

Report on Neutron Powder Diffraction for the Australian Replacement Research Reactor

Neutron Powder Diffraction Workshop

ANSTO

17-18th October, 2000.

Report on Neutron Powder Diffraction for the Australian Replacement Research Reactor

Prepared by participants at the Neutron Powder Diffraction Workshop, held at ANSTO on the 17-18th October, 2000.

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Executive Summary

There is a clear need for two neutron powder diffractometers at the Australian Replacement Research Reactor when it starts operation in 2005. The High-intensity instrument should be capable of measuring a 10mg sample of moderate complexity, or perform single-shot time-resolved experiments with 1-second time slices, or perform stroboscopic measurements with time slices of order 50 microseconds. The High-resolution instrument should have a target resolution of $\Delta d/d \sim 6 \times 10^{-4}$, and be capable of collecting data at this resolution within 1-48 hours depending on sample size and crystal complexity.

Key questions that need to be answered in the next 9 months include: (1) a detailed study of monochromator options, (2) analysing the detector options for the high-intensity machine and exploring ways in which the solid angle can be maximised for both instruments, (3) whether the instruments are better situated at the reactor face or on supermirror guides, (4) how to integrate the two instruments (physically, if they are only the same guide), and scientifically as regards detailed performance specifications.

The user community clearly wants a wide range of sample-environment options, and these are listed in the report. Combinations of these options will be important.

On the 17-18th of October 2000, the first workshop to define the neutron-beam instrumentation for Australia's Replacement Research Reactor was held at ANSTO, Lucas Heights. The subject was neutron powder diffraction. There will be two powder diffractometers at the reactor, representing some 25% of the total instrumentation when the reactor starts operation in late 2005.

The purpose of the workshop was to:

- identify the future needs and opportunities in this area, and
- specify instrument requirements based on the future needs.

This document represents the accumulated views of the participants at the workshop.



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A detailed list of participant names and affiliations is in Appendix A.

1. Introduction

Powder diffraction is one of the most widely used techniques to study the structural and microstructural properties of materials. Neutron powder diffraction, although less commonly available than x-ray diffraction due to its reliance on large-scale infrastructure, plays an essential role in these structural studies. This need for neutron powder diffraction has led the Beam Facility Consultative Group (BFCG) for the Australian Reactor Project to recommend that two neutron powder diffractometers be a leading priority for the new instrumentation at the reactor [1]. Similar recommendations from the recent international reviews of the AUSTRON project [2] and the European Science Foundation on Neutron Scattering Review [3], have also highlighted the essential nature of neutron powder diffraction.

This document outlines the scientific case for two neutron powder diffractometers, the specifications that result from the scientific case, as well as other important issues associated with the eventual operation of these instruments.

2. Scientific Case

Powder diffraction is used to determine the real-space structure of materials at the atomic and microstructural level. Potentially, diffraction allows determination of: long-range structure in polycrystalline materials, short-range atomic structure in disordered or amorphous materials, structural distortions, and any strain and crystal size induced changes to the structure [4]. Accurate structure determinations are essential for understanding a material because there is a direct correlation between a materials composition, structure, and its properties. This means that the applications of neutron powder diffraction are significant and involve the fields of structural physics and chemistry, condensed-matter physics (magnetism, superconductivity), materials science, life science, earth science and engineering.

Neutron powder diffraction has many attractive features that make it complementary to other techniques, such as x-ray and electron diffraction. These features include the neutrons' penetrative ability, light element sensitivity, isotope dependent scattering, and its magnetic interaction. This makes neutron powder diffraction applicable to a wide variety of technologically important materials and scientific investigations.

The ability of a neutron to penetrate a material makes it ideal for *in-situ* experiments using sophisticated sample environments. This allows measurements at low and high temperatures, in electric and magnetic fields, and under varying pressure. Examples include study of phase transitions with temperature, geological samples under pressure, magneto-resistive materials, magnetic transitions at ultra-low temperatures, etc.

Many important compounds involve light elements such as H, D, Li, C, N and O. The neutron's sensitivity to such elements and the difference in scattering between isotopes means neutron powder diffraction plays an important role in determining the structural features of compounds with these elements. Examples include ceramics (oxides), Li batteries, magneto-resistive materials, hydrogen storage materials, superconductors, zeolites, and so on.

Magnetic structures can be studied using neutron powder diffraction due to the magnetic interaction between the neutron and local magnetic moments in the compound. Examples include rare-earth hard magnets, high- T_c related materials, molecular magnets, correlated f -electron systems, magneto-elastic coupling, and so on.

The ability to discern many of the features outlined above relies on the type and function of the neutron powder diffraction instrument(s) available. A powder diffraction instrument has two essential characteristics: its resolution and its intensity. The resolution of a powder diffraction instrument dictates the ability to discern the real space features of the material, while the intensity dictates how quickly one can achieve such a measurement. The two characteristics are (unfortunately) inversely related, in that an increase in resolution means a decrease in intensity. Appendix B gives more detail into this relation.

Broadly, we can separate the classes of experiments into those that primarily need intensity and those that primarily need resolution. Figure 2.1 shows the qualitative relationship between experiment, resolution, and intensity; the trend from lower-left to upper-right reflects the limiting nature of a powder diffractometer (the Intensity-Resolution trade-off).

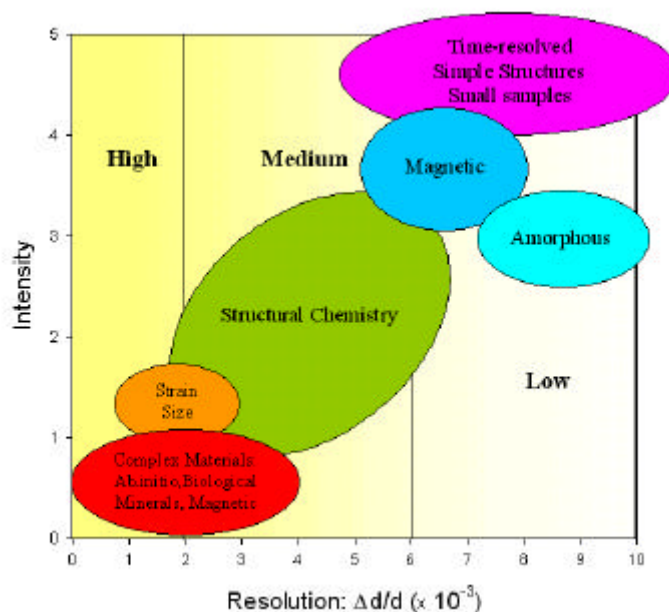


Figure 2.1: Guide to the mind's eye of the relation between experiment, intensity, and resolution.

2.1 High-Intensity Powder Diffraction

Experiments that benefit from high intensity can be categorised into two areas: those requiring speed and those with weak scattering. The first category involves experiments such as time-resolved experiments and phase-transition studies. The second category includes experiments involving extremely small samples (sometimes as little as 10mg), quantitative phase analysis, weak magnetic scattering, and hydrogen containing samples.

The first major benefit of a high-intensity instrument is the reduction in counting time per pattern collection, hence time-resolved experiments can be performed. These experiments are either reversible, and can be repeated *ad infinitum*, or are irreversible experiments - so

called *one-shot* experiments. Reversible experiments can be performed with a time resolution equal to the instruments data acquisition time sequence, typically 30-50 μ s per time-slice. An experiment can be synchronised with the data acquisition system and repeated until sufficient accuracy in the powder pattern has been obtained. Figure 2.2 shows the two types of data acquisition for the D20 instrument (ILL). Types of experiments that use this acquisition mode are ultrasonically excited transitions, fluid dynamics and more.

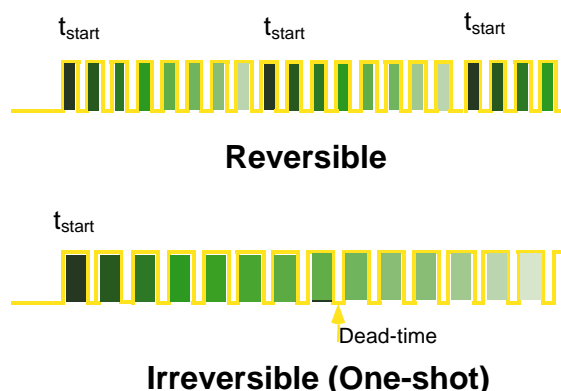


Figure 2.2: D20 (ILL) data acquisition method for reversible and irreversible measurements. Courtesy of T. Hansen (ILL)

Irreversible experiments are "destructive" experiments where the material cannot be cycled continuously. These *one-shot* experiments include such things as chemical kinetic experiments. Due to the irreversibility, the time slice is necessarily longer - the order of 1s or more. Collection speed is important in the area of chemical reactions, such as in hydration of cements. Such reactions may be very fast, but still require a significant number of structural determinations to determine the kinetics of the reaction. Many materials have several reactions proceeding simultaneously with different reaction rates; collecting data quickly allows fewer possibilities of missing quick, but important precursor or intermediary reactions.

Structural phase transitions, both nuclear and magnetic, occur in many important materials, including high- T_c superconductors, magneto-resistive materials, heavy-fermion systems, and so on. The ability to observe the evolution of structure through a transition is important to the fundamental understanding of these materials. Temperature, pressure, magnetic/electric fields, and combinations of these can cause a material to undergo a phase transition and it becomes necessary to map large areas of phase space around a transition. A simple problem of mapping a temperature-pressure region may require on the order of 300 structural determinations (30-40 temperatures at 8 pressures). To perform such an experiment would require over 12 days if each data point took 1hr to collect - this is totally prohibitive. The need is for speed. An instrument that collects a pattern on the order of minutes, say 5 minutes, could perform this experiment in a day. An instrument that can collect data even more quickly could even collect data while the system was ramping (eg thermodiffraction). What then can occur is that the experimenter can study more samples, or there can be more experiments.

Single parameter studies, such as shown in Figure 2.3, benefit from extensive structural analysis, where subtle detail is revealed; sometimes the devil is in the detail, where the subtle Imma phase might have been missed had larger temperature steps been taken. Such studies can only be taken on a high-throughput/high-resolution instrument (in this case HRPD-ISIS).

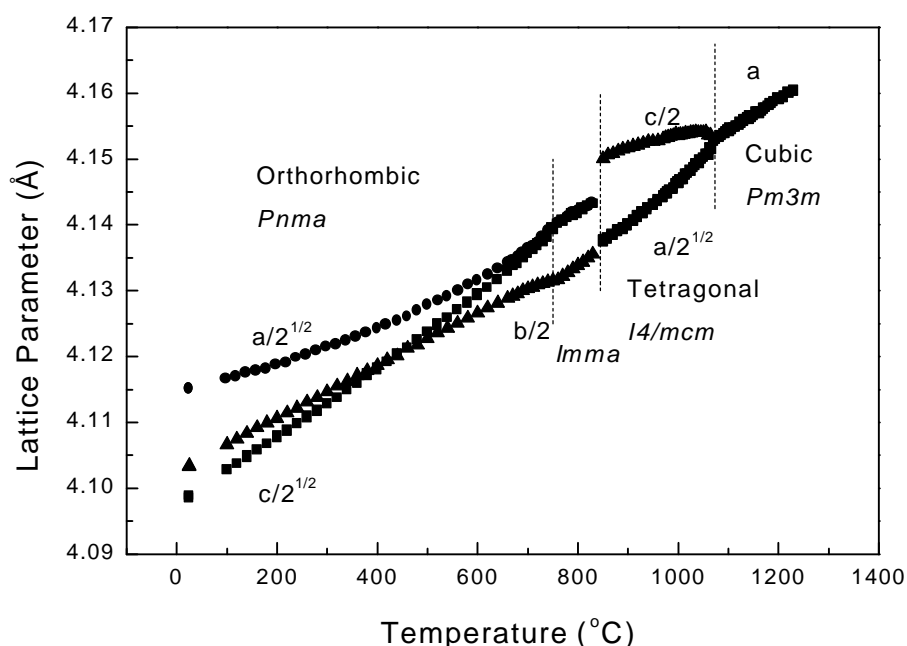


Figure 2.3. Temperature dependence of the reduced lattice parameters for SrZrO_3 . Approximately 120 structural refinements were used from HRPD (ISIS). Courtesy B. Kennedy and C. Howard.

Another benefit from a high-intensity instrument is that many new compounds can not (initially) be made into the 1 - 10g samples usually required for a neutron powder diffraction experiment. This is particularly true for materials that required special processing, such as those made in a high-pressure oxygen atmosphere. Currently there are few places where one can collect a neutron powder diffraction pattern on a small sample, of say 10-100mg (see Appendix D for a list such instruments), but it will become increasingly important to have such an ability.

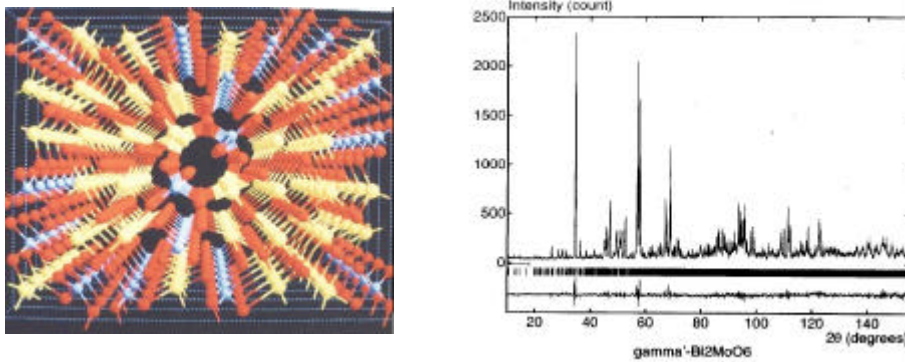
Examples of small samples include: thin films, where the material of interest may only be a few hundred microns thick, or samples with spatial variations of 1mm^3 or less. Also, high pressure cells usually afford only a small sample volume, so high intensity is essential for these experiments.

Hydrogen-containing samples, such as those of interest to the chemistry and life science communities, have small Bragg scattering contributions compared to the incoherent scattering from hydrogen. This means the signal-to-noise ratio is small. Two methods can possibly be used to reduce the hydrogen signal (and hence increase the signal to noise): polarisation analysis and energy discrimination, but it is not clear whether either of these methods lends itself to the high-intensity instrument. In any case, the potential to impact on this field of research is great.

2.2 High-Resolution Powder Diffraction

As the sphere of research expands into new materials the complexity of the structural problem increases. In these cases, a higher resolution instrument can more accurately determine the atomic and magnetic structures. This is because an increase in resolution allows greater peak separation, hence greater accuracy in the determination of the peak intensities needed for structural refinement. There is a plethora of uses for high-resolution powder diffraction, some of which are listed here:

- determining crystal structures *ab initio* from powder diffraction data, in particular when no suitable single crystal can be obtained. This is an (exciting) emerging field and great inroads will be made into this field of structural analysis in the coming years. In most cases a greater resolution will mean a greater chance of successful structure solution
- materials with complex crystal structures, including catalysts (figure 2.4 and figure B-2), incommensurate structures, hybrid materials, organics, cements, natural minerals, zeolites, phase separation, and non-linear optical materials. In many cases, the subtleties of the structure relate fundamentally to the material property.



Figures 2.4: High-resolution neutron powder diffraction of monoclinic bismuth molybdates (industrial catalysts for the selective oxidation and ammon-oxidation of olefins) with unit cell volumes of ranging from 2000 to 8000Å³ [5]. Courtesy of T. Vogt.

- many materials undergo phase transitions where a small peak splitting is the leading indicator of structural change. This includes areas of research into ferroic materials (ferroelectric, ferroelastic, ferromagnetic, etc), critical phenomena, and electronic materials such as superconductors and magneto-resistive materials.
- changes to the peak shape are good measures of strain, crystallite size, and defects. These are important parameters in materials such as batteries, hydrogen storage materials and mesoscopic structures. The coupled use of small angle neutron scattering may provide more insight into such materials.

3. Desired Performance Specifications

The following tables outline the requested specifications for the two neutron powder instruments. It should not be taken as a comprehensive list. Each instrument is divided into two sections: the overall performance requirements in terms of required resolution and

intensity, and a list of requirements for specific components of the instruments that might help achieve the performance and user requirements.

3.1 High-Intensity Powder Diffractometer

The high-intensity instrument should be capable of measuring a complete pattern using a continuous detector (one that generally does not require moving to complete a measurement). The minimum measurement time-slice should be extremely small, say 30 μ s, for performing real-time experiments.

Table 3.1: Summary Table of Desired Performance Requirements for the HIPD

Property	Requirement
Resolution	<ul style="list-style-type: none"> $\Delta d/d > \sim 2 \times 10^{-3}$ should not be outside HRPD's range by more than a factor of 2 wavelengths between 1 - 2.4\AA, possibly also $\sim 5\text{\AA}$
Intensity	<ul style="list-style-type: none"> smallest sample weight $\sim 10\text{mg}$ typical sample size $\sim 10\text{mm}^3$ (restricted due to focusing elements) 1s acquisition for 10mm^3 (17min for 1mm^3) in <i>one-shot</i> irreversible experiments 30μs acquisition in stroboscopic mode (reversible experiments) on large samples

Table 3.2: Desired component requirements for the HIPD

Component	Requirement
Collimators	<ul style="list-style-type: none"> possibility of variable vertical detector slits (at low angles to reduce asymmetry) possibly a focusing arrangement between monochromator and sample for very small samples (eg capillary focusing)
Monochromators	<ul style="list-style-type: none"> accessible wavelength range of between 1 - 2.4\AA several different monochromators including mosaic and non-mosaic types (eg. pyrolytic graphite, Ge, Bent Si/Ge, Cu) different take-off angles, say 40-120$^\circ$ variable vertical focusing
Detector	<ul style="list-style-type: none"> continuous detector bank of 160$^\circ$, rather than a number of cells 0.1$^\circ$ detection cell, with 0.05$^\circ$ by moving the detector(s). should be able to move several degrees to cover a 2θ range from negative angles to 165$^\circ$ have largest solid angle possible use established techniques rather than unproven (eg may have problems with micro-strip technology) should have stability $< 10^{-4}$ (good detector cooling) count rate $> 10^4/\text{cell/s}$ without saturation and dead time corrections types of detector for consideration are: <ol style="list-style-type: none"> multi-wire gas chamber (<i>à la</i> Cerca type) Position sensitive detector (<i>à la</i> Popovici/Berliner) image-plates

Filters	<ul style="list-style-type: none"> need filters for some types of monochromators: <ol style="list-style-type: none"> 1. pyrolytic graphite for 2.4Å 2. Be for $\lambda > 4\text{\AA}$
Dimensions	<ul style="list-style-type: none"> sufficient free space around sample position for ancillaries possible variable sample-detector and monochromator-sample distances

3.2 High-Resolution Powder Diffractometer

The high-resolution instrument should be capable of measuring a complex powder pattern in a reasonable time period. Its resolution should match the current world's best constant-wavelength powder diffractometers, and have the flexibility to vary it. As part of the detection system, a small-angle detector for the study of moderately sized particles and microstructure should be examined.

Table 3.3: Summary Table of Desired Performance Requirements for the HRPD

Property	Requirement
Resolution	<ul style="list-style-type: none"> $\Delta d/d$ capable of 0.6×10^{-3}, better if possible, and less if required (eg 1×10^{-3}) wavelengths between 1 - 3Å
Intensity	<ul style="list-style-type: none"> typical acquisition times of about 1h to 2days, depending on the resolution required and sample size

Table 3.4: Desired component requirements for the HRPD

Component	Requirement
Collimators	<ul style="list-style-type: none"> versatile primary and secondary collimators automatic insertion of collimators don't have a collimator close to the natural limit (just a waste) variable (automatic) slits evacuate (or He fill) all beam areas
Monochromators	<ul style="list-style-type: none"> accessible wavelength range of between 1.0- 3.0Å continuously variable take-off angles, say 120-155° completely automatic changing of wavelength multiple monochromators: use Ge to begin and then look at other monochromator types variable vertical focusing
Detectors	<ul style="list-style-type: none"> types of detector for consideration are: <ol style="list-style-type: none"> 1. Multi-wire <i>à la</i> HRPT(PSI/Cerca) with radial collimator 2. Vertical PSD <i>à la</i> Super D2B (ILL)/FRM-II 3. Standard collimator/detectors <i>à la</i> D2B/HRPD(ANSTO)/BNL 4. Horizontal PSD <i>à la</i> Ron Berliner (MURR) 5. Area Detector 6. Other new technology: solid state, liquid, image-plate 20x40cm, scintillation counters have a (cheap) SANS option for the low angle region, possibly an image-plate arrangement or CCD need excellent detector shielding ⁶Li beam stop
Filters	<ul style="list-style-type: none"> (automatic) filter for PG $\lambda = 2.4\text{\AA}$
Dimensions	<ul style="list-style-type: none"> space enough for ancillaries

4. Ancillary Requirements

Current and future research using powder diffraction is moving towards greater complexity, and this necessitates the need for a well equipped suite of ancillary equipment to deal with the user requirements. By 2005, the number of experiments that will be performed under (only) ambient conditions will be small. The current ancillary usage on the ISIS powder diffractometers, shown in Figure C-1 (Appendix C), indicates that less than one in seven experiments are performed under ambient conditions. This means that significant resources are required for the ancillary equipment on the two new powder diffractometers. A list of typical ancillaries is given in Appendix C.

There are two classes of ancillaries: the instrument specific and the "pool" ancillaries that are common to a number of instruments.

4.1 High-Intensity Powder Diffractometer

Generally to be automated and compatible with other NS instruments and with instruments of user community. The following is a list of ancillaries thought to be required for the high-intensity instrument.

Temperature:

~3.5 K to 2300 C required.

less than 1K is desirable for selected experiments.

Two cryo-coolers per instrument to reduce cool-down time.

Standard orange cryostat (>1.5 K) to be considered, dedicated to HRPD, V-tail of 50 mm to fit pressure devices.

Dedicated furnace with Vanadium heating elements (<900 C) of different diameters, to fit hydrothermal cells (<200 C) (optionally Niobium for higher temperatures). Different heating elements could be used if a radial collimator to used (will also reduce the vanadium incoherent scattering).

Mirror furnace in pool (<1800 C).

Graphite furnace in pool (>1800 C).

Pressure:

Axial pressure: 10GPa

Hydrostatic: 25GPa –can we tackle geological problems?

Hydrothermal: 200C at autogenous pressure (~12bar)

Magnetic Fields:

8 Tesla vertical continuous (with temperature insert), and possibly higher fields.

Ambient:

Eulerian cradle, "C-shape" and/or acentric if possible

Vacuum vessel (RT measurements without air scattering)

Automatic control of omega axis (rocking curves, environment alignment)

Automatic sample changing

At least 2 translations of ~250mm range

Sample translation stage (x,y,z)

When operated without a radial oscillating collimator, dedicated sample environments (eg V-tail cryostat, V-heating element furnace, and vacuum vessel) should have a large evacuated space (80 cm in diameter) around sample, and have an inside shielding/beam-stop of cadmium and/or boron carbide for suppression of all background contributions of aluminium wall scattering. If available in suitable form ⁶Li could also be used.

A radial oscillation collimator with a focus of a few centimetres seems to be an interesting option.

4.2 High-Resolution Powder Diffractometer

It was agreed that there must be a "standard" suite of ancillaries as at ILL and ISIS. This would include: cryo-coolers, cryo-furnaces, furnaces, pressure cells, cryomagnets (0-14T), omega table, sample platform (xy trans), tanzboden floor, etc. The controllers should be, where possible, all be the same. There should be a dedicated technical group to maintain the ancillaries. A standard configuration should be adopted, so that more than one instrument can use the same ancillary.

Particular items require special mention. The cryostats have a larger range in temperature (down to 1K) compared with a cryorefrigerator, but more importantly, they give temperature stability over the entire sample not found in cryorefrigerators. This will be important when studying low temperature phase transitions. One or two "orange" cryostats, or equivalent, should be considered. Also, maybe a hut should be built over the detector to reduce neutron and electronic noise.

In regard to user interactions, the facility should be able to easily accommodate of user provided equipment. It could be that detailed technical specifications on the instruments sample space are provided via a web interface, as well as information and forms for safety clearance procedures

5. User Interface

An experiment not only involves equipment but also people. The interaction between the user, the equipment, and the facility must be addressed.

5.1 High-Intensity Powder Diffractometer

Only one point was stressed, and that was the software should be user friendly, in particular in regard to changing the instrument configuration.

5.2 High-Resolution Powder Diffractometer

Several issues were raised as important considerations. In accessing the facility there was a significant emphasis on ease of access. This was not only in regard to the local community access, but also that the access scheme should/must allow international access. A consultation process with the user community should be used to address this issue. Also raised was the issue of industrial interaction, and whether the facility could allow for extensive industrial usage. An example is in-line process monitoring and control of industrial plant and material.

Software is an important aspect of any instrument, and ease of use is vital. Remote viewing software should be incorporated into the instrument software, to allow users to access their data from their home institute or wherever they might be. Also the software should use the latest software standards, in particular in terms of data formats (eg Nexus).

6. Issues to be resolved within the next 9 months

6.1 *High-Intensity Powder Diffractometer*

What are the geometrical constraints with the different take-off angles?
 What are the differences between bent Si monochromators and bent Ge or Be?
 What are the differences between non-mosaic and mosaic monochromators?
 Will an end-guide position be required?
 Will a reactor face or guide position be better? (This needs to be asked in light of the large support for PDF analysis, as a reactor face position would give more useable short wavelength neutrons)
 Can the detector be vertically extended?

6.2 *High-Resolution Powder Diffractometer*

Need to look at new monochromators - gradient crystals, Bent crystals, etc.
 Which is best: large high PSD's, multiple detectors/collimators, or some other detector?
 Need to look at instrument compatibility - between HIPD and HRPD and with other instruments (such as software, components, ancillaries, etc)
 Will a reactor face or guide be better?
 Look at the background and what shielding design is needed.
 Examine the Ni coated Si collimators.
 Is a long wavelength for magnetic studies needed (even an instrument on the cold guides?)
 Can we put $m = 1$ guides between HIPD and HRPD?
 Can vertical focusing be improved with guides between the monochromator & sample?
 Look into the detector sensitivities.

7. Other Discussions

A significant issue raised in general discussion was the ability to perform pair distribution function (PDF) analysis. Although the high-intensity instrument may provide some ability this is the domain of a dedicated liquids and amorphous diffractometer on a hot source. It would certainly require a reactor face position for a reasonable short wavelength spectrum, even if the high-intensity instrument is used.

7.1 *High-Intensity Powder Diffractometer*

None reported.

7.2 *High-Resolution Powder Diffractometer*

Computing advances may make it possible to perform online refinement and/or solution. Areas where this would benefit are in quantitative phase analysis, and measuring a pattern until the required statistical errors on various atomic parameters are obtained.

Standardisation is an important part of instrument building as large savings in time and money can be achieved, therefore, wherever possible standard connections/fittings/components should be used.

Due to the complexity of some detectors, a dedicated technical worker may be required. For example, a dedicated technician could be used to deal with wire detectors on 2 or 3 instruments.

High-pressure experiments may be best performed on another machine, such as a TOF residual stress instrument. If such an instrument is built, and a high-pressure capability incorporated, then it might change some design parameters on the two powder instruments.

When obtaining vanadium cans, one should ask for a 5% Nb doping of the vanadium (to make a null metal), because as experiments become more demanding even the small vanadium Bragg peak can be observed.

8. Summary

It is clear there is a well-founded scientific case for the two neutron powder diffractometers, and both instruments should be world-class when commissioned in 2005. The participants came to the following conclusions:

Conclusion 1:

The high-intensity instrument should be capable of performing: 30-50 μ s stroboscopic experiments, 1s time-slice irreversible experiments, and of measuring a 10mg sample.

Conclusion 2:

The high-resolution instrument should be capable of measuring with a resolution of about $\Delta d/d \sim 0.6 \times 10^{-3}$, and collect in a reasonable time (1hr – 2 days).

Conclusion 3:

A complete suite of ancillary equipment is essential for each instrument.

Conclusion 4:

Both instruments should be versatile, and be fully (automatically) controlled.

Conclusion 5:

User access should be universal and easy.

9. References

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Appendix A: Participation List

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Appendix B: Intensity-Resolution

For normal mosaic optics, there is a fundamental relation between Intensity and Resolution [6]. This means increasing resolution reduces the intensity. Resolution gives the ability to differentiate real-space features, while intensity gives the ability to perform real-time experiments.

There are several ways to adjust resolution, and hence intensity. The parameters that affect the Intensity-Resolution are:

- sample size
- collimators
- efficiency of the detectors
- area coverage of detectors
- number of detectors
- wavelength
- monochromator
- take-off angle

Most of these are interrelated. Typical resolution curves at different wavelengths are shown in Figure B-1

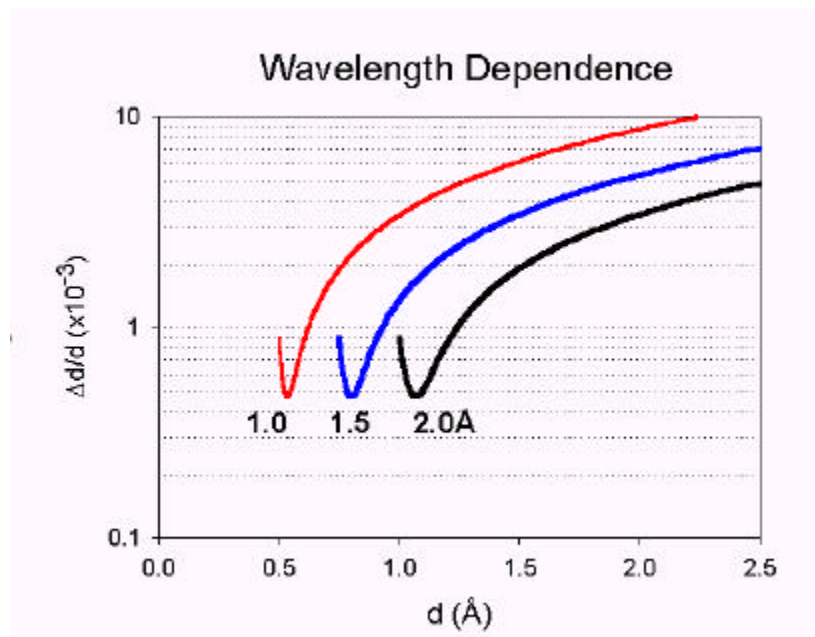


Figure B-1: Calculated resolution curves for D2B(ILL) ($\alpha_1 = 5'$) at various (artificial) wavelengths.

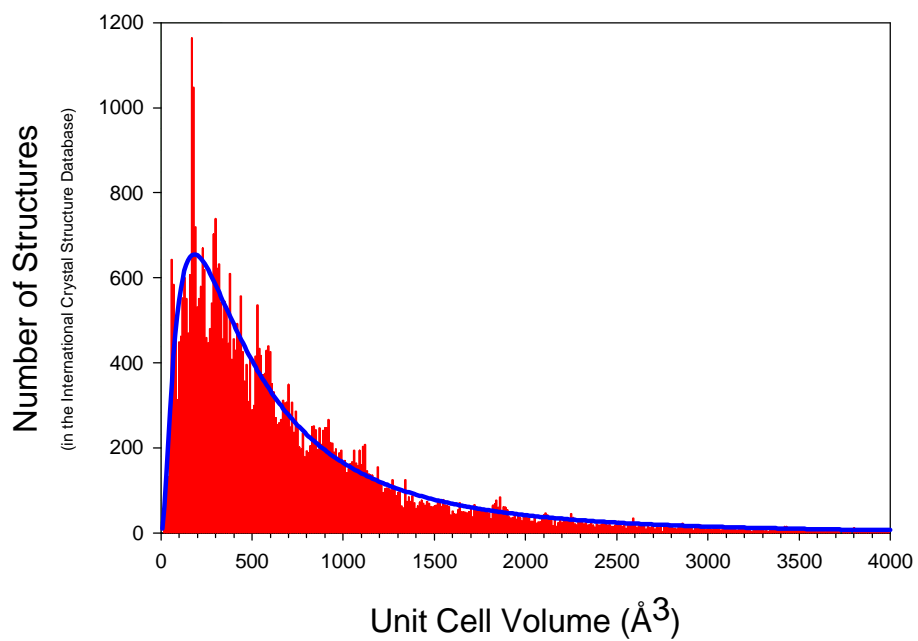


Figure B-2: Number of structures in the Inorganic Crystal Structure Database as a function of unit cell volume (see <http://icsd.ccp14.ac.uk/icsd/>).

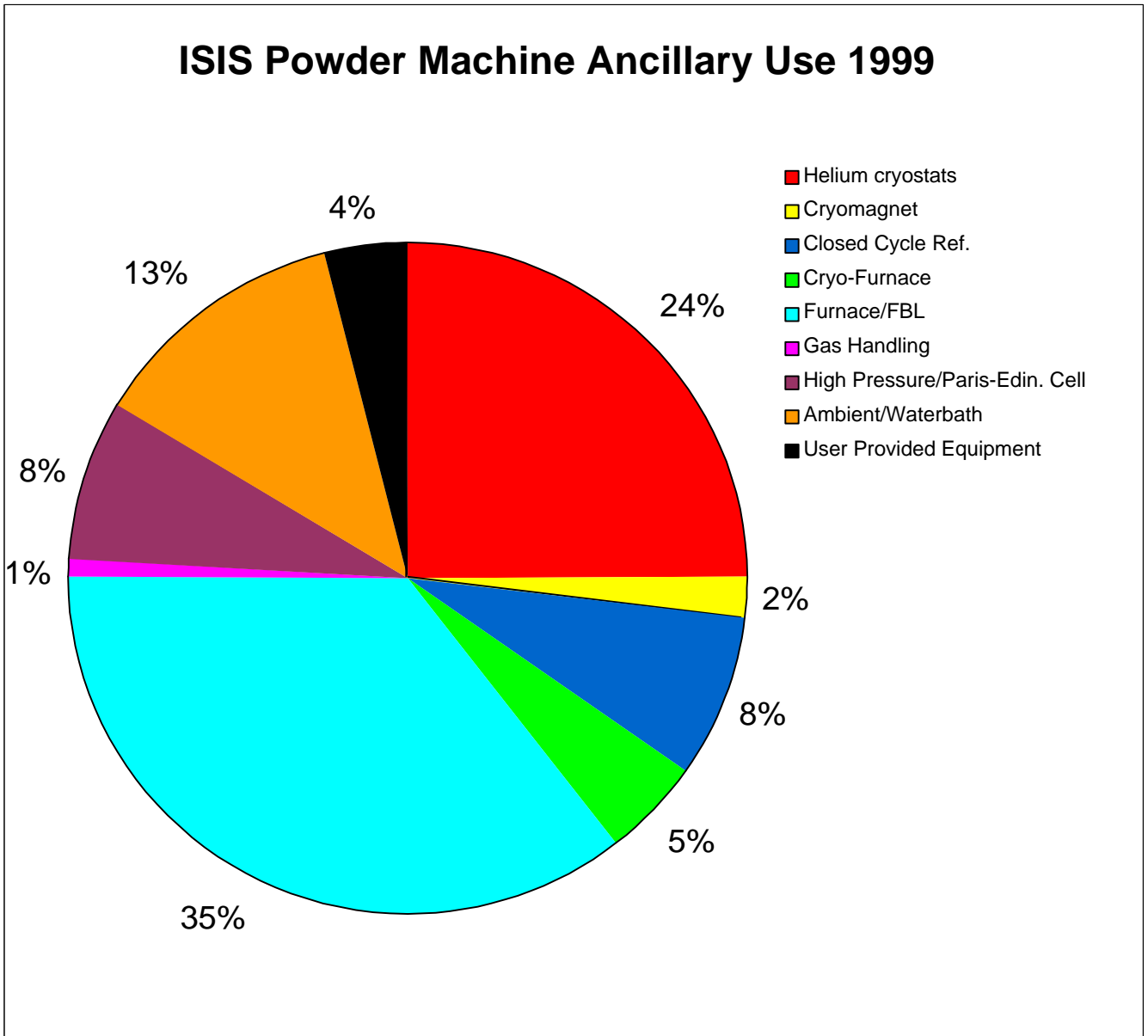
Appendix C: Ancillaries

Table C-1: List of Common Ancillaries

Ancillary	Range	Use	Pros	Cons	HIFAR
Closed-cycle refrigerator (CCR) – ordinary	8 - 300K	Low temperature studies	No expensive fluids	Limited low temperature	Y
Closed-cycle refrigerator (CCR) - Joule-Thompson	~3.5 - 300K	Low temperature studies	No expensive fluids better temperatures	Limited low temperature more expensive plant	Y
Cryostat (He)	1.4K - 300K	Low temperature studies	low temperatures	expensive He fluids	N
Cryo-furnace (CCR)	10 - 600K	Low and medium temperatures	No expensive fluids No changing ancillaries	Limited low temperature Limited high temperatures custom made (?)	N
Cryo-furnace (He)	1.4K - 600K	Low temperature studies	low temperatures	expensive He fluids	N
Dilution Refrigerator (CCR)	100mK -	ultra low temperatures	no helium cryogenes needed	expensive, new technology	N
Dilution Refrigerator (He)	50mK -	ultra low temperatures	standard	needs helium cryostat expensive	N
Cryomagnet (CCR)	0-8T	high magnetic fields	reasonable fields	expensive	Y
Cryomagnet (He/CCR?)	0-14T	high magnetic fields	high fields	more expensive may need cryogenic fluids	N
Pulsed Magnet (He)	0-40/50/60T	very high magnetic fields	very high fields with strobe measurements it would be possible to collect more than field during a pulse	much more expensive needs cryogenic fluids needs stroboscopic detection system time of pulses is small (<2s) and time between pulses large (~1hr).	N
Furnace: ordinary (air/gas)	50-1100°C	High temperature studies	gases ok	background/absorption due to insulating material	Y
Furnace: high temp (air/gas)	200-1700°C	Very High temperature studies	higher temperatures gases ok	background/absorption due to insulating material RT-200 not accessible sample cans not available	Y
Vacuum furnace	50-1100 °C	High temperature studies	less background problems	no gases allowed vacuum could affect samples	N

Mirror Furnace: extreme HT (air/gas)	200-2200 °C	Very High temperature studies	gases should be ok	not common hard to do at the very high temperatures	N
High Pressure: hydrostatic	0 - 0.6GPa	Pressure studies	well behaved can do low temperatures non-directional	low pressures (0.6Gpa)	N
High Pressure: quasi-hydrostatic	0 - 30GPa	Pressure studies	higher pressures	some directional behaviour	N
High Pressure: uniaxial	0 - 20Tonnes	Stress/Strain studies	easy	directional	Y
Eulerian Cradle	3D	positioning of samples			N
X-Y-Z Stages	+/- 25mm	positioning of samples			Y
Electrochemical Cells		Electrochemical reaction studies		unique for each experiment needs D2O, not H2O	User
Gas Charging Cells		Gas incorporation studies		unique for each experiment no H (but D ok)	User

Figure C-1: ISIS Powder Diffraction Usage for 1999



Appendix D: Neutron Powder Diffraction Instruments Worldwide

High-Intensity

<i>Instrument</i>	<i>Resolution (Dd/d)</i>	<i>Flux</i>	<i>Steradians</i>	<i>Detectors</i>	<i>Range</i>	<i>Comments</i>
D20 (ILL)	varies $\sim 5 \times 10^{-3}$	$3.7 - 6.0 \times 10^7$	0.285	1600, 15cm high	50 μ s time slice	42-120° takeoff PG, Cu
GEM (ISIS)	$\sim 2-3 \times 10^{-3}$ $\sim 5 \times 10^{-3}$ $\sim 10-15 \times 10^{-3}$	-	3.5sr (eventually)	ZnS - lots	145° 90° 62°	TOF 17m flight path

High-Resolution

<i>Instrument</i>	<i>Resolution (Dd/d)</i>	<i>Flux</i>	<i>Steradians</i>	<i>Detectors</i>	<i>Range</i>	<i>Comments</i>
HRPD, ISIS	$\sim 0.4 \times 10^{-3}$ $\sim 2 \times 10^{-3}$ $\sim 20 \times 10^{-3}$	-	0.41sr 0.08 eventually →0.7sr 0.01sr		150° 90° 30°	TOF
D2B (ILL) HRPD (BNL)	$\sim 0.6 \times 10^{-3}$ $\sim 0.8-1.0 \times 10^{-3}$	10^6-10^7		64, 10cm high 64, 10cm high		135°, Ge 30cm 120°, Ge 30cm
Sirius, KENS HRPT, PSI FRM-II	$\sim 0.9-1.0 \times 10^{-3}$ $\sim 0.8-1.0 \times 10^{-3}$ $\sim 1 \times 10^{-3}$	-	? sr 0.285 0.48	1600 80, 30cm high	150°	TOF 90-155°, Ge 30cm

General Purpose

<i>Instrument</i>	<i>Resolution (Dd/d)</i>	<i>Flux</i>	<i>Steradians</i>	<i>Detectors</i>	<i>Range</i>	<i>Comments</i>
HRPD, HIFAR	$\sim 1.6 \times 10^{-3}$	$\sim 8 \times 10^4$	0.09	24		120° takeoff
MRPD, HIFAR	$\sim 4.2 \times 10^{-3}$	$\sim 10^6$	0.12	32		100° takeoff
SEPD, IPNS	$\sim 3.4 \times 10^{-3}$ $\sim 5.4 \times 10^{-3}$ $\sim 8.8 \times 10^{-3}$	-	0.086 0.086 0.052	80 40 24		TOF 145° 90° 60°

GPPD, IPNS	~2.6x10 ⁻³ ~4.9x10 ⁻³ ~7.6x10 ⁻³	-		144 total		TOF 148° 90 60
HB-4, ORNL	~2x10 ⁻³	2 x 10 ⁵		32		70° takeoff
BT-1, NIST	~0.8 x 10 ⁻³	1 - 7 x 10 ⁵		32		75,90,120° takeoff
G4-2 HRPD, LLB	~2x10 ⁻³			70		112° takeoff
D1B, ILL	~2x10 ⁻³ (check)	0.4-6.5 x 10 ⁶		400 cells 1545mm s-d		44o, PG, Ge
D1A, ILL	~10x10 ⁻³	2 x 10 ⁶		25 detectors 650mm s-d	0.04Hz bkg	120° takeoff 25-30cm high
Polaris, ISIS	~ 30x10 ⁻³		0.009sr	80	0.5-21Å, 14°	TOF
	~ 10x10 ⁻³		0.046sr	80	0.5-8.15Å, 35°	
	~ 7x10 ⁻³		0.48sr	216	0.3-4.1Å, 90°	
	~ 5x10 ⁻³		0.29sr	58	0.2-3.2Å, 150°	

Special Purpose

<i>Instrument</i>	<i>Resolution (Dd/d)</i>	<i>Flux</i>	<i>Steradians</i>	<i>Detectors</i>	<i>Range</i>	<i>Comments</i>
G4-1, LLB	?	1.5 x 10 ⁶		800 cells	PG 42-110°	focus on magnetic

Appendix E: Web Sites (Links active)

Select Instrument Web Sites:

ILL	http://www.ill.fr/YellowBook/
PSI	http://www1.psi.ch/www_sinq_hn/SINQ/instr/HRPT.html
FRM-II	http://www.frm2.tu-muenchen.de/e/instrumente/spodi/rahmen.html
ISIS	http://www.isis.rl.ac.uk/crystallography/
ANL	http://www.pns.anl.gov/IPNSINST.HTM
ORNL	http://neutrons.ornl.gov/NSatHFIR/HFIRNSFac.HTML
NIST	http://www.ncnr.nist.gov/
LLB	http://www-llb.cea.fr/index_e.html
HIFAR	http://www.ansto.gov.au/ansto/neut/index.html

Other Web Sites:

AUSTRON	http://www.austron.at/
OECD	http://www.oecd.org/dsti/sti/
ICSD	http://icsd.ccp14.ac.uk/icsd/