowledgements

This brief review presents the work of an impressive list of individuals, groups and organisations. The strength and vitality of neutron scattering science is due to the enormous contributions made by the neutron scattering community that extends into Australian universities, government and industrial research laboratories, and our colleagues in many institutes and laboratories around the world. The author wishes to acknowledge the many valuable contributions made by all neutron scattering scientists in Australia and our colleagues overseas.

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Table 1: Characteristics of neutron scattering instruments located on HIFAR

Instrument		**Monochromator**	**Detectors**	**Neutron Wavelength (Å)**	**Maximum Neutron Flux ($cm^{-2}sec^{-1}$)**	**Resolution**	**Beam size (mm)**
2tanA	High Resolution Single Crystal Diffractometer	Cu single crystal	1 (BF_3) (2D (16^2 elements) ^3He)	1.235	6×10^5	0.4° at 5° (2θ) 0.2° at 50° (2θ) 0.75° at 105° (2θ)	10 diameter
2tanB	Medium Resolution Single Crystal Diffractometer	pyrolytic graphite	1 (BF_3)	1.239	10^6	0.4° at 5° (2θ) 2° at 90° (2θ)	10 diameter
LONGPOL	Long Wavelength Polarised Neutron Spectrometer	pyrolytic graphite	8 (^3He)	3.6	3×10^4	$1 \leq \Delta E \leq 10meV$ $0.3 \leq q \leq 3.0Å^{-1}$	30(H)×20(V)
MRPD	Medium Resolution Powder Diffractometer	Ge multiple single crystals	32 (^3He)	1.06 -5.0	10^6	0.4° - 0.8° (2θ)	20(H)×50(V)
HRPD	High Resolution Powder Diffractometer	Ge single crystal	24 (^3He)	1.2 - 2.26	8×10^4	0.25° - 0.4° (2θ)	20(H)×50(V)
SANS	Small Angle Neutron Scattering Instrument	multilayer	2D (128^2 elements) (^3He)	2.0 - 8.0	$\sim 10^4$	$0.05 \leq q \leq 0.1Å^{-1}$	40(H)×50(V)

Table 2. Comparison of relevant features for the HIFAR reactor and the replacement research reactor.

Feature	**HIFAR reactor**	**Replacement research reactor**
Reactor power heat output (MW)	10-15	14-20
Neutron Flux (× 10^{14} n cm^{-2} s^{-1})	1	At least 3
Number of fuel elements	25	#
Fuel enrichment (% uranium-235)	60%	20%
Fuel load (kg uranium-235)	7	#
Core	Loose array of fuel elements	Compact array of fuel elements
Spent fuel elements a year	37	#
Coolant	D_2O	H_2O
Reflector	D_2O	D_2O
Experimental positions*	11	17 (max)
Neutron guide hall	No	Yes
Beamline geometry	Radial	Tangential
Cold source	No	Yes
Hot source	No	Yes

Notes:

* for neutron scattering instruments

dependent upon design

Table 3. List of neutron scattering instruments proposed for the replacement research reactor.

	Instrument
1	Small Angle Neutron Scattering (SANS) Instrument (30 metre)
2	Horizontal Neutron Reflectometer
3	High Intensity Powder Diffractometer
4	High Resolution Powder Diffractometer
5	Polarisation Analysis Spectrometer
6	4-Circle Diffractometer
7	Quasi Laue Diffractometer
8	3-Axis Spectrometer
9	High Resolution Backscattering Spectrometer
10	Amorphous Materials Diffractometer
11	Residual Stress Diffractometer
12	Radiography Station
13	Small Angle Neutron Scattering Instrument (6 metre)
14	Neutron Spin Echo Spectrometer
15	Vertical Neutron Reflectometer
16	4-Circle Diffractometer
17,18	Thermal and Cold Neutron Instrument Development Stations

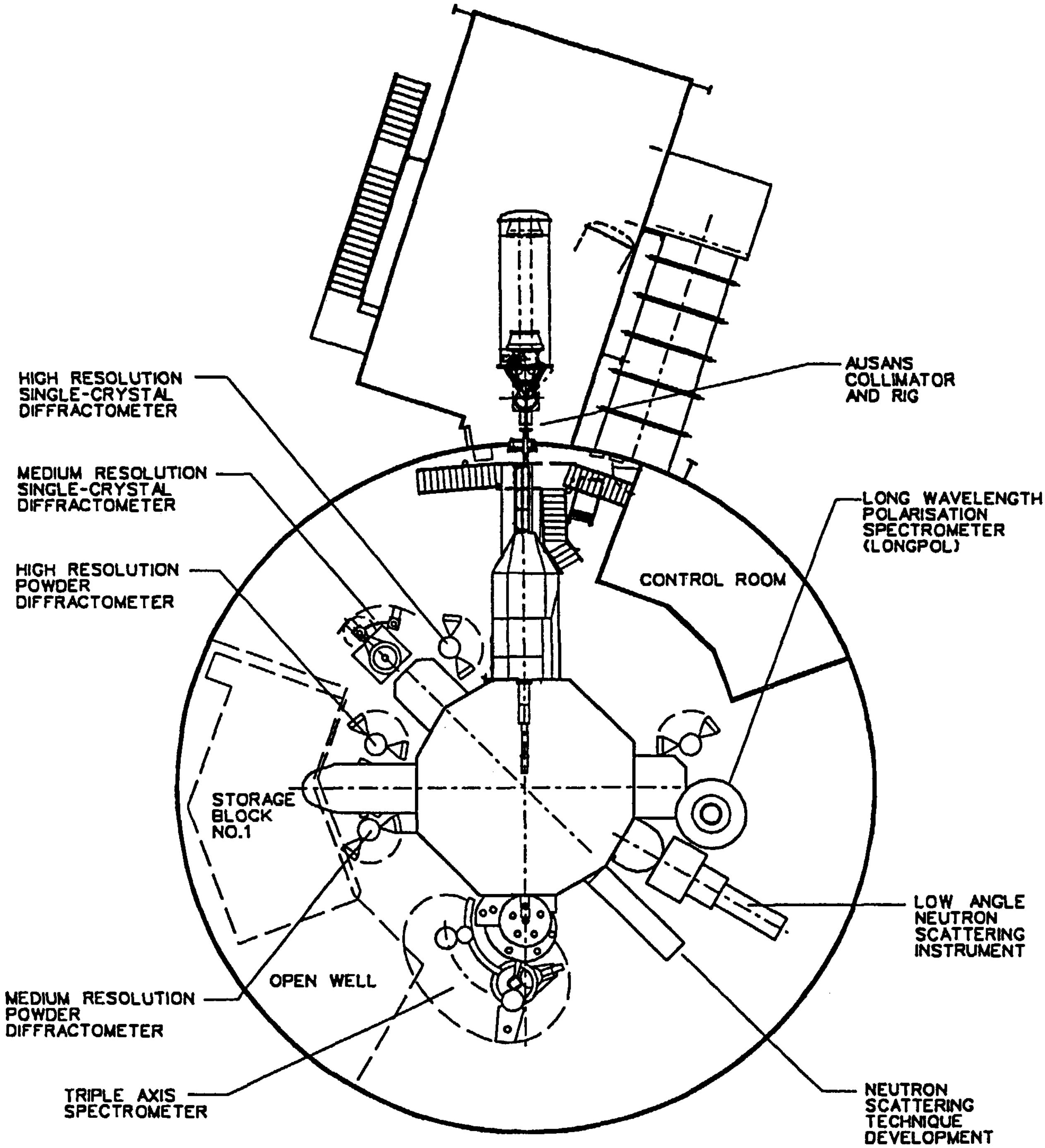

Figure 1. Disposition of the neutron scattering instruments on HIFAR.

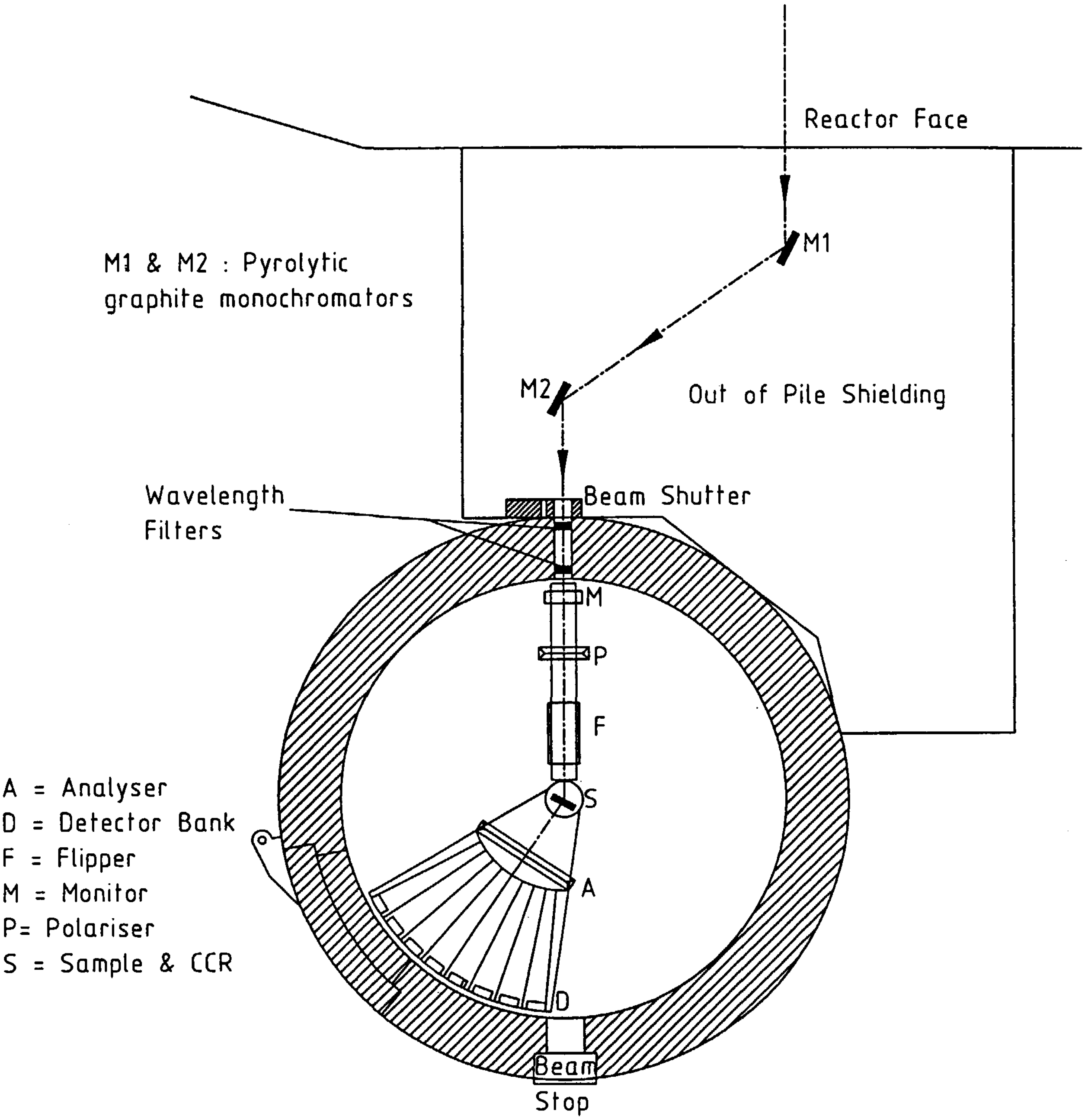

Figure 2. Schematic of the Long Wavelength Polarised Neutron Spectrometer (LONGPOL) on HIFAR.

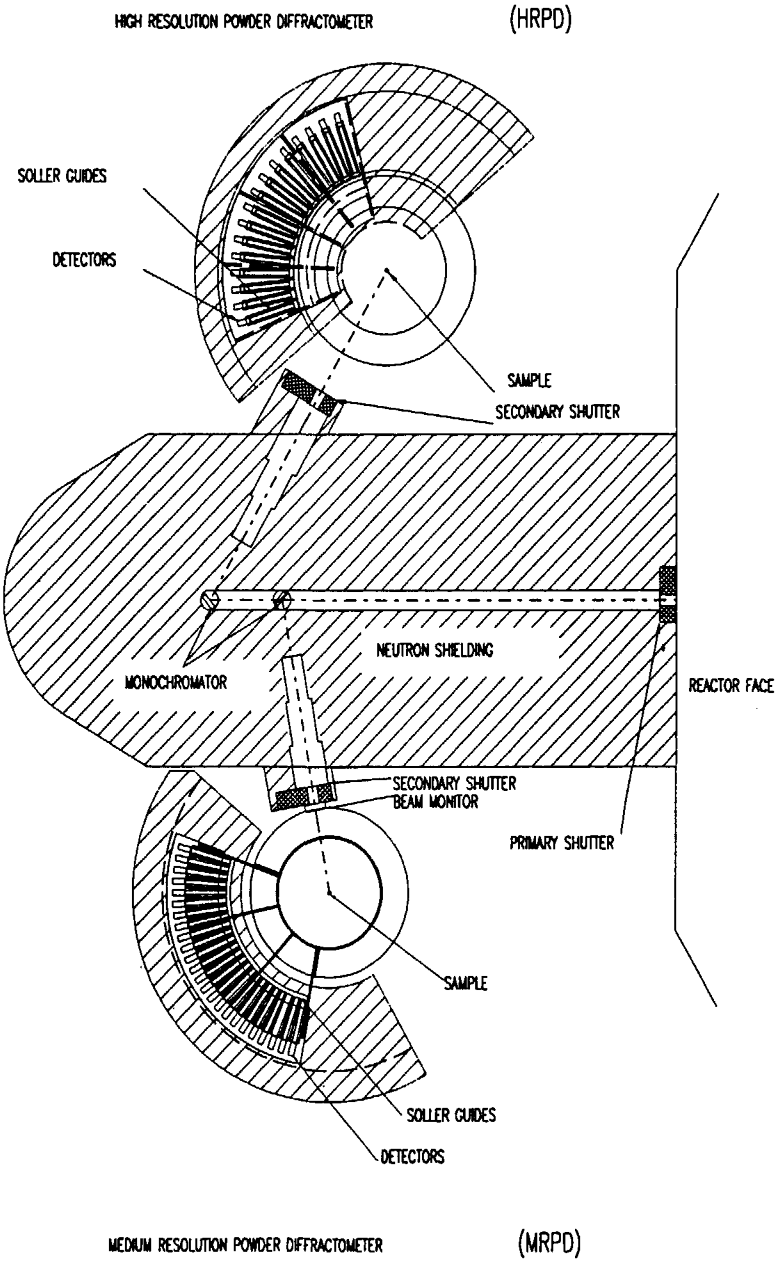

Figure 3. Schematic of the two powder diffraction instruments (MRPD and HRPD) on HIFAR

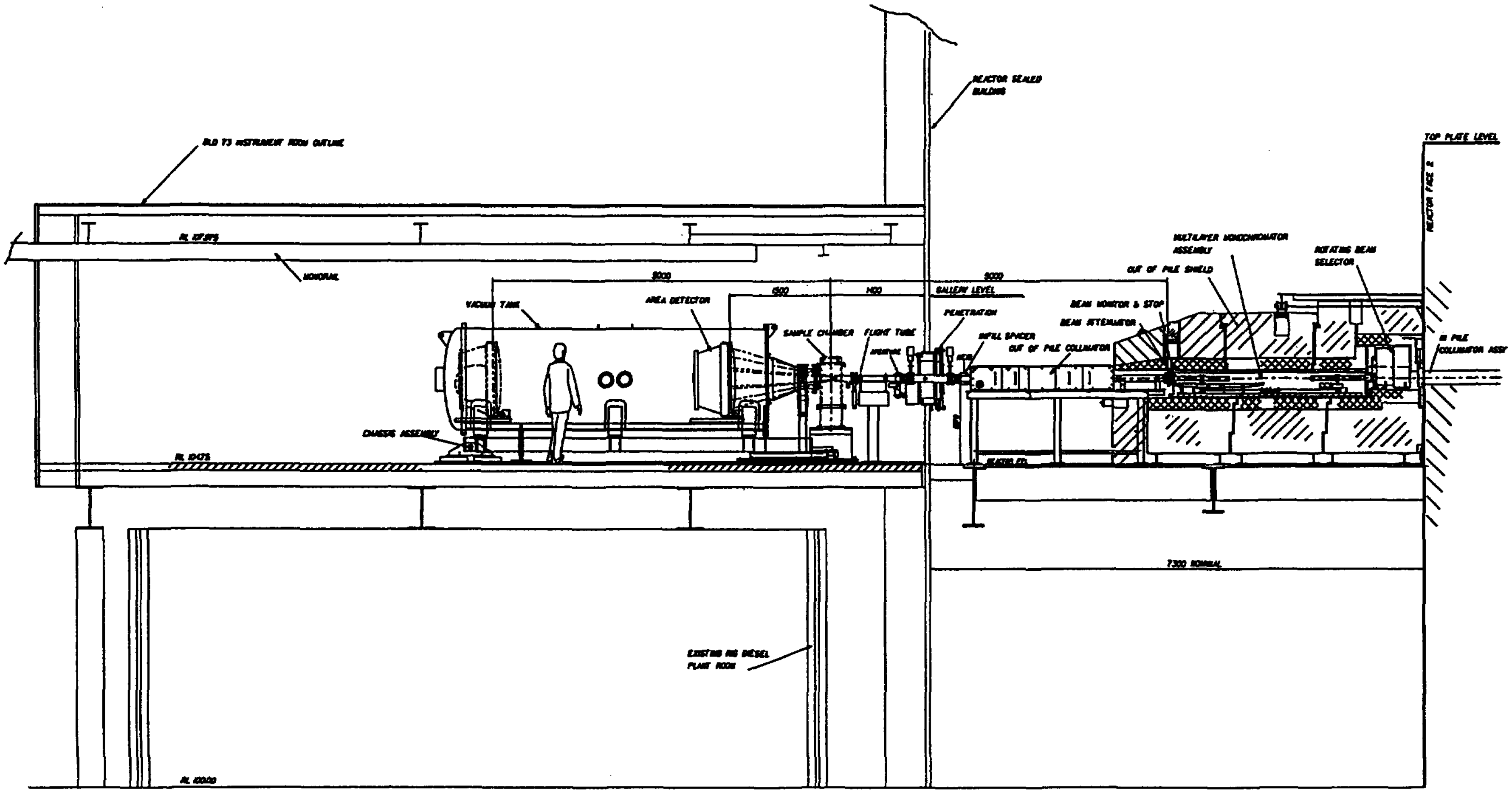

Figure 4. Schematic of the small angle neutron scattering (SANS) instrument on HIFAR.

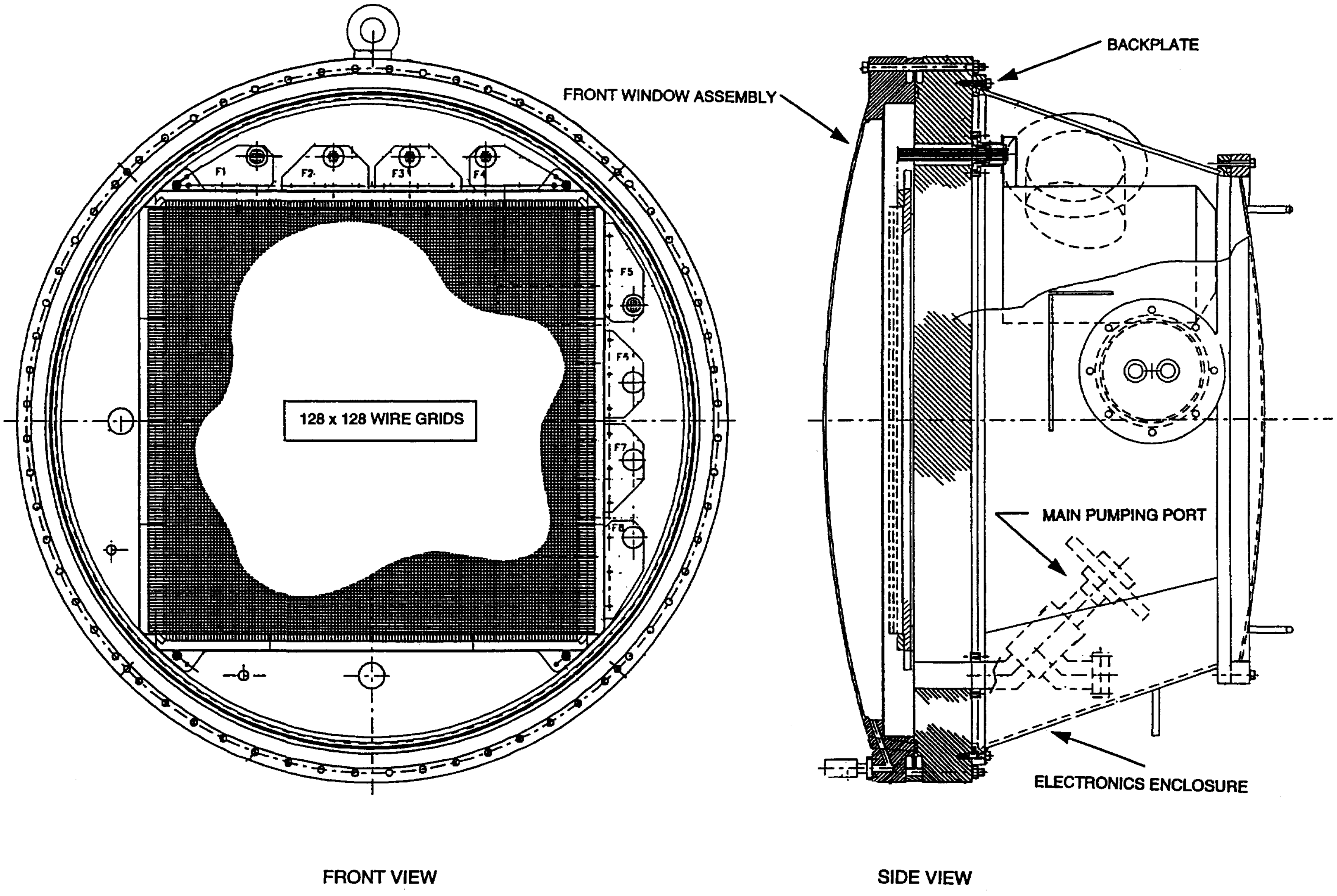

Figure 5. Schematic of the two-dimensional position sensitive neutron detector built for the SANS instrument on HIFAR.

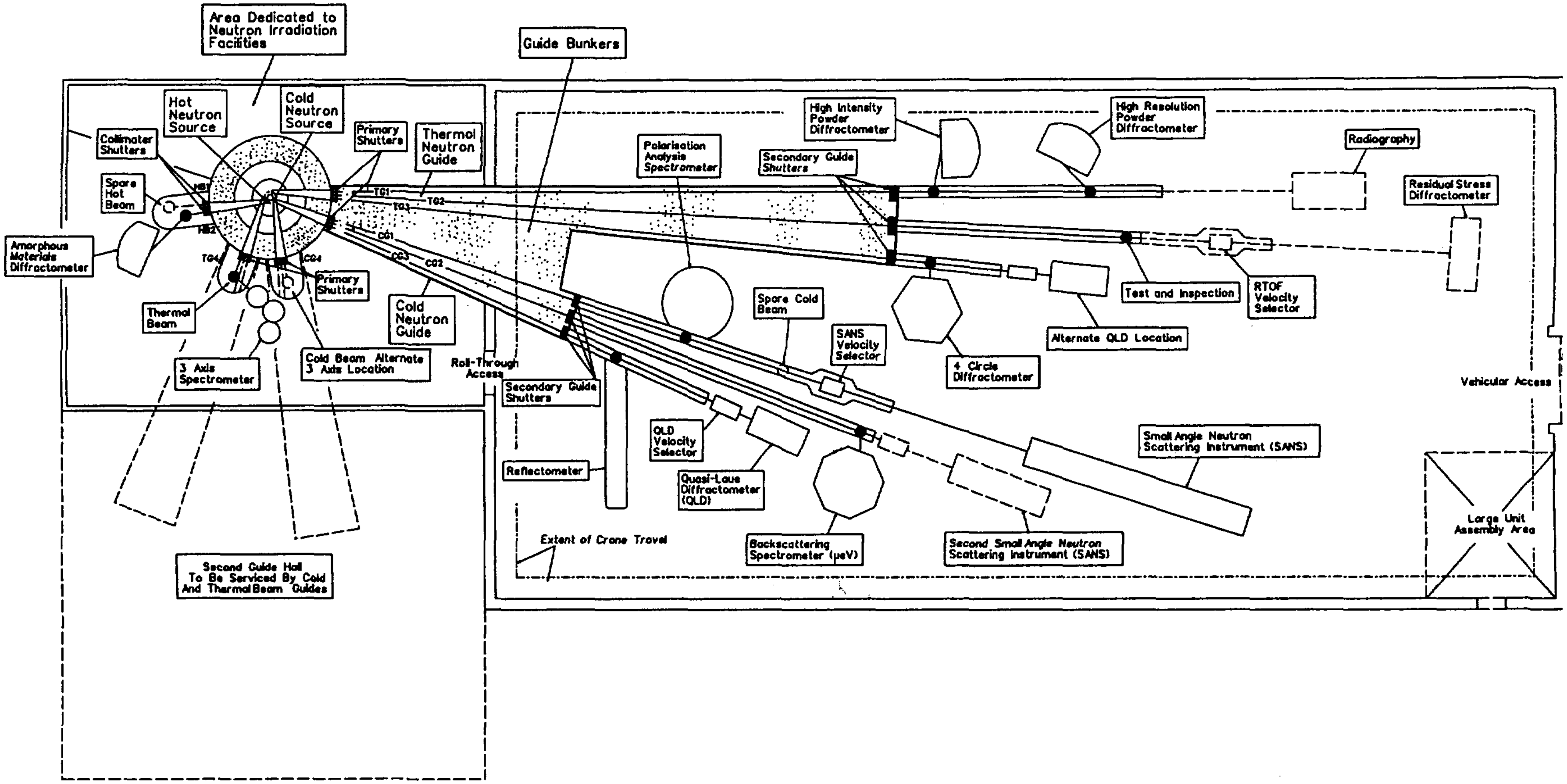

Figure 6. Schematic layout of the neutron scattering instruments proposed to be located on the replacement research reactor.