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SAMPLE ENVIRONMENTS AT THE INTENSE PULSED NEUTRON SOURCE (IPNS)

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ABSTRACT

Neutron diffraction is a powerful tool for structural studies of samples in special sample environments because of the high penetrating power of neutrons compared to x-rays. The Intense Pulsed Neutron Source (IPNS) at Argonne National Laboratory (ANL) offers its users a variety of sample environments for pulsed neutron scattering and diffraction experiments. At the present time over 80% of all experiments performed at the IPNS involve some type of ancillary equipment to control the sample environment[1]. These include closed-cycle refrigerators, cryostats, furnaces, magnets, and pressure cells. There are also devices for automatic sample changing, positioning, and orientating. Most instruments have at a minimum, a dedicated closed cycle refrigerator (10K to RT) configured for the instrument's typical sample dimensions and scattering angles. Standardization in instrument sample well dimensions, process control equipment, and control software has made multi-instrument use of many of the furnaces and cryostats possible. General use, multi-instrument equipment is maintained by the facility's technical staff. Instrument dedicated equipment is maintained by the respective instrument scientist with help from the ancillary equipment group. The design and upgrading of equipment is done by the ancillary equipment engineer with the oversight and input of instrument scientists, instrument engineer, and technical staff. Ancillary equipment conception and design is science driven, with the instrument scientists and even users providing the initial input for design criteria.

1. Introduction

Special sample environments have become one of the neutron scatterer's primary tools in the study of the structure and dynamics of materials. While many sample environments have been developed for general use, others have been developed to investigate certain specific properties of a particular material or class of materials. Often these are then modified to accommodate other types and sizes of samples. Over the past 17 years, ancillary equipment that provides a special sample environment for the neuron scatterer have been purchased, developed, invented and reinvented by our operations staff, instrument scientists, and users. Listed in Table 1 are the various pieces of ancillary equipment now in routine use on the neutron scattering instruments at the IPNS.

[1] IPNS Progress Report 1996.

Table 1. Ancillary Equipment at the IPNS		
Ancillary Equipment	**Parameter Range**	**Instruments**
High Temperature		
Block Heater	T = RT to 300°C	SAND
Coffee Can Furnace	T = RT to 350°C	GLAD, SEPD, GPPD
Heating Circulator	T = 30 to 100°C	QENS
Hot Stage Displex®	T = 20 to 800K(527°C)	QENS, GLAD, SEPD, LRMECS, GPPD
Howe Furnace	T = RT to 1100°C	QENS, GLAD, SEPD, LRMECS, GPPD
In-Situ Furnace	T = RT to 250°C	POSY II
Klipper Furnace	T = RT to 700°C	SAD
LUCYFE Furnace	T = RT to 1100°C	GLAD
Miller I & II Furnaces	T = RT to 1300°C in a static air, flowing inert, oxidizing, or reducing atmosphere, or vacuum.	SEPD, GPPD
SCD Furnace	T = RT to 700°C	SCD
Low Temperature		
Closed Cycle Helium Refrigerator	T = 10K to RT	HRMECS, QENS, GLAD, SCD, SEPD, LRMECS, SAND, SAD, HIPD, GPPD, CHEX
Heli-tran®	T = 2.5K to RT	SCD
Orange Cryostat	T = 1.5K to RT	HRMECS, QENS, GLAD, SEPD, LRMECS, HIPD, GPPD
Oxford Instruments Cryostat 5 kG Magnet	T = 1.5K to RT B = 0 to 5 kG	POSY
High Pressure		
4 kbar Liquid Pressure Cell	P = 0 to 4 kbar	SAD
2 kbar Pressure Cell 2W Displex®	P = 0 to 2 kbar T = 10K to RT	SCD
7 kbar Pressure Cell 12W Displex®	P = 0 to 7 kbar T = 20K to RT	SEPD
High Magnetic Field		
5 kG Magnet Oxford Instruments Cryostat	B = 0 to 5 kG T = 1.5K to RT	POSY
14 kG Magnet	B = 0 to 14 kG	SAD

Sample Changers		
Room Temperature Sample Changer	10 Positions	GLAD, SEPD, GPPD
Sample Changer	4 Positions	POSY II
Sample Positioners		
Sample Orienter	360° ϕ and 360°χ	GPPD
Sample Orienter	360° ϕ and 90°χ	SCD
Sample Rotator		SAD
X-Y-ϕ Translator	360° ϕ	SCD
X-Y-Z-ϕ Translator	360° ϕ	GPPD
Special Equipment		
Hydrogen Charging System		POSY II
Liquid Cell		POSY II
Residual Stress Analyzer		GPPD
Shear Cell		SAND

NOTE: It is best to contact the respective instrument personnel for specific instrument sample configuration and dimensions restrictions prior to submitting experiment proposal. The instrument acronyms are as follows:

Diffractometers	
Special Environment Powder Diffractometer	SEPD
General Purpose Powder Diffractometer	GPPD
Glass, Liquid, & Amorphous Solid Diffractometer	GLAD
Single Crystal Diffractometer	SCD
High Intensity Diffractometer	HIPD
Small Angle Diffractometer	SAD
Small Angle Neutron Diffractometer	SAND
Spectrometers	
High Resolution Medium Energy Chopper Spectrometer	HRMECS
Low Resolution Medium Energy Chopper Spectrometer	LRMECS
Quasi-Elastic Neutron Spectrometer	QENS
Chemical Excitation Spectrometer	CHEX
Reflectometers	
POSY	POSY
POSY II	POSY II

Detailed information on specific individual ancillary equipment at the IPNS is available on the IPNS web site at http://www.pns.anl.gov.

2. General Rule of Standardization

In order to maximize the efforts of our operations staff, standardization has become the deciding factor in many of our instrument designs. This standardization has lead to a desired portability among instruments, non-duplication of effort, a centralized inventory of spare parts, and maximized the experience of staff with respect to operation and maintenance. Of course with any rule there are exceptions. We have tried to keep these to a minimum and they have almost always resulted from designs in which the IPNS technical staff was not involved, at least not until the equipment showed up at our door.

2.1 Instrument Geometry

From the beginning of IPNS[2], instrument geometry has been built around either a twelve inch diameter or twenty-four inch diameter sample well. The sample well flange o-ring surface to beam centerline dimension has been kept to a standard twelve inches for the small sample well and twenty-four inches for the larger well. This has allowed the ancillary equipment to move from instrument to instrument as required and also for out dated retired equipment to be refurbished or cannibalized for use on newer instruments.

2.2 Temperature Control

We have tried to standardize our temperature controllers so that our staff and users can become familiar with the manual operation, over remote computer control, if required. It also minimizes the effort in writing software drivers for each individual brand or model of controller.

For our low temperature cryostats and closed-cycle refrigerators, we have installed Lake Shore Cryotronics Model 330 Temperature Controllers. These can be field configured to read either silicon diodes or platinum resistors. They can also be factory ordered to read a thermocouple on one or both of their two sensor inputs, such as a Fe/Au thermocouple for low temperature applications. The output of the controller is 0-1 A to a maximum 50 W heater. The Lake Shore controller is a full PID controller with an autotune feature.

For our high temperature furnaces we have, after many false starts, decided on Eurotherm Controls' Model 905D Temperature Controllers. These must be factory ordered to read specific thermocouple type or RTD inputs. The output of the controller is 0-10 V to a voltage controlled DC power supply. The Eurotherm controller is also a full PID controller with an autotune feature. In addition to the Eurotherm controller we have installed Newport Model 81 Temperature Alarms in the control circuit. These are manually set by our operations staff when completing a Furnace Check List procedure for each experiment before power is allowed to be applied to the furnace itself.

[2] Sample-related Peripheral equipment At IPNS, D.E. Bohringer and R. K. Crawford, Proceedings of the Eighth Meeting of the International Collaboration on Advanced Neutron Sources (ICANS VIII), July 2-12, 1985, Rutherford Appleton Laboratory, UK, Rutherford Appleton Laboratory Report RAL-85-110.

Both the Eurotherm and Lake Shore controllers are configured for RS232 communications. Setpoints, PID values, ramp rates, and autotuning can be set or be toggled on/off from the instrument computer terminal.

2.3 Motion Control

Position, both linear and rotary, has been performed using Superior Electric Slo-Syn synchronous stepping motors controlled with Bi-Ra stepping motor controllers. These units have been used to orient samples on Huber goniometers, sample changers, and bar calibrators for linear position sensitive detectors.

2.4 Control Software

It has been a long-standing dictum at the IPNS that the runfile control software be standardized among the instruments. With minor differences, to account for choppers and such, the software interface is the same for the diffractometers, spectrometers, and reflectometers. So with the sample environment control software. From a single menu, accessed during the setup of a run, a user can install ancillary equipment control to change temperature, position, or orientation, into the run file. The user can also control the available ancillary equipment outside the run file setup by accessing the individual control software.

2.5 Exceptions to the General Rule of Standardization

There are always exceptions to any rule and these have almost always been either a user provided piece of ancillary equipment or one with a very narrow and specific use. We have learned over the years to adapt and accommodate these exceptions. And when it is warranted, we have taken on some exceptions as our standard because they have worked and worked well.

3. Instrument Dedicated Ancillary Equipment

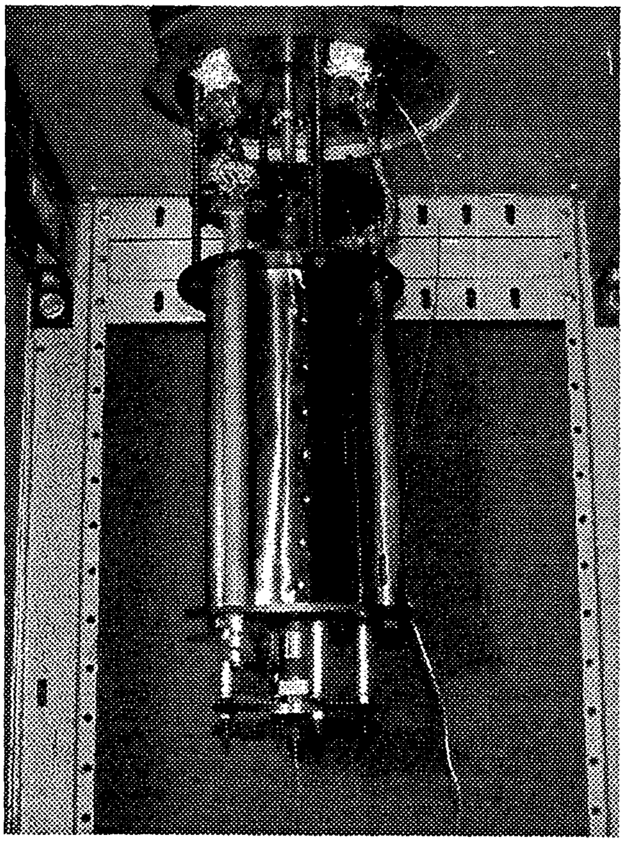
Most instruments at the IPNS have at the minimum a dedicated closed-cycle helium refrigerator. These are dedicated full time to the instrument since they are in almost constant use and are configured for the respective instrument's specific beam dimensions and scattering angles. Some instrument dedicated ancillary equipment have been developed by the instrument scientists to exploit some particular feature of their respective instruments such as the 5 kG magnet on the POSY reflectometer or the 7 kbar High Pressure Cell on the SEPD powder diffractometer. Other ancillary equipment have been developed to address specific needs of our users. A good example of this would be the Residual Stress Analyzer developed and used on the GPPD powder diffractometer. The instrument dedicated ancillary equipment are developed and maintained by the respective instrument scientists with assistance from the NGS Operations staff.

4. General Use Ancillary Equipment

Many of the high temperature furnaces are shared among several instruments, as is our low temperature cryostat. The IPNS Ancillary Equipment engineer develops and maintains these pieces of ancillary equipment with the assistance from the NGS Operations staff and the oversight of the instrument scientists. This is a relatively new position in the IPNS organization and recognition of the importance of safe, reliable, and well-characterized ancillary equipment. We are increasing our effort to perform more routine testing, profiling, and maintenance of the furnaces during our scheduled shutdown periods rather that during the course of an experiment. The ancillary equipment that falls under the general use/maintained category are as follows:

4.1 Howe Furnace

The Howe is a vanadium resistive-element type furnace used for sample heating to 1000°C. Incident and scattered neutrons pass through two vanadium heat shields, a vanadium heating element, an inner vanadium sample housing, and a sample container during experimentation. The sample environment is kept separate from the instrument chamber by a sample housing which enables sample exchange-gas purging and quick sample changes without the need to remove the entire furnace from the instrument. The furnace utilizes the instrument vacuum to protect the vanadium heating element from oxidation. A Balzers dual vacuum controller provides vacuum interlocks for the sample volume and the instrument chamber. The furnace is controlled using an Eurotherm controller and has remote computer interface capabilities. A Newport temperature alarm unit provides an independent temperature interlock. An Electronic Measurements 20V, 250A DC power supply is used to heat the furnace. A portable Edwards roughing pump and Balzers turbo pump are used to evacuate the sample volume. All control and interlock equipment are supplied in a mobile cabinet rack. The furnace uses one sheathed type K thermocouple positioned within the instrument vacuum in the center of the heating element for furnace control. A dual probe type K thermocouple is used for sample temperature monitoring and police interlocking, and is used as the sample container mount within the sample vacuum volume. This furnace is used primarily on the SEPD, GPPD, and GLAD instruments, but could be used on the following instruments as well but with a smaller sample than normally used: HRMECS and LRMECS. The Howe furnace can also be outfitted with a tantalum element and heat shields for use on QENS.

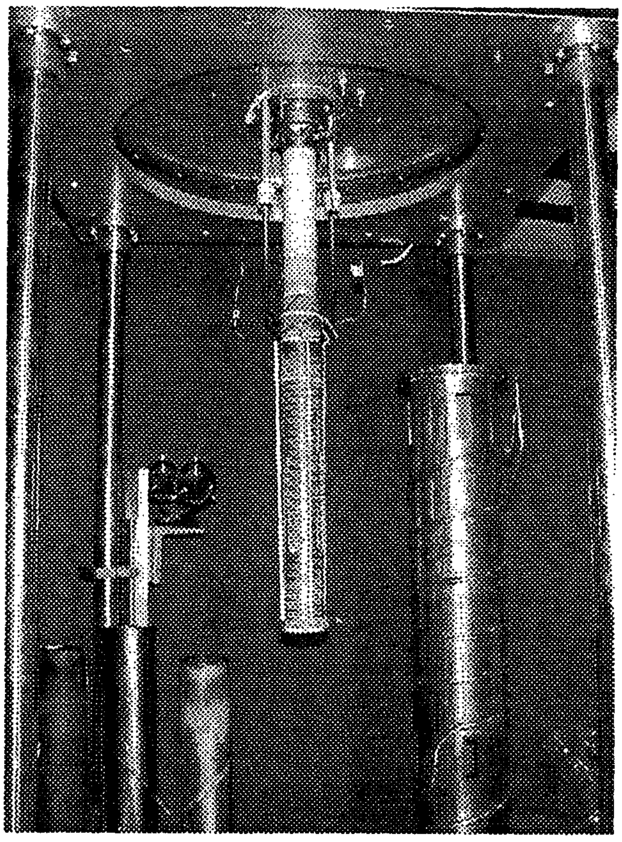


4.2 LUCYFE Furnace

The LUCYFE is a vanadium resistive-element type furnace used for sample heating to 1000°C, and is very similar to the Howe furnace. Differences between these two furnaces primarily involve the sample volume. The Howe has a separate sample volume from the instrument chamber, and can be used in conjunction with a purge gas during experimentation. The LUCYFE sample shares its volume with the instrument, so is therefore not purge compatible. The LUCYFE does, however have a sample stick which enables quick sample changes without having to break instrument volume. The furnace utilizes the instrument vacuum to protect the vanadium heating element from oxidation. A Balzers dual vacuum controller provides vacuum interlocks for the sample volume and the instrument chamber. The furnace is controlled using an Eurotherm controller and has remote computer interface capabilities. A Newport temperature alarm unit provides an independent temperature interlock. An Electronic Measurements 10V, 250A DC power supply is used to heat the furnace. A portable Edwards roughing pump and Balzers turbo pump are used to evacuate the sample volume. All control and interlock equipment are supplied in a mobile cabinet rack. The furnace uses one sheathed type K thermocouple positioned within the instrument vacuum in the center of the heating element for furnace control. A dual sheathed type K thermocouple mounted to the sample centering stick is used for sample temperature monitoring and police interlocking. This furnace is intended primarily for use on the GLAD instrument.

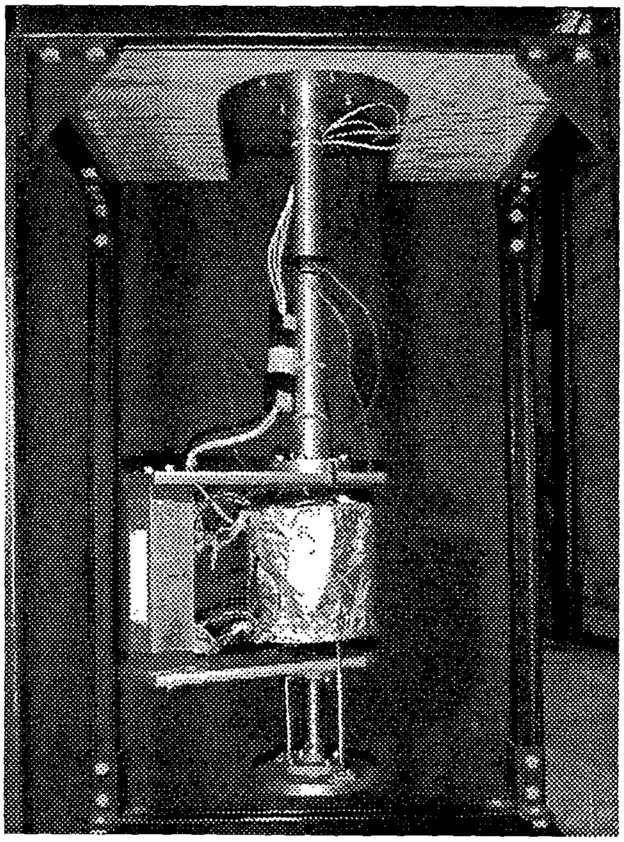
4.3 Miller Furnaces

The Miller furnaces are tantalum ribbon resistive-element type furnaces used for heating samples to 1450°C. Alumina is the primary material used in construction of these furnaces. The sample volume is separate from the instrument volume, so the samples can be used with a static air, purge gas, or vacuum, and sample changes can be done without breaking instrument vacuum. Typically, powder samples are pressed into pellets for use in the Millers, but other sample container type arrangements can also be used upon request. The instrument vacuum protects the tantalum ribbon and heat shields from oxidation, and is interlocked using a Teledyne Hastings analog vacuum gauge. The furnaces are controlled using Eurotherm controllers and have remote computer interface capabilities. A Newport temperature alarm unit provides an independent temperature interlock. Electronic Measurements 250V, 20A DC power supplies are used to heat the furnaces. All control and interlock equipment are supplied in mobile cabinet racks. Each furnace uses tow sheathed type S thermocouples for control and police interlocking. The sample temperature is monitored using a bare-wire type S thermocouple located inside the sample volume and typically in contact with the sample. Due to the scattering geometry, only 90° scattering angles are available with the Miller furnaces; therefore, the only compatible instruments are the GPPD and SEPD.



4.4 Coffee Can Furnace

The Coffee Can furnace is intended for experiments ranging from room temperature to 400°C. Four Watlow cartridge heaters heat this furnace. Two of these heaters are positioned above the sample embedded in the top aluminum plate: The other two are located in the bottom aluminum plate. The sample is heated primarily through radiation heat transfer to help ensure sample temperature homogeneity. A 7.16" diameter vanadium flanged tube 2.5" in length is typically used as the sample container, but other containers can be used upon request. The furnace operates within the vacuum volume of the instrument, and sample changes require complete furnace removal from the instrument well. The furnace is controlled using an Eurotherm controller and has remote computer interface capabilities. An independent Newport alarm unit provides additional defense in prohibiting furnace run-away. A 100V Kepco DC power supply is used to heat the furnace. All electronic components are located inside a mobile furnace control cabinet. The furnace utilizes three independent thermocouples for furnace control and sample monitoring. These three K thermocouples are physically located as follows: thermocouple #1 measures the temperature used to control the furnace, and is positioned directly on top of the top aluminum plate that houses the top heater cartridges; thermocouple #2 measures sample temperature, and can be positioned as necessary by the user, but is usually wrapped amount the thread that connects the sample container to the top plate; thermocouple #3 is the police thermocouple, and is located on the bottom aluminum plate that houses the two lower heater cartridges. This furnace is used primarily on the GLAD, SEPD, and GPPD instruments.



4.5 Hot Stage Displex®[3]

A "Hot Stage" Displex® has been implemented at the Intense Pulsed Neutron Source (IPNS) for performing neutron diffraction experiments in the temperature range 20K to 800K. The new piece of ancillary equipment allows for extended temperature range studies through room temperature without having to change sample containers or environments. The hot stage was designed for and installed on a refurbished closed-cycle helium refrigerator cold head. The unit

[3] A "Hot Stage" Displex®: 20-300K In A Neutron Scattering Environment, K. J. Volin and D. E. Bohringer, These proceedings.

employs four (4) single crystal sapphire stand off rods to provide a thermal conduction path to the sample at low temperatures, where sapphire is a conductor, and to isolate the sample and heater at high temperatures, where sapphire is an insulator. As with a standard closed cycle refrigerator, the first stage heat shield is held at about 20K throughout the temperature range. The second stage heat shield, along with the cold finger holding the sample container, is heated to the desired temperature with a resistance heater. A silicon diode located in the second stage and a platinum resistor mounted in the hot stage monitor temperature. The temperature is controlled by a Lake Shore Cryotronics Model 330 Temperature Controller with a RS-232 communications link to the neutron scattering instrument's data acquisition computer, allowing for run file temperature control. To ensure thermal equilibrium throughout the sample, the sample container is typically filled with one ATM. of helium. A new flange design for the standard closed-one-end vanadium sample cans has been incorporated to contain the helium atmosphere throughout the extended temperature range. Preliminary tests on a standard sample have shown a thermal gradient of <1 K/cm at 10K and at 800K over the length of the sample container. Cool-down times to 10K are on the order of 90 minutes, slightly longer than a standard Displex®. The neutron scattering background compares favorably with a standard closed-cycle refrigerator or mid-range furnace currently in use at the IPNS. This refrigerator/furnace is used primarily on the QENS, GLAD, SEPD, and GPPD instruments.

4.6 Orange Cryostat

The standard ILL "Orange" Cryostat was developed by the ILL cryogenics department to meet the needs of experimental scientists. The main requirements were for a robust and simple to use yet high performance cryostat suitable for neutron scattering. The main features of the Orange Cryostat include large sample top loading capability (50mm ϕ), isolated sample area with static exchange gas, low helium consumption and fast sample cycling times. The temperature of the Orange Cryostat is controlled between 4.2 and 300K with a Lake Shore Cryotronics Model 330 Temperature Controller. Temperature is monitored by two 100Ω platinum resistors located on the sample holder and on the variable temperature heat exchanger. The heat exchanger is fitted with a 50Ω heater. Several sample holders are available to accommodate the different sample configurations of the individual neutron scattering instruments. A Kinney pump is available to achieve temperatures below 4.2K. This cryostat is used primarily on the QENS, HIPD, GLAD, SEPD, and GPPD instruments.

5. Future Directions

Two things drive our ancillary equipment development; One is science driven, that is, to expand the limits of phase space that our current equipment is capable of accessing, enabling our users to perform experiments previously not realizable. The other is a desire to make the most efficient use of the scheduled beam time. As our source and moderators become more efficient and our neutron flux increases, run times are decreasing and there is the desire to shorten the sample turn around time to make the best use of beam time. We have done this in the past with sample changers, which allow the user to switch samples without breaking instrument vacuum. The directions in which we are developing ancillary equipment, at least in the near term, are as follows:

5.1 Low Temperature

- Purchase larger cooling capacity closed cycle refrigerators to decrease cool down.

- Develop a Low Temperature Sample Changer utilizing a stepping motor to move an entire closed-cycle helium refrigerator up or down through a double o-ring piston seal in a evacuated sample well. This unit is already constructed and its operation is being debugged.

5.2 High Temperature

- Construct a second Hot Stage Displex® to accommodate the scheduling conflicts among the instruments due to its over-subscription.

- Develop a larger bore furnace, on the order of 50 to 100 mm, to accommodate the larger sample configurations of the inelastic spectrometers.

5.3 High Pressure

- Interface pressure sensors to monitor and log the sample pressure during the run.

- Assemble a high pressure cell test facility. As the interest in high pressure continues to increase we have a real need to pressure test cells that a user might bring to perform an experiment and those of our own design.

6. Summary

IPNS has a wide range of ancillary equipment that provides special sample environments for the neutron scatterer. Standardization of instrument geometry, process control equipment, and control software has maximized the reliability, interchangeability, and efficiency of the equipment. Current equipment is constantly being upgraded and new equipment developed to meet the ever-expanding requirements of our user community.

7. Acknowledgements

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