# Time-of-Flight Diffraction at Pulsed Neutron Sources: An Introduction to the Symposium

James D. Jorgensen
Materials Science Division
Argonne National Laboratory
Argonne, IL 60439



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# Time-of-Flight Diffraction at Pulsed Neutron Sources: An Introduction to the Symposium

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Materials Science Division, Argonne National Laboratory, Argonne, IL 60439

In the 25 years since the first low-power demonstration experiments, pulsed neutron sources have become as productive as reactor sources for many types of diffraction experiments. The pulsed neutron sources presently operating in the United States, England, and Japan offer state of the art instruments for powder and single crystal diffraction, small angle scattering, and such specialized techniques as grazing-incidence neutron reflection, as well as quasielastic and inelastic scattering. In this symposium, speakers review the latest advances in diffraction instrumentation for pulsed neutron sources and give examples of some of the important science presently being done. In this introduction to the symposium, I briefly define the basic principles of pulsed neutron sources, review their development, comment in general terms on the development of time-of-flight diffraction instrumentation for these sources, and project how this field will develop in the next ten years.

## PULSED SPALLATION NEUTRON PRODUCTION

It was recognized as early as the 1940's that nuclear reactions that occur when energetic beams from accelerators struck an appropriate target material offered an alternative to fission for the production of neutrons for research purposes.[1,2] Some of the earliest of such experiments were done with electron beams, which produce neutrons by a two-stage process via high-energy gamma rays. Present day pulsed neutron sources, however, are based on the spallation process, which takes its name from the verb to spall, which means to splinter or chip.[3,4] Spallation of a nucleus occurs when the accelerated particle has mass comparable to nuclear particles, and a high enough energy. Relativistic proton beams are used in the pulsed neutron sources now operating. When the high-energy proton collides with a target nucleus, recoiling nucleons and pions collide with other nuclei before emerging. In this way, up to 30 neutrons per incident proton can be produced, depending on the proton energy and the atomic weight of the target nuclei.

Fig. 1 shows the components of a typical spallation neutron source.[4] Protons are accelerated in either a circular or linear accelerator and then directed at a heavy metal target. Since the total penetration depth of the proton beam in the target does not exceed a few tens of centimeters, the target is of rather modest dimensions (e.g., a cylinder approximately 10 cm in diameter and 21 cm long for the IPNS at Argonne). The neutrons produced by the spallation process have high energies, extending up to the energy of the incident protons, and are not useful for scattering experiments. They must be slowed to lower energies. For diffraction experiments, the desired neutron wavelengths correspond to thermal energies. This slowing process is called moderation. Appropriate moderators are placed around the spallation target.[3] In the moderators, the neutron energies are reduced by repeated inelastic collisions in which the neutrons lose energy.[5] Hydrogenous materials are most commonly used because of the large inelastic cross section of hydrogen. The temperature of the moderator controls the energy distribution of the moderated neutron beam. Thus, typical moderator materials include polyethylene or water at room temperature and liquid hydrogen, liquid methane, or solid methane if "colder" neutrons (i.e., neutrons with longer wavelengths) are desired.

If beam were extracted from the accelerator to the target on a continuous basis, a steady state neutron beam would be produced. However, it is more efficient to operate accelerator based neutron sources in a pulsed mode. Protons are accumulated in the accelerator and extracted in short, intense bursts, producing intense pulses of neutrons. With proper control of pulse width and repetition rate, the neutron production can be matched to the needs of time-of-flight diffraction instrumentation. Thus, every neutron that is produced is used and the neutron source is off when neutrons are not needed. It is possible to produce peak neutron fluxes (i.e., the flux during the pulse) equal to or higher than the peak fluxes available at the most powerful reactors, with only a small fraction of the average flux and, thus, only a small fraction of the total heat generation.[6]

The production of pulses for time-of-flight diffraction experiments can be compared to the production of a small band of wavelengths (through the use of a monochromator) for angle-dispersive diffraction experiments at reactor sources.[1] The advantage of the pulsed source is that the source is off between pulses, as compared to reactor which produces a broad spectrum of wavelengths, even though only one narrow band of wavelengths is being used. Because of this, for the same peak flux the heat that must be dissipated is orders of magnitude higher for the reactor than for the pulsed source. Ultimately, it is the ability to handle this overall power density that limits the neutron flux that can be obtained from reactor sources. For this reason, it was recognized early in the development of pulsed neutron sources that, with proper accelerator, target, and moderator design, the peak neutron flux available at pulsed sources would eventually surpass the practical limit of what could be done at reactors. Fig. 2 shows the effective thermal neutron flux as a function of the year of beginning operation for a number of reactor and pulsed neutron sources.[7] It is clear that reactor design has been near the practical limits for a number of years, while the performance of pulsed sources is continuing to increase at an impressive pace. The ISIS pulsed source at the Rutherford Appleton Laboratory in the U. K. already produces a peak flux that exceeds the most powerful reactors and future pulsed sources promise further significant increases.

For some inelastic scattering experiments, the ability to utilize this peak flux depends on the nature of the experiment. Thus, reactors will continue to offer advantages for such experiments even though pulsed sources offer higher peak flux.[6] However, for most diffraction experiments the peak flux of the pulsed source is fully utilized and the higher peak fluxes projected for future pulsed sources will open new horizons for diffraction experiments. The advanced state of diffraction experiments being done on present pulsed sources confirms this prediction.

## DEVELOPMENT OF PULSED NEUTRON SOURCES

The use of accelerators to produce pulsed neutron beams for neutron scattering studies was suggested by Alvarez[8] in the late 1930's and Cockcroft[9] in the 1940's. Egelstaff[10] and others pointed out in the 1950's that one advantage of this technique should be lower backgrounds, because the neutron source would be off most of the time. However, even though accelerator-based neutron sources were used for various nuclear physics experiments (e.g., cross section measurements) during these years, the first meaningful diffraction experiments were not done until later.

For effective neutron scattering experiments, high neutron fluxes were needed. This required high accelerator currents and properly designed target/moderator systems. Meaningful progress on accelerator-based neutron sources for neutron scattering experiments was reported in the late 1960's. Simple time-of-flight diffraction experiments were done as part of some of the first tests of accelerator-based pulsed neutron sources, such as those based on electron linear accelerators at Rensselaer Polytechnic Institute[11] and Tohoku University[12]. A diffractometer operated at the Harwell Electron Linac Source in 1974 demonstrated that high-quality powder diffraction data, suitable for Rietveld refinement, could be obtained at pulsed sources.[13] In the same year Argonne National Laboratory began the operation of an experimental spallation pulsed neutron source called ZING using beam from a proton synchrotron that was the injector for a high-energy physics machine called the Zero Gradient Synchrotron (ZGS).[14] Even though the neutron fluxes were relatively low, a substantial amount of valuable experience in target/moderator design and time-of-flight instrument design was obtained from these early experiments.

It was clear from the prototype experiments that the concept of accelerator-based neutron production for scattering experiments was viable and several laboratories began construction of spallation pulsed neutron sources in the late 1970's. The history of this rapid development is summarized in Table I. Over a period of less than ten years, proton beam current increased a factor of forty, resulting in peak neutron fluxes as high as the most powerful reactors. This rapid development was made possible by the advances in accelerator design that had been achieved with facilities that served the nuclear physics and high-energy physics communities.

All of the facilities listed in Table I used accelerator hardware and expertise associated with such facilities. The ZING-P' source at Argonne National Laboratory used the linear accelerator and rapid cycling proton synchrotron that had been constructed to serve as an injector to the Zero Gradient Synchrotron. The rapid cycling synchrotron was later upgraded to provide 15 μA beam current for the Intense Pulsed Neutron Source (IPNS). A large instrument hall and other valuable hardware were made available by the closure of the ZGS, allowing IPNS, which has run continuously since 1981, to be constructed for $13M, including instruments.

In similar fashion, the Weapons Neutron Research Facility (WNR) at Los Alamos National Laboratory was operated using part of the beam from the LAMPF proton linear accelerator, whose primary use is for nuclear physics experiments. The later addition of a proton storage ring to allow the proton beam to be stored and then extracted in higher intensity pulses at a more favorable repetition rate allowed the beam current to be increased to 80 μA. At the same time, a new instrument hall, with several new instruments, was added, creating the Los Alamos Neutron Scattering Center (LANSCE).

The KENS facility in Japan uses beam from the 500 MeV injector accelerator of the KEK high energy physics center. Although beam current is relatively low and the space for instruments is very limited, this facility has produced an amazing number of important results and still operates.

The ISIS facility at the Rutherford Appleton Laboratory in the U. K. was the first facility designed to achieve a proton beam current that would allow peak neutron fluxes higher than any existing reactor. ISIS was designed around the site of the 7 GeV Nimrod accelerator that had been closed in 1978 and used the 70 MeV proton linear accelerator, magnet ring enclosure, and experimental hall, but a new 800 MeV proton synchrotron was constructed. Although the design current of 200 μa was not achieved initially, the facility now operates routinely near its rated peak current making ISIS the most intense pulsed neutron source in the world.

## DEVELOPMENT OF TIME-OF-FLIGHT DIFFRACTION INSTRUMENTS AT PULSED SOURCES

The first diffraction experiments at pulsed neutron sources were done for the purpose of demonstrating the feasibility of the technique and investigating instrument design and data analysis concepts. Such experiments began in the late 1960's and continued in the 1970's at facilities in the United States, the U. K., and Japan. The most significant work was done by the groups at Tohoku University[12], Harwell[13], and Argonne National Laboratory[14]. Fig. 3 shows a time-of-flight diffraction pattern from NaCl taken at ZING in 1974.[14] A Rietveld refinement analysis method was used with these data, showing that the asymmetric peak shape characteristic of pulsed-source data was not a serious hindrance to modeling the data accurately.

This early success with time-of-flight powder diffraction led to the design of a truly state of the art diffractometer, the High Resolution Powder Diffractometer (HRPD), for the ZING-P' source which began operation at Argonne in 1977.[15] Using an 18.4 m incident flight path the HRPD achieved resolution of $\Delta d/d \leq 3x10^{-3}$. The performance was comparable to the best diffractometers at reactors. Although the resolution of the D1A diffractometer was slightly higher for a limited range of scattering angles, the HRPD demonstrated the ability of time-of-flight pulsed source diffractometers to achieve nominally constant resolution, $\Delta d/d$, over a wide range a d spacings. The HRPD also demonstrated how time focusing concepts could be used to dramatically increase the detector area with no loss of resolution. Data collection required 12-24 hours for typical samples.

In 1978 Von Dreele finished work on a user friendly Rietveld refinement code for use with pulsed-source time-of-flight data.[16] This code was heavily used during the three years of operation at ZING-P' as well as at the WNR and in the U. K. Dozens of publications on a wide range of topics in solid state physics, materials science, and chemistry resulted. The success of this Rietveld code dispelled any concerns about the difficulty of accurately modeling the asymmetric peak shape and the variation of neutron flux versus wavelength characteristic of the pulsed source.[17]

This early success set the stage for the construction of advanced time-of-flight powder diffractometers that now operate at all of the pulsed sources. The Special Environment and General Purpose Powder Diffractometers (SEPD & GPPD) at IPNS were designed to match the resolution of the HRPD (for the case of GPPD) and to provide multiple scattering angles for coverage of a wider range of d spacings.[18] Electronic time-focusing principles were used to achieve large detector areas at all scattering angles. These instruments have proven especially useful for studies of samples in special environments, where a 90° scattering angle allows optimum collimation.[19] At LANSCE, the Neutron Powder Diffractometer (NPD) achieves resolution of $\Delta d/d=0.15\%$, while the High Intensity Powder Diffractometer (HIPD) provides lower resolution with a much higher count rate. The highest resolution is available on the High Resolution Powder Diffractometer (HRPD) at the ISIS facility in the U. K.[20] This instrument uses an incident flight path of over 100 m to achieve $\Delta d/d=4x10^{-4}$. Competitive count rates are maintained by using a neutron guide along the incident flight path and a large time focused detector in back scattering. This resolution has enabled structure refinements from powder data that rival those obtained from single crystal data.[17,21]

Single crystal diffraction at pulsed sources has enjoyed a similar development. Time-of-flight single crystal diffractometers are based on the Laue method.[1,2,4,22] However, the ability to separate neutron wavelengths by measuring their times-of-flight results in a three-dimensional view of reciprocal space, rather than projecting into two dimensions as is the case for X-ray Laue techniques.[22] Thus, there is no loss of information from overlapping of orders of reflections.

Early demonstration experiments were done at the Tohoku University[23] and Harwell[24] electron linear accelerators. Routinely operating instruments were later installed at all of the pulsed sources.[22,25,26] The development of these instruments relied heavily on the development of suitable position-sensitive detectors -- the important criteria being resolution, accuracy of the position determination, and uniformity of neutron sensitivity. Both scintillation[27] and gas-filled[28] detectors have been used. Because these instruments collect data over large continuous regions of reciprocal space, they have proven to be especially powerful for experiments involving modulated structures, diffuse scattering, or phase transformations where one is not able to predict which regions of reciprocal space will be of interest.

Position-sensitive detectors are also used in time-of-flight small-angle (or low-Q) diffractometers. In this regard, the time-of-flight small-angle diffractometer is very similar in design to constant-wavelength small-angle diffractometers at reactor sources. The difference is in the way data are collected and processed; the time-of-flight instrument uses a white beam with the times-of-flight being used to bin data according to wavelength as well as scattering angle.[29] To reach the lowest possible Q's, small angle diffractometers make use of long wavelength neutrons. One important success has been the development of cold moderators for pulsed neutron sources. Liquid hydrogen[30] and solid methane[31] have been used as very effective moderator materials. Because the heat from the neutron production is much lower for pulsed sources, it has been found that it is easier to produce a cold neutron beam at a pulsed source than at a reactor. This came as somewhat of a surprise since, during the initial development of pulsed neutron sources, much attention was given to the fact that the spallation process offered a way to obtain high fluxes of neutrons at energies higher than are produced by a reactor source. No one had expected that the pulsed sources would excel at both ends of the neutron energy spectrum.

## THE FUTURE OF PULSED SPALLATION NEUTRON SOURCES

The conclusion to be drawn from the success of pulsed spallation neutron sources is that in approximately twenty years since the first large-scale prototype experiments these facilities have taken a place along side reactor sources for neutron diffraction experiments. The effective fluxes of the two kinds of sources are now nominally equivalent for most diffraction experiments, with each type of source offering advantages for certain experiments. For example, pulsed sources are best for diffraction in special samples environments because of the fixed scattering geometry[19] while reactor sources are best when one wishes to search for a weak reflection rather to obtain a full diffraction pattern. The two premier neutron scattering facilities in the world today are the High Flux Reactor at the Institut Laue Langevin (ILL) in Grenoble, France and the ISIS pulsed neutron source at the Rutherford Appleton Laboratory in the U. K. These facilities have comparable effective neutron fluxes and include a number of state-of-the-art neutron scattering instruments capable of serving large user communities. Fig. 4 shows the instrument hall of the ISIS facility which presently contains fifteen instruments.[32]

Whereas reactor sources have not provided a significant increase in neutron flux for a number of years, the flux available from the pulsed sources continues to increase at an impressive rate (see Fig. 2). This results from advances in high-current accelerator designs and target/moderator designs. Proposals now being considered would push the peak neutron flux of the next generation of pulsed sources well beyond what is possible at a reactor. The Advanced Neutron Source (ANS), a new research reactor proposed for construction at Oak Ridge National Laboratory, will provide an increase in neutron flux over the ILL reactor of a factor of five.[33] The European Spallation Source (ESS), a next-generation pulsed source being considered by the European scientific community, would provide an increase in flux of a factor of thirty over ISIS with a beam power of 5 MW.[34] Perhaps even more impressive, the time-averaged flux of the ESS would be equivalent to the ILL reactor. Thus, experiments of the kind that are best done at a reactor could be done at the ESS by simply ignoring the pulsed nature of the source. In the United States, at least two National Laboratories (Argonne and Los Alamos) are preparing designs for a 1 MW pulsed source. If the U. S. Department of Energy proceeds with the construction of both the ANS and a 1 MW pulsed source, qualitatively new capabilities in neutron scattering will be made possible.

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Table I. Major pulsed spallation neutron sources.

| Dates of operation | Facility | Laboratory | Proton beam current (μA) |
|---|---|---|---|
| 1977-1981 | ZING-P' | Argonne (USA) | 5 |
| 1977-1985 | WNR | Los Alamos (USA) | 8 |
| 1980-present | KENS | KEK (Japan) | 5 |
| 1981-present | IPNS | Argonne (USA) | 15 |
| 1985-present | LANSCE | Los Alamos (USA) | 80 |
| 1984-present | ISIS | Rutherford (UK) | 200 |

FIGURE CAPTIONS

Fig. 1. Major components of a pulsed spallation neutron source. (Figure courtesy of J. M. Carpenter.)

Fig. 2. Effective thermal neutron flux of major reactor and pulsed neutron sources as a function of the year of beginning operation. (Figure adapted from Neutron Scattering, edited by K. Skold and D. L. Price, Academic Press, 1986.)

Fig. 3. Rietveld refinement profile for NaCl data collected on the powder diffractometer at Argonne's ZING prototype pulsed source in 1974. Data collection time was approximately 12 hours. (Figure reproduced from Ref. 14.)

Fig. 4. The neutron scattering instrument hall of the ISIS pulsed neutron source at the Rutherford Appleton Laboratory in the U. K. (Figure reproduced from Ref. 32.)

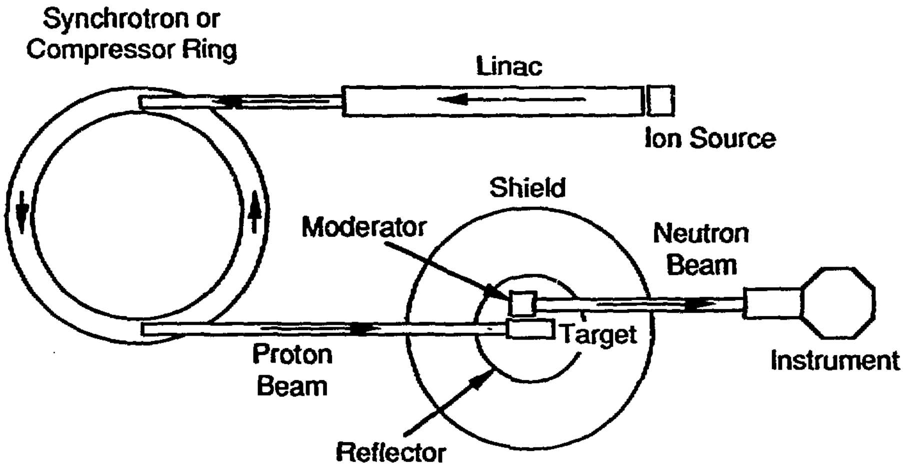

Synchrotron or
Compressor Ring
Linac
Ion Source
Shield
Moderator
Neutron
Beam
Target
Instrument
Proton
Beam
Reflector

Fig. 1

## Evolution of the Performance of Reactors and Pulsed Spallation Sources

(Updated from *Neutron Scattering*, K. Sköld and D. L. Price: eds., Academic Press, 1986)

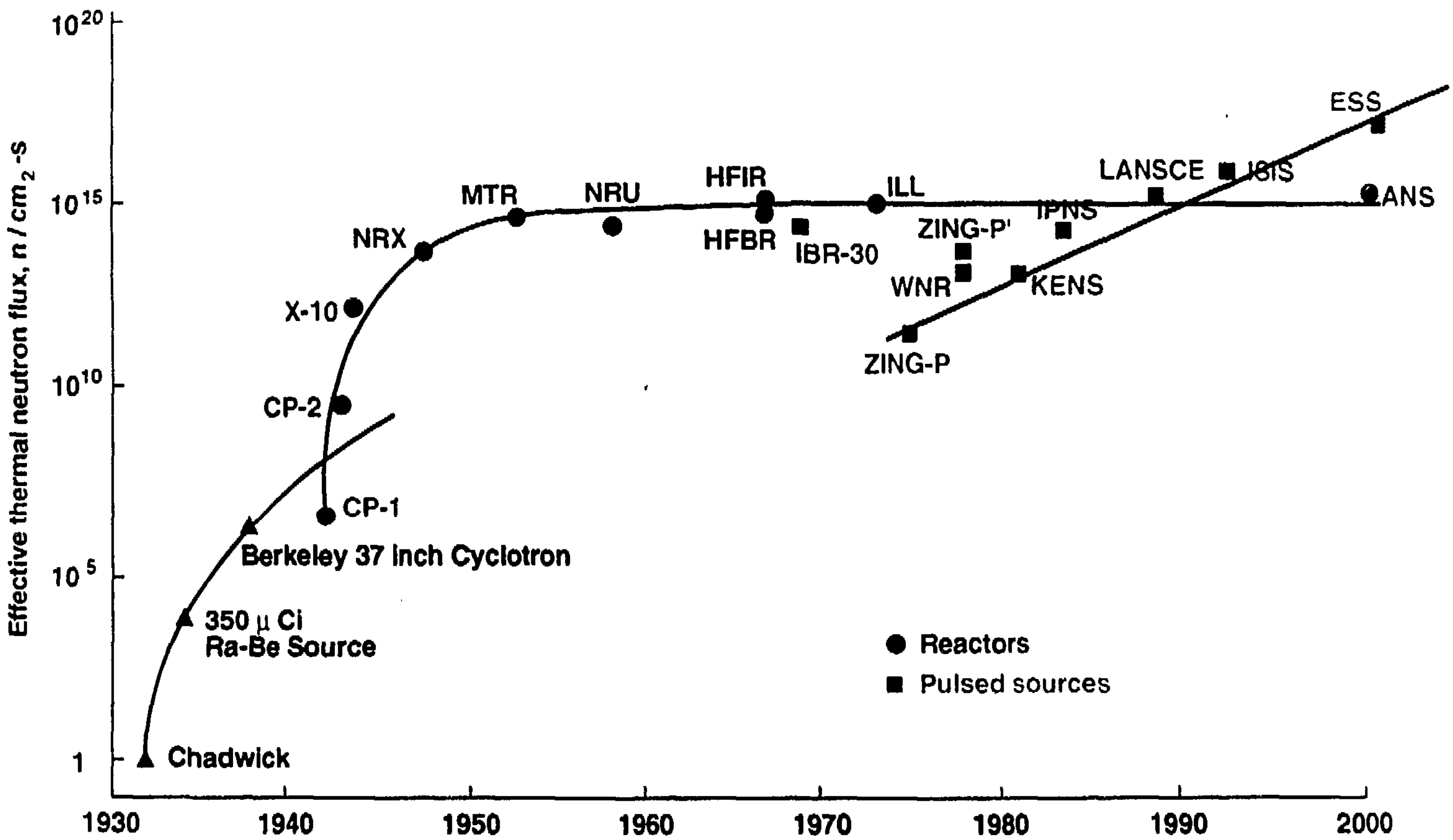
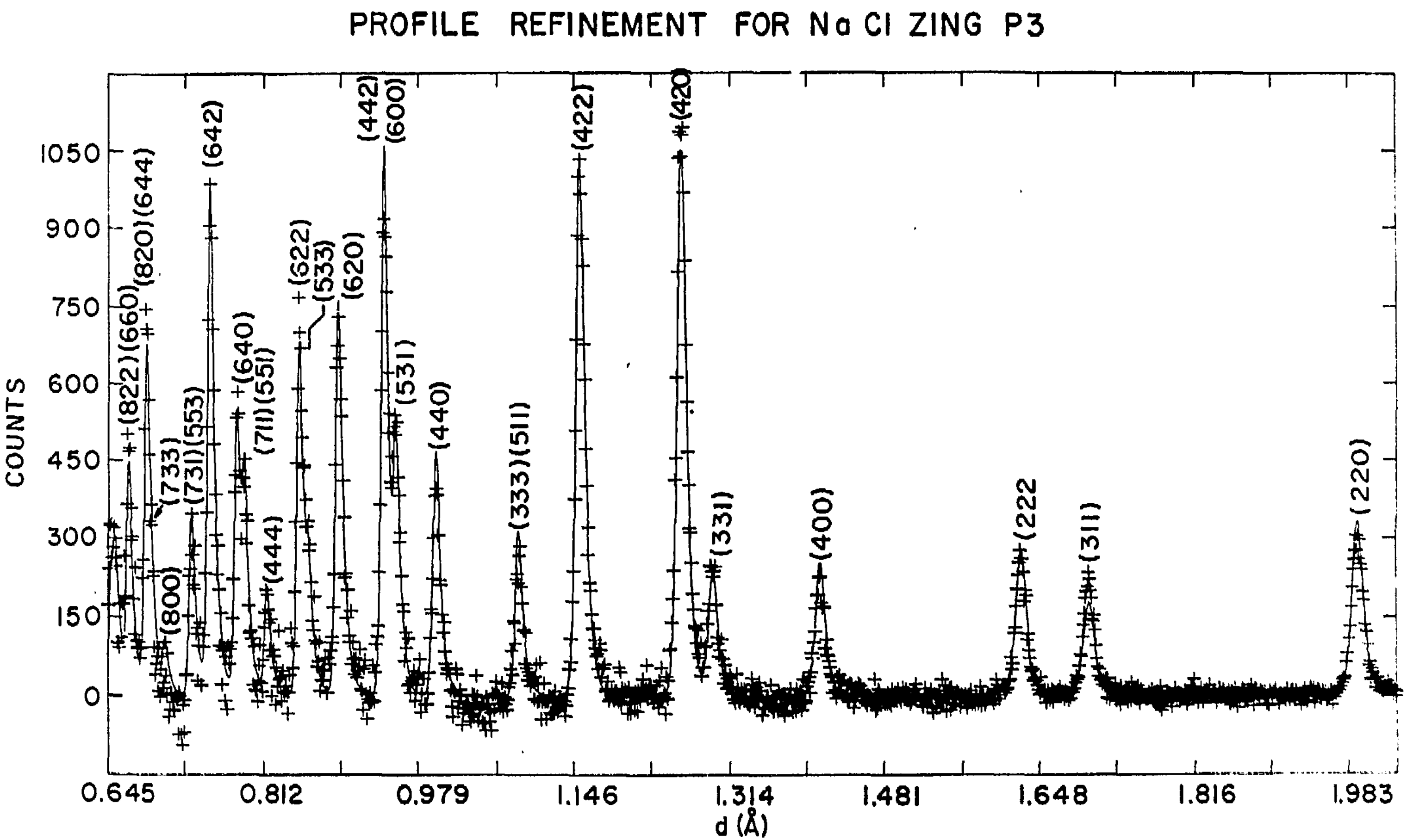



Fig. 2.

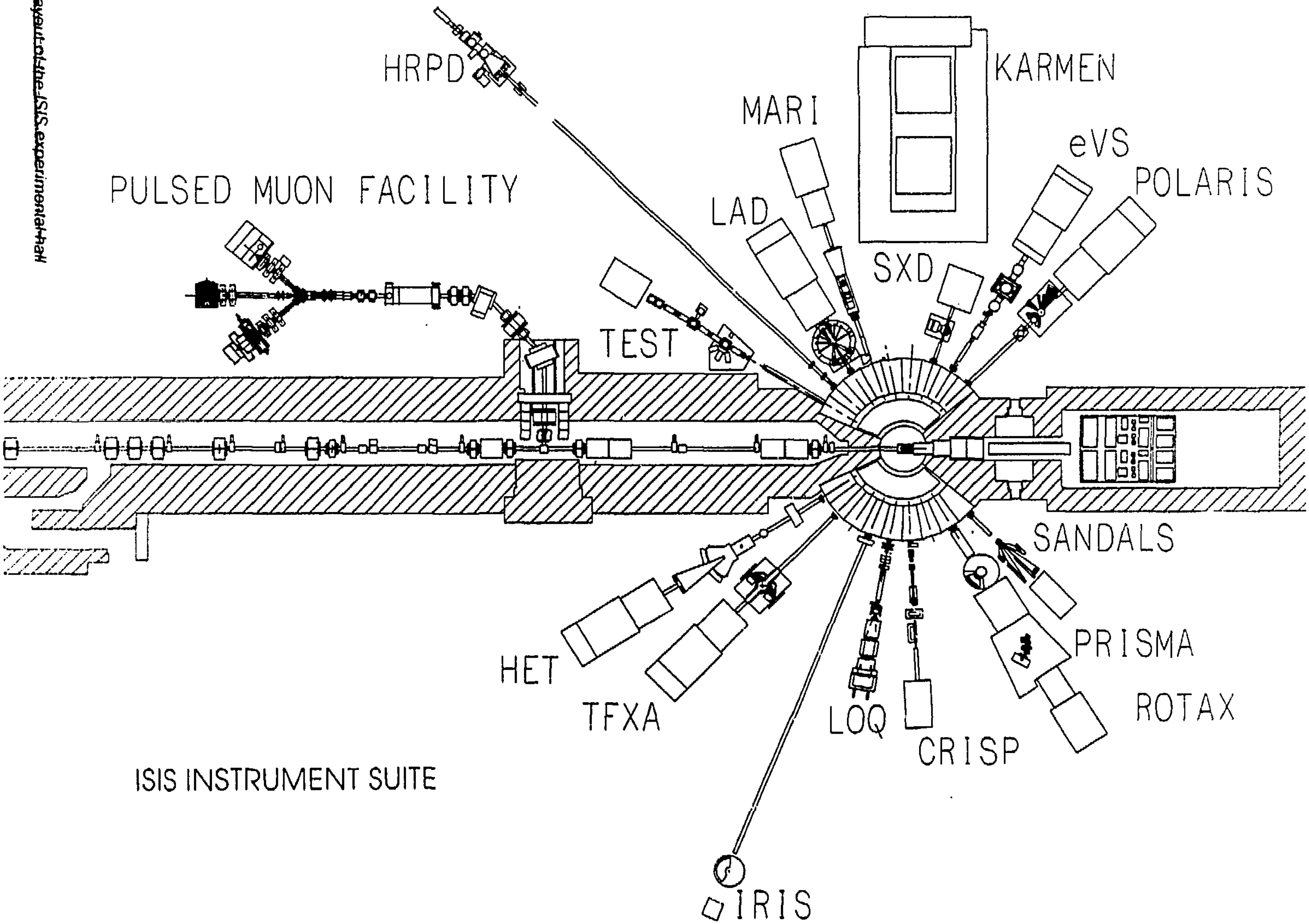


PROFILE REFINEMENT FOR Na Cl ZING P3
COUNTS
d (Å)
0
150
300
450
600
750
900
1050
0.645
0.812
0.979
1.146
1.314
1.481
1.648
1.816
1.983
(822)
(660)
(820)
(644)
(733)
(800)
(731)
(553)
(642)
(640)
(711)
(551)
(444)
(622)
(533)
(620)
(442)
(600)
(531)
(440)
(333)
(511)
(422)
(420)
(331)
(400)
(222)
(311)
(220)


Fig. 3

Layout of the ISIS experimental hall

7




ISIS INSTRUMENT SUITE

Fig. 4.