Future perspectives for liquids and amorphous materials diffraction at ISIS

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ABSTRACT: A review is given of the current state of neutron diffraction from liquids and amorphous materials at ISIS. In particular the justification and status of the SANDALS diffractometer, which is now undergoing detailed design and construction, is reviewed, and compared with competing diffractometers in Europe and the U.S.A.. A general description of this instrument is included.

1. Introduction

In the last three years the proton current at ISIS has increased steadily so that it now operates routinely at 100μA. Although this is still below the design current of 200μA, it has become apparent that the majority of working neutron instruments are performing at least as well as originally planned and in some cases much better than expected. For liquids and amorphous materials diffraction the Liquids and Amorphous Diffractometer (LAD) was originally designed as the main workhorse for structure factor (S(Q)) measurements, and it has produced high quality datasets on such diverse materials as deuterium gas, superionic glasses, molten salts, molecular liquids, and aqueous solutions. Figures 1 and 2 show the measured structure factor and pair correlation function, g(r), for amorphous boron as measured on LAD (R G Delaplane and U Dahlborg, 1987, unpublished data). Table I lists the experiments accomplished since May 1988. Recently the solid angle of the lower scattering angle detector banks on LAD was increased by a factor of 8 and this, combined with the high proton currents, means that more difficult experiments such as isotope substitution experiments have been tackled successfully.

ISIS and therefore LAD have come rather late in the field of disordered materials diffraction because intense reactor neutron facilities have been available in Europe since the early 1970's for this type of work. The ILL at Grenoble and the Orphée reactor at Saclay, both in France, have high count rate liquids instruments available, and several other European institutions have useful albeit lower flux facilities. Additionally in the time it has taken to bring ISIS on line liquids diffraction facilities at the ILL have undergone several upgrades with substantial rises in count rate. In the case of liquids or amorphous materials, where structural features are intrinsically broad the most difficult experiment that can be attempted correlates directly with the number of neutrons per measuring bin accumulated in the course of an experiment. ILL with its diffractometers D4B and D20, has generally taken the lead in terms of count rate, it being routine to obtain 10^7 counts in a Q-bin of width of say 0.05Å^{-1} in a few hours of running time. As a result there have

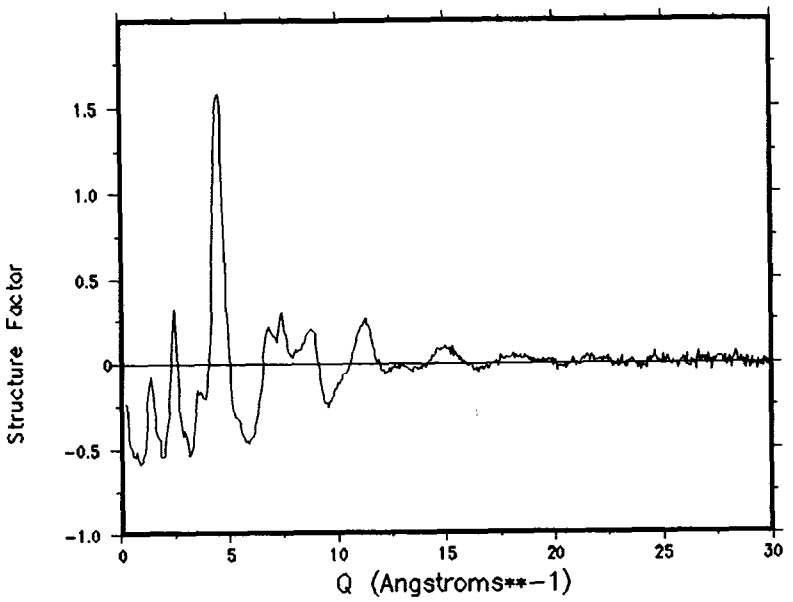

Figure 1. Measured structure factor for amorphous boron on LAD at room temperature.

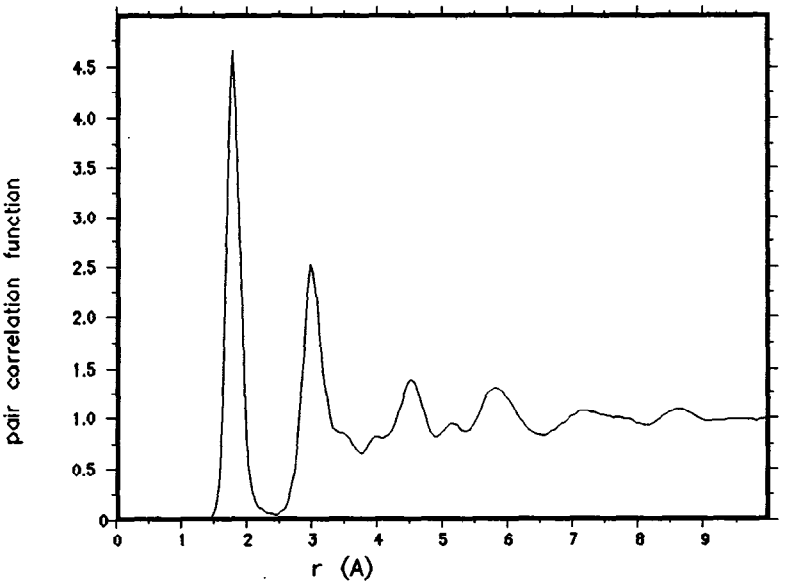

Figure 2. Calculated pair correlation function for amorphous boron, based on the structure data of figure 1. The coordination number of the first peak is 6, indicative of the icosahedral packing which has been proposed for this material.

been several significant achievements in liquid and amorphous diffraction at ILL: perhaps the most important contribution is that isotope substitution has become a routine technique for probing complex materials. It has for example revolutionized the study of ions in melts and aqueous solutions.

TABLE I Experiments on LAD May - September 1988

Investigator	Institution	Sample
Wood/Howe	Leicester University	molten $KCl/ZnCl_2$
Borjesson/Torell	Chalmers U., Sweden	superionic glasses
Wright/Sinclair	Reading/Harwell	Pb-germanate glasses
Neilson/Sandstrom	Bristol/Stockholm	Cr-perchlorate solutions
Dupuy	Lyon, France	$LiCl.6D_2O$ glasses
Skipper	Oxford	$Ni\text{-}vermicullite.3H_2O$
Fontana	Parma, Italy	Cu-nitrate solutions
Dahlborg	Stockholm, Sweden	iso-propanol glasses
Bermejo	Madrid, Spain	butane, methanthiol
Burgess	ICI	battery polymer
Orton	Brunel	molten antimony
Neilson/Adya	Bristol	molten ammonium nitrate
Yamaguchi	Fokoda, Japan	lathanide perchlorate solutions

Therefore the case for using a pulsed source for diffraction on liquids and amorphous materials is based not on count rate, at which reactor sources have traditionally excelled, but on two instrinsic weaknesses of the reactor experiment which are unavoidable. First of all the region of Q over which it operates, typically in the region of $0.4Å^{-1}$ to 17 $Å^{-1}$ for D4B (wavelength = 0.7Å) is always finite. This range can be extended by using several wavelengths but that reduces the effective count rate by a factor of 2 or 3. Also the reactor experiment necessarily involves a scan in scattering angle at fixed incident energy. This means that recoil or Placzek effects (Placzek 1952) deteriorate with increasing Q value resulting in great controversies about to how to cope with the corrections. (See for example the various attempts to measure the partial structure factors in liquid water: Thiessen and Narten, 1982; Soper 1984; Dore, 1985)

For time-of-flight diffraction, which uses fixed scattering angles, the range of Q values available is much broader, (for example LAD has a range in Q from $\sim 0.15Å^{-1}$ to $>50Å^{-1}$) and the recoil correction is nearly independent of Q at small scattering angles (see figure3). It will be noted that the correction is particularly small for scattering angles below 20°.

The LAD diffractometer at ISIS has detectors at scattering angles of 5°, 10°, 20°, 35°, 58°, 90° and 150°, and resolutions ($\Delta Q/Q$) at 20° of ~2% and at 150° ~0.5%, Howells (1980). These are certainly good for most applications concerning disorderd structures. In fact in the past LAD has doubled as a good medium resolution powder diffractometer. In addition the background is exceptionally low, the beam-on, no-sample count rate being essentially zero, due to the high degree of collimation in the scattered beam. Figure 4 shows a diagram of the instrument.

However this high degree of secondary flight path collimation ultimately limits the usefulness of LAD because it imposes detector solid angle

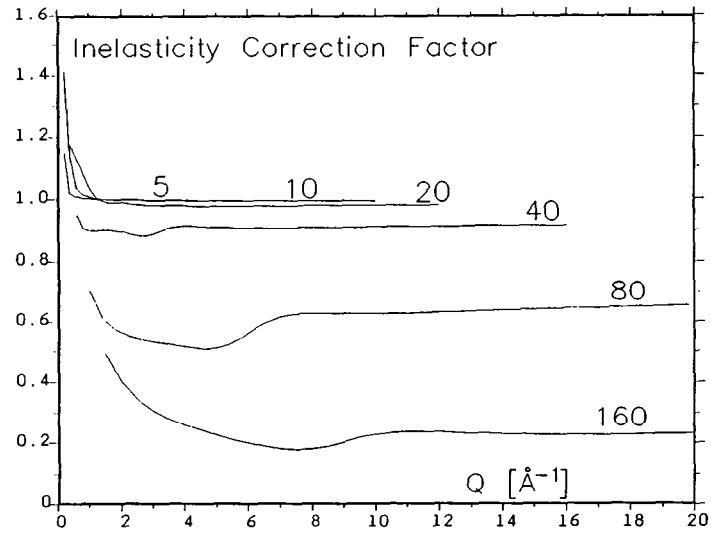

Figure 3. Calculated single atom scattering for a free particle of mass 2 at 40K. The number on each line refers to the scattering angle.

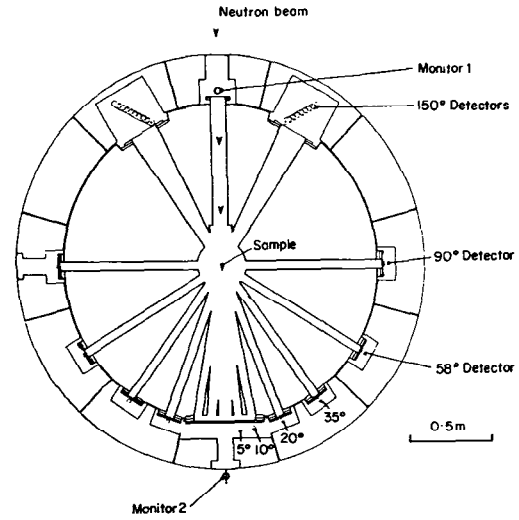

Figure 4. Layout of LAD. The unshaded regions between scattered flight tubes is filled with shielding wax. The solid angle of two 20° banks combined is ~0.02 sr.

constraints which mean the count rate is only as good as the ILL diffractometers over a rather limited range of Q near $Q=1\text{Å}^{-1}$. (See the discussion in Section 3 and Figure 5.) Therefore several years ago a proposal was made to complement LAD with a Small Angle Neutron Diffractometer for Amorphous and Liquid Samples (SANDALS) which would emphasize the small angle scattering region. By doing so it would reduce even further the smallest Q attainable to approximately 0.05Å^{-1} (this is crucial for experiments, such as those aiming at determining the pair potential, where accurate pair correlation functions are required), at the same time as reducing recoil (or Placzek) corrections by performing the whole diffraction measurement at small scattering angles. Subsequently two further conditions were established: to be viable SANDALS would have to be very competitive with ILL in terms of count rate. Also there has been a long-standing interest in the possibilities of exploiting the anomalous dispersion of the neutron scattering length near a nuclear resonance to tackle the problem of extracting partial structure factors from multicomponent systems: this would require a continuous span of detectors with scattering angle. These added requirements have necessitated several redesigns, but the various ideas have now converged to a final design which is currently under construction. The present schedule calls for the main detector tank and part of the detector bank to be in operation by the end of 1989.

This article therefore is devoted for the most part to a review of the ideas that have lead to the final SANDALS proposal.

2. Tests with the SANDALS prototype

Before final design of SANDALS could proceed it was necessary to check certain key aspects of the instrument, in particular the detector performance, the beam collimation and the count rate, and also to gain experience with data analysis with a large number of detectors. The original proposal called for a 14m flight path and since much of the collimator and beam stop had already been purchased these were installed on the beamline. A tank (volume 1x1x3m) from the NIMROD accelerator was filled with argon and used as a detector tank: the detectors were two glass scintillator optically encoded modules, formerly from the LOQ instrument, making a total of 1120 detectors. These detectors subtended scattering angles of 4° - 8° and 11° - 26° respectively at the sample position. In addition several other detector types and configurations were tested. One experiment, on the water correlation functions in concentrated solutions of urea, was completed, Finney, Soper and Turner (1988).

The prototype produced the expected count rate based on known moderator parameters. However backgrounds were quite severe at high neutron energies (>1eV), particularly at the smallest scattering angles. Comparison with other ISIS instruments indicated that there was a similar problem although much scaled down on LAD at 5° scattering angle and also on HET (with both choppers removed) at small scattering angles. In all three cases the problem appears to be related to the fact that the B_4C used in the collimator becomes a partial scatterer of neutrons and is less efficient at neutron capture at high neutron energies. As a result since the small angle detectors are difficult to shield they may view this B_4C and the background problem becomes exacerbated. Therefore careful attention has been paid in the SANDALS design to the final collimation stage.

The other main achievement of the prototype was to test various detectors. On the assumption that it would be prohibitively expensive to build the final detector bank from ^{3}He tubes, then the only practical alternative was scintillator detectors, which could be built for a fraction of the cost of ^{3}He

tubes. Because they have not been tested nearly so extensively in neutron scattering applications, scintillator detectors inevitably require a longer development time. Two types of scintillator detector have been tested: glass scintillator and zinc sulphide scintillator. Both rely on ^{6}Li for initial neutron capture. The former have an intrinsic deadtime on the order of 100ns, but have a high background, on the order of 0.5cts/s/cm^2, and more importantly cannot be made completely γ-insensitive. The latter property turns out to serious for liquids and amorphous solids diffraction since many potential samples are likely to be sources of γ radiation when in a neutron beam. It is particularly serious for SANDALS where the demand for high efficiency detectors means thick scintillators are needed.

On the other hand the zinc sulphide scintillator which is built from a sandwich of scintillator materials between glass sheets to assist light extraction, had γ sensitivity only marginally higher than a ^{3}He tube and had a very low intrinsic background. Its deadtime was on the same order as a ^{3}He tube. However the count rate in an individual module on SANDALS is unlikely to be sufficient that dead time would be important for these detectors. The zinc sulphide detector has the important advantage that it can be made more efficient than a ^{3}He tube at epithermal energies: a module 20mm deep can be made ~30% efficient at a neutron energy of 10eV, which is twice as efficient as the corresponding ^{3}He tube. Therefore it was to decided to employ the zinc sulphide scintillator for SANDALS.

3. Count rate calculation and comparison of instruments

For liquids and amorphous materials diffraction, irrespective of whether it is constant wavelength or time-of-flight diffraction, the structure factor S(Q) is measured versus the momentum transfer, $hQ/2\pi$, where for elastic scattering

$$Q = 4\pi \sin\theta / \lambda \qquad (1)$$

and 2θ is the scattering angle and λ the neutron wavelength. Because the features in S(Q) are rather broad (compared to crystalline powder diffraction) the requirements for resolution are relatively relaxed, but an adequate count rate can be crucial to obtaining a useful result, particularly for those experiments which involve differencing datasets as a function of pressure, temperature, isotope, etc. In these cases the differential behaviour is usually more important than the total scattering pattern. Therefore count rate is almost always the primary quantity of interest. Typically the data for S(Q) are mapped out in bins of width 0.05Å^{-1}, and the quantity of interest in rating the performance of a diffractometer is therefore the count rate per Q-bin per unit volume of standard scatterer which is normally vanadium:-

neutrons / s / 0.05Å^{-1} / cm^3 of vanadium (2).

This definition serves to normalize out differences between instruments which are purely geometric in origin, (usually the size and shape of the beam at the sample position). This number is also useful to know for a given diffractometer: a rough estimate of the count rate for a given sample can be obtained by multiplying it by the volume of sample times the ratio of sample scattering cross section to vanadium scattering cross section. It would be helpful therefore if this number could be specified for all liquids diffractometers so that realistic intercomparisons could be be made. For convenience I shall refer to the count rate number according to (2) as the "C-number" for a given diffractometer.

For reactor experiments the count rate is almost independent of Q, but for pulsed sources the spectrum falls as 1/Q in the epithermal region. In addition the detector efficiency is proportional to $\lambda \sim 1/Q$, so the measured count rate falls as $1/Q^2$. In practice efficiency corrections mean that the intensity falls more like $1/Q^{1.7}$, but even so there is a dramatic fall in measured count rate with increasing Q, as shown in figure 5, where the measured spectrum for LAD at a scattering angle of 20° is displayed.

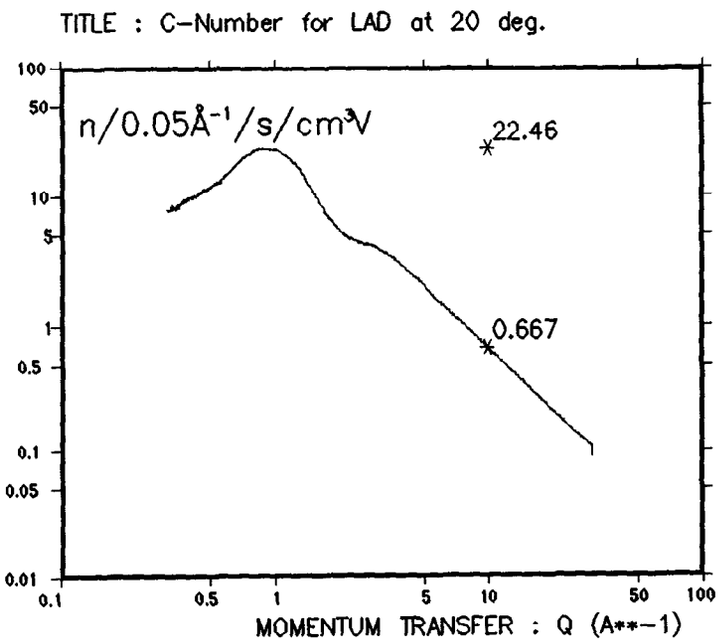

Figure 5. Measured count rate (or "C-number" - see text) for LAD at 20° scattering angle as a function of Q. Note the log axes.

For the methane moderator at ISIS, the parameter that describes the epithermal flux is Φ_0 and at 100μA proton current and 750 MeV energy, this has the value for a moderator area of 100 square cm, Taylor (1984)

$$dI/dE = \Phi_0 = 2.7 \times 10^{12}/E^{0.92} \text{ n/eV/sr/100cm}^2\text{/s} \quad (3).$$

Now $E \sim Q^2$ so $dI/dQ = 2(E/Q)dI/dE \simeq 5.4 \times 10^{12}/Q$ n/Å^{-1}/sr/100cm2/s

$$= 2.7 \times 10^{11}/Q \text{ n/0.05Å}^{-1}\text{/sr/100cm2/s} \quad (4).$$

Using these values the expected count rate on LAD can be estimated. It is assumed that:-

(i) the collimator views most of the active area of moderator;
(ii) the incident flight path is 10m;

(iii) the sample is a cube of vanadium, 10 x 10 x 10mm^3 in volume (corresponding to a 30% scatterer);
(iv) detector is 30% efficient (corresponds to a ^{3}He tube at 2eV, and $Q \simeq 10\text{Å}^{-1}$ for 20° scattering angle);

then the scattered count rate per unit detector solid angle is

$$\underset{\substack{\uparrow\\ \text{moderator}}}{2.7 \times 10^{11}} \times \underset{\substack{\uparrow\\ \text{fraction scattered by sample}}}{0.3} \times \underset{\substack{\uparrow\\ \text{sample solid angle at moderator}}}{1.0 \times 10^{-6}} \times \underset{\substack{\uparrow\\ \text{detector efficiency}}}{0.3 / 4\pi Q}$$

$$= 1940/Q \text{ n/0.05Å}^{-1}\text{/sr/s/cm}^3\text{V}. \qquad (5)$$

It will be noted that this number, which of course applies strictly only to the epithermal region of the spectrum, is **independent** of scattering angle for a given Q value. The fact that count rates vary for different scattering angles and different Q values in practice arises because the thermal part of the spectrum eventually takes over at low neutron energies and in any case the detector efficiency will be different for the same Q value at different scattering angles. For LAD at $2\theta = 20^\circ$ the detector area is 2 x 0.04 x 0.2m^2 and the final flight path is 1.0m, and so for $Q \simeq 10\text{Å}^{-1}$ the C-number is **3.1n/0.05Å^{-1}/s/cm^{3}V**. I quote the C-number at $Q = 10\text{Å}^{-1}$ since it is important to remember the very rapid decline in count rate at a pulsed diffractometer with increasing Q value.

Figure 5 shows that the measured C-number is much lower at **~0.7n/0.05Å^{-1}/s/cm^{3}V** for this Q value (and **~22n/0.05Å^{-1}/s/cm^{3}V** at $Q = 1\text{Å}^{-1}$), and the reason for this disagreement is not clear at the present time. It should be born in mind of course that for most experiments several LAD detector banks can be combined so that the count rate should be multiplied by a factor of ~2-3 to get a realistic estimate of likely count rates. Even so the very rapid fall in count rate with increasing Q is clear from figure 5.

For the new glass diffractometer at IPNS, GLAD, the estimated C-number assuming a full complement of detectors at the same Q value is **~20 - 25n/0.05Å^{-1}/s/cm^{3}V** at $Q = 10\text{Å}^{-1}$, Montague and Price (1988), which includes the factor of 2.5 enhancement which has taken place since the booster target was installed.

Finally for the D4B diffractometer at ILL the measured neutron flux on the sample is 4 x 10^7 n/cm^2/s for a wavelength of 0.7Å, so the C-number for the standard cube of vanadium would be

$$\underset{\substack{\uparrow\\ \text{flux}}}{4 \times 10^7} \times \underset{\substack{\uparrow\\ \text{fraction scattered by sample}}}{0.3} \times \underset{\substack{\uparrow\\ \text{detector solid angle for } 0.05\text{Å}^{-1} \text{ bins}}}{5.5 \times 10^{-4}} \times \underset{\substack{\uparrow\\ \text{detector effic.}}}{0.67} \times \underset{\substack{\uparrow\\ \text{sampling factor}}}{0.15 / 4\pi}$$

$$= 53 \text{ n/0.05Å}^{-1}\text{/s/cm}^3\text{V}. \qquad (6)$$

The sampling factor arises here because the present detector does not scan all scattering angles simultaneously. This number is entirely in accord with the observed count rate from a sample of vanadium placed in the neutron beam on D4B (A C Barnes, 1988, private communication). Comparison of this C-number with figure 5 shows that when several angles are combined the LAD diffractometer is already as intense as D4B, but only in a narrow region of Q, around $Q \approx 1 Å^{-1}$. Outside this region the intensity falls off rapidly.

4. Design of SANDALS

The count rate numbers of the previous section tell their own story: whatever the cause for the measured count rate on LAD being lower than the number based on moderator performance figures there was no avoiding the conclusion that the count rate on LAD was inherently lower than on competing diffractometers over a range of important Q values, i.e. $Q = 5Å^{-1}$ to $20Å^{-1}$. This was a key result which has directed the design of SANDALS. The principal goal has been to strive for the maximum solid angle of detector in the "small" angle region (i.e. for $2\theta < 40°$), with a continuous span in scattering angle, that can be achieved within the engineering constraints imposed by allowed sizes of vacuum tanks and windows and the restrictions imposed by including some shielding in the scattered flight path. As a result the available solid angle will be approximately 40% of the theoretically maximum. As seen in table II this still will ensure that SANDALS is highly competitive in count rate if the full detector complement is available.

TABLE II Some Design Specifications for SANDALS

Moderator:	Methane, 100K
Incident Flight Path:	11m
Beam Cross Section:	Circular
Maximum Beam Aperture:	32mm (diameter)
Final Flight Path:	0.75m - 4.0m
Detectors:	Zinc sulphide sandwich detectors 200 (high) x 10 (wide) x 2 (deep) mm 30% efficient at 10eV

Range In 2θ	Detector Solid Angle	High Resolution		Low Resolution	
		Resolution	C-number	Resolution	C-number
(deg.)	(sr)	$\Delta Q/Q$ (%)	(at $Q=10Å^{-1}$)	$\Delta Q/Q$ (%)	(at $Q=10Å^{-1}$)
3 - 11	0.043	11 - 2	0.3	16 - 4	7
11 - 21	0.121	2	1.6	4 - 3	35
19 - 31	0.222	1.5	3.9	3	84
29 - 41	0.301	1.3	6.1	2.5	131

The projected count rates have been achieved by shortening the original proposed flight path from 14m to 11m, by opening up the beam from 10mm diameter to 32mm diameter and by increasing the detector solid angle from the original SANDALS proposal (Appendix to NBRC 9-85, 1985). Of course there is a penalty in resolution that has been paid in doing so, although this is not likely to be serious for the high count rate, isotope substitution diffraction work which is likely to feature very frequently in the SANDALS experimental program. However since there will undoubtedly be some experiments which require better resolution than the default resolution, provision is being made to narrow the beam and view a smaller area of moderator if so desired, by placing beam defining apertures of 24mm and 16mm at 6.25m and 9m from the target respectively. Although these will give lower count rates, they will double the resolution in the small angle region. As a further provision the sample tank will include windows for detectors at larger scattering angles up to 120° in the event that better resolution is needed in the future.

An obvious problem that arises at small angles when dealing with large arrays of detectors is that the resolution can vary sharply with scattering angle, which can make combining detectors from different angles problematic if resolution effects are apparent. Therefore a further feature of the design is that the detectors lie on a trajectory of continuous and nearly constant resolution. At small scattering angles this trajectory corresponds approximately to the surface of a cylinder whose axis is coincident with the transmitted beam. With this geometry the resolution varies by a factor of ~2 over the scattering angle range 10° - 40°.

Table II lists the principal characteristics of the proposed SANDALS diffractometer and figures 6 and 7 show two views of the sample vacuum tank. It will be noted that the reduction of count rate from low resolution to high resolution options is on the order of a factor of 20: this is because the resolution of the instrument at small angles is dominated by the angular divergence of the incident beam. The only way to improve this resolution is to restrict the aperture of the beam with a consequent large reduction in count rate. To build the "ideal" geometry with equal contributions to the resolution from moderator, sample and detector would require a much a larger area of detector than the present proposal, would increase the linear dimension of the instrument by a factor of at least 1.5, would lead to increasing difficulties with frame overlap at the ISIS repetition rate of 50Hz, and would escalate the cost significantly above the present allocation. To compensate for the increased cost there would have to be reduced solid angle coverage which would correspondingly negate the advantages of the larger instrument. It is felt that the present design is probably optimal given the conflicting demands of count rate, resolution, cost and engineering constraints. It should also be noted that in the high count rate/low resolution mode SANDALS will remain competitive in count rate with ILL even if further proposed modifications to D4B are implemented.

Outside the vacuum tank the scattered flight path to the detector modules will be filled with argon gas to reduce air scattering. The gas will be contained in boxes lined with B_4C baffles to reduce neutron backgrounds from sources other than the sample. The detectors themselves will be surrounded in B_4C (except in the direction of the sample!) Finally the entire instrument will be entombed in wax shielding to remove external sources of background. It is anticipated that the full array of sample environment equipment which is used on the other ISIS instruments will be available for use on SANDALS as well.

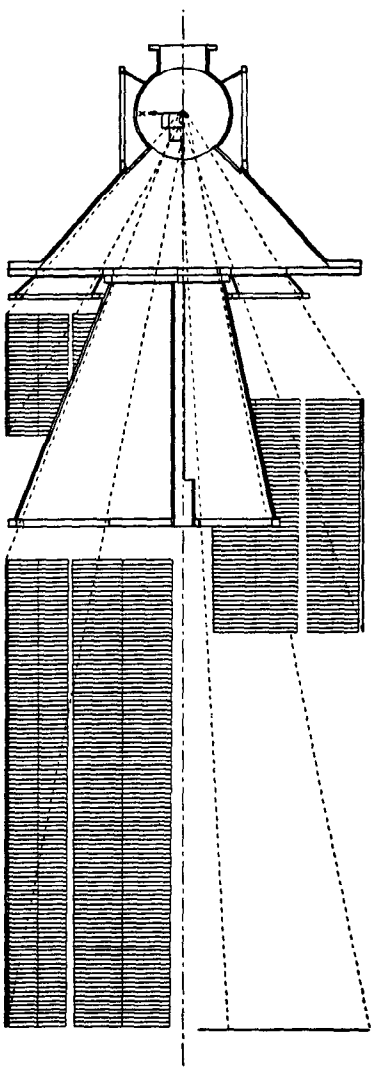

Figure 6 Plan layout of the SANDALS sample tank and detectors. The beam enters the tank at the top of the diagram and leaves at the bottom, and the sample is placed at the centre of the circular tank. The distance from sample to small angle detector is 4m, and the other detector banks are 0.75m from the transmitted beam axis.

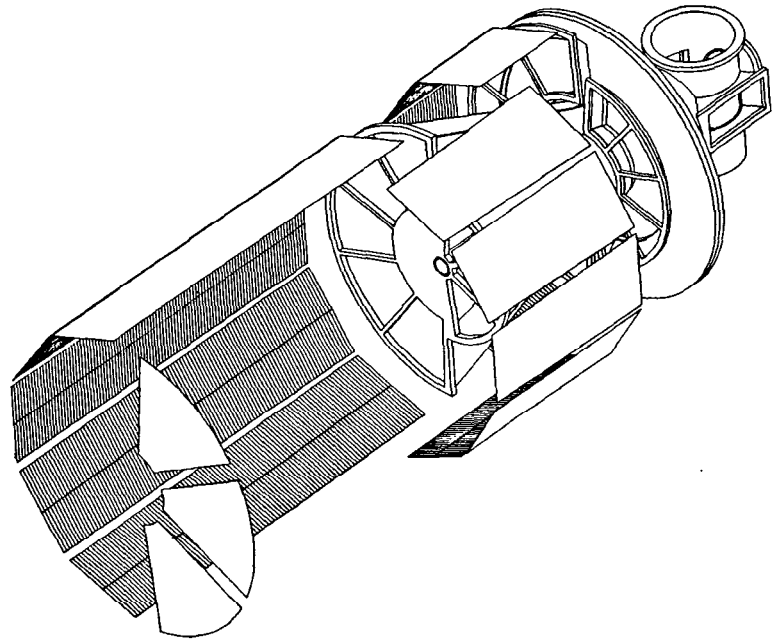

Figure 7 Three-dimensional view of the SANDALS detector tanks. The beam enters at the upper rightmost corner of the diagram and exits at lower left. The scintillator detectors lie on the surface of a cylinder of radius 0.75m.

5. Conclusion - Beyond SANDALS

The availability of high count rate diffractometers has increased the complexity of systems that can be investigated. This complexity increases roughly as the square root of the count rate and so there will in the future, as count rates are pushed even higher, be a trend to look at more technologically interesting materials, such as liquids under extremes of pressure and temperature, local coordination in dilute mixtures, complex molecular fluids, and fluids at surfaces. For example the structural changes which occur near the glass transition are real but rather subtle and would greatly benefit from the detail possible with isotope substitution. In this sense therefore the field of liquid and amorphous material diffraction is unlimited in scope. However the limiting factor at the present time is not count rate, but detector stability and sample preparation: count rate is only useful if the sample is good enough to withstand the precise investigation possible with higher statistics and the detector efficiency fluctuations over the course of an entire experiment are no worse than the statistical precision. It is not clear whether either condition is being met for the instruments presently available, although it is likely that as count rates improve still further they will necessarily drive a demand for better characterised samples and more stable detectors.

The data analysis stage is also crucial, and whilst it is essentially routine to proceed from measured diffraction data to a reliable pair correlation function, this is really only the first stage of the experiment: the real job is to interpret the correlation functions. At present the only practical way to achieve this is to computer model the system under investigation because the process of going from assumed potential function to correlation functions is extremely non-linear. Current methods centre mostly on assuming pair-wise forces, a serious limitation which will have to be removed in the future. Since the process of data analysis yields advances over a period of time it likely that the techniques of instrument development and data handling will develop concurrently.

If the past is an indication of the future then it is clear that worldwide there has been been a continuing interest in the structure of the fluid and amorphous states for many years now. This interest is fueled by unresolved fundamental and technological issues. Neutron diffraction is therefore likely to remain an important tool in the rather large array of techniques that can be applied to this problem because it yields accurate, absolute values for the underlying correlation functions.

In this article I have attempted to outline the existing "state of the art" for liquids and amorphous diffraction at ISIS, and the reasons for having adopted the current specification for SANDALS. It is clear that the proposed instrument will probably make optimal use of the present ISIS neutron source. It will form a unique facility for liquids and amorphous diffraction by providing a wider range of Q values, and by reducing recoil corrections, compared to equivalent reactor based instruments. At the same time the count rate on SANDALS will be highly competitive with other liquids diffraction facilities around the world.

Because SANDALS will fully exploit the current ISIS neutron source, the next generation of liquids diffractometers must look towards a revised source and moderator configuration. Assuming the cost of building a diffractometer is not the limiting factor the primary constraints imposed by the present source for this work are the repetition rate, which as discussed above leads to an instrument which is too small to make full use of the available neutron flux, and the neutron pulse width, which currently makes a negligible contribution to the resolution. Increases in both resolution and count rate could be achieved with a lower repetition rate source, and a moderator which produces broader neutron pulses.

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