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Sample-related Peripheral Equipment at IPNS*

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Introduction

This paper will briefly describe sample environment equipment provided by IPNS to visiting users and staff scientists. Of the twelve horizontal neutron beam stations, (ten now operational, two under construction) all use one or more form of such support equipment. An in-house support group devotes a significant fraction of its time to development, calibration, and maintenance of this equipment.

Vacuum Systems

Because of our many visiting neutron scientists and limited instructional time, simplicity is a key word. With sample change frequencies recorded in hours, easy vacuum control becomes a must. Our users have available a vacuum control station we like to refer to as user friendly. Operation of this control chassis is a simple two-step function of automatic-manual switch placement, plus a push button start. The control system will then rough pump the sample area, followed by a predescribed sequence to final vacuum of 10^{-6} to 10^{-7} torr. The control chassis can operate turbomolecular, diffusion or cryo pumps within the pump down routine. We now have five of these systems in operation.

Standardized Flanges

Maximum effective usage of peripheral equipment means interfacing IPNS instruments to ancillary devices. This has been accomplished via standardized flanges. With little or no adjustments, most IPNS instruments can cross use peripheral equipment, adding to user flexibility.

Furnaces

Listed in Table 1 are the furnaces we now use, plus a brief description. Due to the number of high temperature experiments submitted at IPNS, these units have had much use. Our first high temperature furnace built at IPNS was the "Wehrle Design". Since its preliminary test runs, many modifications in heater element material, thermo shield standoffs, and heater element geometry, have taken place. With the addition of 2 vanadium heat shields, our heater ribbon life increased from 50×10^3 C°-HR to over 100×10^3 C°-HR. Replacing the 2.36 mm x .05 mm tantalum ribbon with .5 mm tantalum wire further increases heater life to 180×10^3 C°-HR. (The temperature used to determine C°-HR was 1000°C x actual operating hours.) Considering this furnace's fragile design, the increase in running time was indeed progress. The above changes have made the "Wehrle" furnace an early workhorse for our high temperature users.

Since that time we have added two other custom built +1000°C furnaces, the "Miller" furnace and the "Howe" furnace. These new designs have provided us with giant steps forward in furnace performance and reliability. Testing is still being performed on updating the "Miller" furnace temperature vs. power requirements. To date, this unit has operated well over 200×10^3 C°-HR (operating temp. = 1200°C)[1]. Fig. 1 illustrates the progress of lower power -

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*Work supported by the U.S. Department of Energy higher temperature due to these modifications. One advantage this unit has over our other furnaces is the sample area is independent of the furnace element environment. Fig. 2 provides design characteristics.

The "Howe" furnace (Fig. 3) is the newest addition to our family of high temperature devices. Designed with a .05 mm vanadium foil heater element, the radiating surface area is ~ 200 cm^2. To insure low power requirements, dual .1 mm thick vanadium heat shields surround the heating element. This above combination has proven very sturdy, providing over 270 x 10^3 C°-HR of operation before a failure due to poor vacuum conditions. Another important feature of this unit is that the construction provides access to all scattering angles with minimum coherent scattering interaction in the vanadium.

Fig. 4 shows the custom furnace used at the Single Crystal Diffractometer (SCD). The temperature range of this device is from 200 - 800°C using a small platinum foil heater element. Due to its size, the power requirement at 800°C is ~ 30 amps at 10V. Sample size is approximately 10 mm^3 with scattering at 90° to the incident beam. The usable diffracted beam, as shown, covers approximately 60°.

Additional information of heater element life vs. temperature plus thermocouple longevity is of primary importance in establishing timely maintenance. Using the March 1984 to April 1985 running period as a ruler, our total down time due to furnace failures was 66 hours. The total furnace experimental hours were 1098 on two instruments giving our furnace experiments a +99% operating efficiency.[2] Closer evaluation of problem areas, plus better understanding of physical limitations of each piece of furnace equipment will improve our working knowledge while climbing the all important high temperature ladder.

Furnace Controllers

The variety of controllers available is large, but after some qualifications are made, the field of choice is narrowed somewhat. A decision to standardize our peripheral needs lowered the number of candidates. Our final selection, Micricon model 82300[3], best fit the projected needs regarding flexibility, reliability, accuracy and the ability to computer interface as discussed in the Automated Control of Peripheral Devices section.

The controller operating specifications using type K (chromel-alumel) thermocouple are 0-1370°C at 1°C selectability (tests have shown .2°C control accuracy).[4] Dual inputs, connected in a cascade configuration can further increase control accuracy, or these inputs can be arranged in the normal control feedback plus sample readout logic. In addition to the usual gain, rate, and reset capabilities, the options of preselected or adjustable heating ramps and multi level temperature plateaus are also available.

By changing preprogrammed input/output boards, additional choices of control and readout thermocouples can be operating. Our latest high temperature test has been with type S (platinum-platinum 10% rhodium), with an upper range of 1760°C. These tests are being conducted on our "Miller" (FM-1000C-V) furnace, for control and thermocouple longevity.

Displex Expander Module and Controllers

Displex closed-cycle He refrigerators are the workhorses for most low temperature experiments. With a proper maintenance schedule, our displex heads, Air Products Model DE-202K, and compressor units have had less than 1% down time.

Air Products Model K Series 5500[5] controllers with optional RS-232 communications are used for computer interfacing. These controllers now numbering 5, along with these displex units, have an operating range of 7 to 300 K. Using gold-chromel thermocouples, the experiment has two choices of control and readout location. These controllers are interfaced to the data acquisition computers as referenced in the Automated Control of Peripheral Devices section.

Cryostat and Controller

Our Thor variable temperature helium cryostat was designed to operate at temperatures as low as 2 K. To date, we have successfully operated at 2 K with more fine tuning needed. The cryostat can be operated on both powder instruments with minimum hardware setup. At the business end of the cryostat is a copper sample holder, extending from a cold plate. This holder can accommodate a sample size of x = 3.25 cm by y = 5.18 cm.

Silicon diodes, plus readout thermometry, regulate power needed to maintain temperatures above 2 K using T.R.I. Research T2000 controller.[6] This system has two level operational specifications of ± .01 K from 1.5 to 25 K, coupled with ± .1 K from 25 to 380 K coupled with internal heating control that operates at 0 to 25 volt with 0 to 2 amperes output.

Sample Changers

Until recently, we have had only one automatic multi-sample changer. This unit has performed well over the last 3 years at the Small Angle Diffractometer (SAD). The changer shown in Fig. 5 has two vertical rows of 6 sample positions in each. Two stepping motors are driven by a CAMAC XY motor controller with position accuracy ± .3 mm.

Our newest sample changer in Fig. 6 can hold 10 samples. This unit known as the Geneva Drive Sample Changer can be used in either the General Purpose Powder Diffractometer (GPPD) or Special Environment Powder Diffractometer (SEPD) and operates in a vacuum environment. With the addition of local cadmium skirts around each sample, plus collimation of the scattered beam by two large 9.5 mm thick boral discs, a quiet background is established. Due to the drive motor being located in a vacuum environment, the geneva drive system was chosen to position the samples. Final position repeatability is ~ .5 mm.

Camac control modual #3063[7] located in the PDP11/34 operates the drive system, along with an additional local-remote chassis with position readout residing at the instrument.

Sample Orienter

This single-axis orienter allows orientation of a sample in vacuum, and can be used on the High Resolution Medium Energy Chopper Spectrometer (HRMECS), Low Resolution Medium Energy Chopper Spectrometer (LRMECS), GPPD and SEPD. The sample is positioned on a feed thru shaft, adjustable in y position to beam center and can be rotated 360° in .01° increments. Operating a displex with the orientor is now an option that adds to this system's flexibility.

High Pressure Gas Cell

The SEPD has just completed its first experiment using a new high pressure gas cell.[8] The cell shown in Fig. 7 provides high count rates, low background, plus a large sample volume (5 cm^3). When attached to a displex or cryostat, the operating temperature range is from ambient to ~ 15 K. The latest operating parameters showed the pressure cell at 4 K bars with excellent neutron scattering results.

Another high pressure cell used on the SEPD is shown in Fig. 8. This room temperature piston-cylinder cell is well collimated at 90°. The sample volume of 0.25 cm^3 has a pressure range of 0 - 35 kbar, with a hydrostatic sample environment. This unit can also be used on the GPPD. A third pressure cell, now being developed, is listed in Table 2.

Chart Recorder/Data Logger

This system is called multipoint recorder/logger.[9] It is an 8 input channel (expandable to 48) multiple-microprocessor based, user settable data acquisition instrument with RS-232 option. In Fig. 9, the MRL uses a high performance, low-power, 16 bit, CMOS microprocessor as the central hub. Also, two additional 8 bit microprocessor and additional high level integrated circuits control various peripheral functions internally.

Data reporting formats include chart recording, with paper speed from 1 through 12,000 mm-per-hour. The paper speed stepper motor drive advances paper at equivalent speed with printing trend line information. Each channel is easily identifiable. Another format is a peak-valley report. This report is generated at a user-selected interval to document in hard-copy the precise high and low values that have occurred for each selected channel and the times at which they occurred. An important feature is the ability to select a "both" mode. This provides both a selectable periodic data printout, with interconnected time selected chart recordings. Alarm function can be established on both data logger and recorder formats, with internal and/or external alarm capabilities. The model we selected is portable and is easily programmable.

Watchdog Timer

Fig. 10 shows the location of the Watchdog Timer WDT-1000[10] in data flow lines. Its function is to provide monitoring of real time environments at the front end of our data acquisition system. By adding commands that will direct control and select alarms of a furnace, displex, cryostat, etc., the Watchdog Timer will notify the main control room personnel if any of these commands are violated or if the data system crashes.

A simple software command energizes this system and might provide the cherished midnight call in. This devious device is available to all our instrument scientists.

Automated Control of Peripheral Devices

With a wide variety of ancillary devices now available, and more in the development stage, it was important to provide a unified framework for interfacing these devices to the data acquisition computers. The devices range from the sophisticated, such as the Micricon temperature controllers, with a high degree of local programmable intelligence and a consequently large number of settable parameters, all the way down to a "dumb" on-off device such as a Watchdog Timer which has no settable parameters. Since IPNS is a user facility, any such device control software has to be easy to use (and relatively foolproof) for the casual user. However the same software should allow the knowledgeable user (e.g., - instrument scientist) access to all the parameters and the full range of capabilities of the various devices. Furthermore, the software framework should allow for unlimited expansion to an even wider variety of future devices.

To simplify and standardize the required interfacing hardware and software, it was decided to limit controlled devices to two types of hardware interfacing, either RS-232 communications (so the device looks like a terminal to the computer) or communication via CAMAC modules of various sorts. Devices so far interfaced, or for which interfacing is in progress or currently contemplated are shown in Table 3.

In interfacing a device, all the device parameters which are to be accessible from the data acquisition computer are defined and entered into a master "device table" which is stored in each instrument computer and contains all such parameters for all interfaced devices. In order to maintain the ease of use for the casual user, these parameters are divided into two types, "user parameters" and "non-user parameters". User parameters are those which will typically be changed from run to run (e.g., - set point temperature and limits for a temperature controller, sample position for a sample changer, etc.). These are the only parameters with which the casual user interacts. The non-user parameters (e.g. - proportional, integral, and derivative settings for a temperature controller) may influence the operation of the device in ways transparent to the user, and may be changed as desired by specific commands.

In addition to this master device table, a second table, different for each instrument, contains the current device configuration for that instrument. This includes the names of the devices available on that instrument, a device-number reference to the master device table for each of these available devices, the slot in the control CAMAC crate or the RS-232 terminal line being used to communicate with the device, and a parameter indicating whether the device is to be set up at the beginning of the run and then ignored during the run (as for a sample changer or a stepping motor) or is to be monitored during the run as well (as for a temperature controller, analog-to-digital converter, etc.). When setting up a run, the user selects which, if any, devices from this table are to be controlled for this run. Any number can be selected.

Devices being monitored during a run will cause suspension of data acquisition whenever they are outside the user-specified limits for that run. Data acquisition will automatically resume whenever all monitored parameters are back within their limits. If the Watchdog Timer has been selected as one of the controlled devices for this run, it will time-out and set an alarm alerting operators in the accelerator Main Control Room whenever a monitored device is outside its limits for more than four minutes. (See Fig. 10 for a typical application.)

Operating Experiences

Fig. 11 provides a percentage breakdown of equipment operating time on the GPPD. As can be seen, experiments involving altered sample environments dominate. Although GPPD is on the high side of the peripheral equipment usage scale, the trend extends to other instruments. Due to the increase, timely maintenance is all important. To provide a manageable system, a "maintenance data base" program was developed to fit our needs.[11] This program allows the operator to add, delete, modify, or update data records requiring periodic attention. These records range from annual qualification tests to weekly ordering of helium gas. This program also gives us a hard copy of completed tasks in chronological order, making maintenance and record keeping a breeze. These records (now over 500) also provide documented evidence of required procedures in the operation of IPNS.

Conclusion

In the last year of operation, 10 experimental beam lines comprised a total of 29,000 operating hours. Of this total, well over half involved the use of peripheral equipment. Our total lost time due to peripheral equipment failure was less than 1%. This is a strong reflection of the quality of this equipment and of experimental support personnel at IPNS.

Acknowledgements

The following individuals provided us with our high temperature furnaces; R. Wehrle, ANL; Harold Miller, E.G.G.; Alan Howe and Nigel Wood, Leicester University, England. For designing all other peripheral equipment, we thank R. Kleb and R. Stefiuk, ANL. Without the able support group, our operating record would be non-existing. Denis Wozniak has provided all important guidance in upgrading our ever growing furnace systems, and has shouldered the burden of troubleshooting the Thor cryostat. Assisting Denis and myself are John Urban, whose experience is tantamount to efficient operation. David Leach guided us thru the early experiments with the Wehrle furnace. Al Paugys's background is refrigeration and cryogenics. In the electronics spectrum, necessary individuals would be Joe Haumann, Donald Emery, Merlyn Faber, and Wally Czyz. Special thanks to Thomas Worlton and Mariangela Sanelli for their infinite patience in providing the programs and data base needed to establish operating trends and maintenance schedules.

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[2] M. Sanelli and D. Bohringer (IPNS Operating Data, 1985).

[3] Research Inc., Box 24064, Minneapolis, Minnesota, 55424, U.S.A.

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[6] T.R.I. Research (St. Paul, Minnesota, 55114, U.S.A.).

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[8] R. Kleb, D. L. Decker and J. D. Jorgensen, (High Pressure Gas Cell, to be published).

[9] Esterline Angus Instrument Corp. (Box 24000, Indianapolis, Indiana, U.S.A.)

[10] Standard Eng. Corp. (44880 Industrial Dr., Fremont, California, 94538, U.S.A.).

[11] T. Worlton (Operating Files IPNS Manual).

TYPE	INSTRUMENTS	TEMPERATURE RANGE	CONTROL PRECISION	SAMPLE VOLUME	COMPUTER INTERFACE	COMMENTS
"MILLER"	GPPD, SEPD	~200-1400°C	0.2-2°C	~6 CM^3	YES	90°±5° SCATTERING ONLY. SAMPLE CAN BE IN CONTROLLED GAS ENVIRONMENT OR IN VACUUM. FURNACE IS WELL COLLIMATED OUT OF SCATTERED BEAM.
"WEHRLE"	GPPD, SEPD	~200-1000°C	0.2-2°C	~6 CM^3	YES	RANGE OF ACCESSIBLE SCATTERING ANGLES. LIMITED AMOUNT OF HEATER AND SHIELD IN BEAM.
"HOWE"	GPPD, HRMECS, LRMECS, SEPD, EVS	~200-1000°C	0.2-2°C	~6 CM^3	YES	ALL SCATTERING ANGLES ACCESSIBLE. HEATER AND SHIELDS ARE VANADIUM SO VERY LITTLE COHERENT SCATTERING FROM FURNACE.
SAD TUBE	SAD	~200-1000°C	0.2-2°C	~2.3 CM DIAMETER	YES	NO FURNACE PARTS ARE IN THE INCIDENT OR SCATTERED BEAM.
ILL	SAD AND POSSIBLY OTHERS	~200-900°C	0.2-1°C	~6 CM^3	YES	ALL SCATTERING ANGLES ARE ACCESSIBLE. FURNACE PARTS ARE DIFFICULT TO COLLIMATE OUT OF SCATTERED BEAM.
SCD	SCD	~200-800°C	~5°C	~10 MM^3	NO	90°±30° SCATTERING.
"300 K"	GPPD, SEPD, HRMECS	~30-300°C	0.2-1°C	~6 CM^3	YES	ALL SCATTERING ANGLES ACCESSIBLE.

TABLE 1: Description of IPNS Furnaces

HIGH PRESSURE CELLS

TYPE	INSTRUMENTS	PRESSURE RANGE	SAMPLE VOLUME	COMMENTS
ROOM TEMPERATURE PISTON-CYLINDER	GPPD, SEPD	0-35KBAR	0.25 CM^3	90°±2.5° SCATTERING. CELL IS WELL COLLIMATED OUT OF SCATTERED BEAM AT 90°. HYDROSTATIC SAMPLE ENVIRONMENT.
LOW-TEMPERATURE LOW-PRESSURE GAS CELLS (1 CELL NOW, 1 MORE IN FUTURE)	GPPD, SEPD	0-5KBAR AT PRESENT 0-8KBAR FUTURE	2 CM^3	90°±5° SCATTERING. CELL IS ALUMINUM AND IS PARTIALLY COLLIMATED OUT OF BEAM. CAN BE MOUNTED ON DISPLEX OR CRYOSTAT. HYDROSTATIC SAMPLE ENVIRONMENT.
LOW-TEMPERATURE CLAMPED PISTON-CYLINDER CELL	GPPD, SEPD	0-25KBAR	1 CM^3	90°±2.5° SCATTERING. CAN BE MOUNTED ON DISPLEX OR CRYOSTAT. HYDROSTATIC SAMPLE ENVIRONMENT. (QUASI-HYDROSTATIC AT LOW T.)

Table 2 : High Pressure Cells

Table 3: Device Interfacing

Device	Interface	#	Module or Controller
*Furnace	RS-232	3	Micricon 82300 controller
*Displex	RS-232	5	Air Products Instruments 5500 controller
*Watchdog Timer	CAMAC	10	Standard Engineering WDT-1000 Watchdog Timer module
*SEPD/GPPD sample changer	CAMAC	2	Kinetic Systems 3063 16-bit input gate/output register module
*SAD sample changer	CAMAC	1	BiRa 3101A 15-channel stepping motor driver module
		1	Kinetic Systems 3473 24-bit change-of-state module
*Sample orienter	CAMAC	1	BiRa 3101A 15-channel stepping motor driver module
°SCD goniometer motors	CAMAC	1	BiRa 3101A 15-channel stepping motor driver module
°QENS table drive	CAMAC		BiRa 3101A 15-channel stepping motor driver module
°Proton current monitor	CAMAC	1	Kinetic Systems 3610 6-channel 50 MHz scaler module
°Digital to analog	CAMAC	1	Kinetic Systems 3112 8-channel 12-bit D/A module
Analog to digital	CAMAC	1	Standard Engineering AD-112 2-channel 12-bit A/D module
Multichannel analyzer	RS-232	1	Nuclear Data ND-100 Analyzer
		1	Nuclear Data ND-60 Analyzer
Thor cryostat	RS-232	1	TRI T-2000 Controller
Chart recorder/data logger	RS-232		Esterline Angus Multipoint Recorder/Logger
Chopper drive system	RS-232	2	System developed in-house

*Interfacing completed

°Interfacing in progress

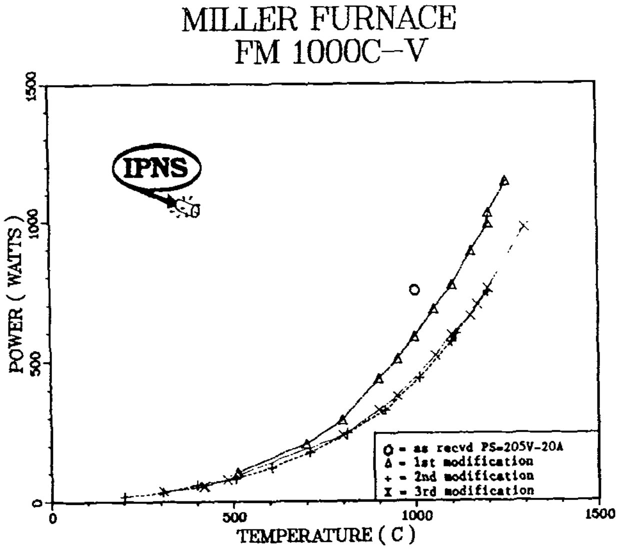

Fig. 1 – Modifications Improving Temperature vs. Power

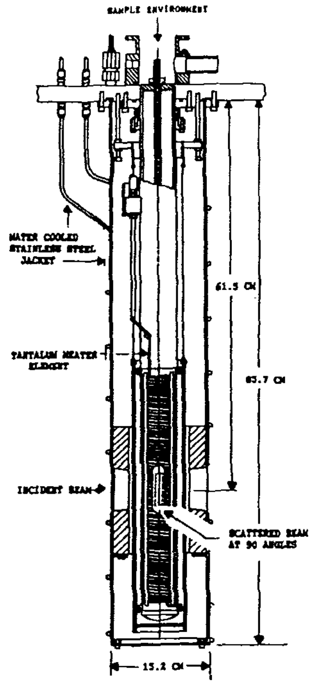

Fig. 2 – Miller Furnace

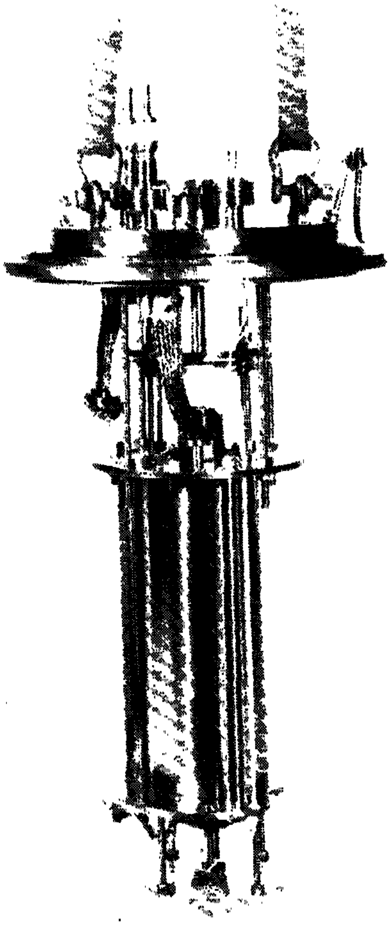

Fig. 3 - Hove Furnace

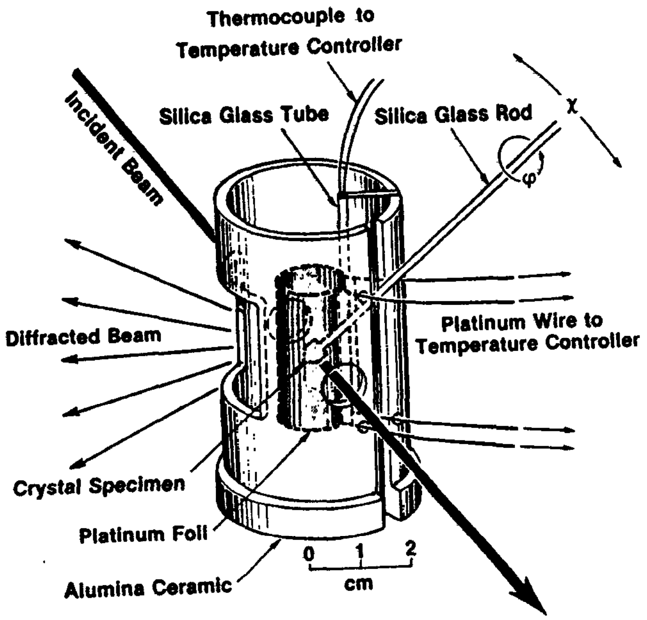

Fig. 4 - Single Crystal Diffractometer Furnace

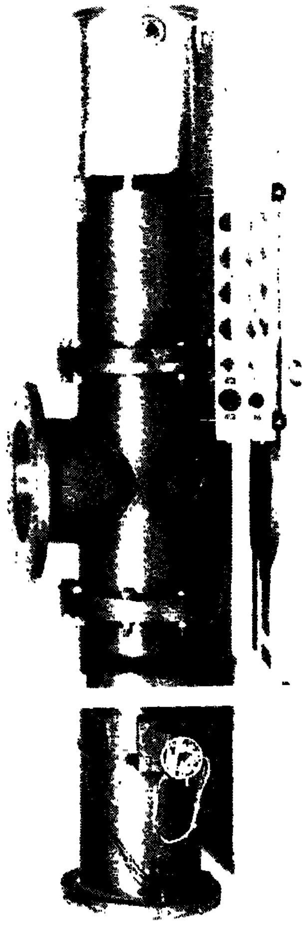

Fig. 5 - Vertical Cassette Sample Changer

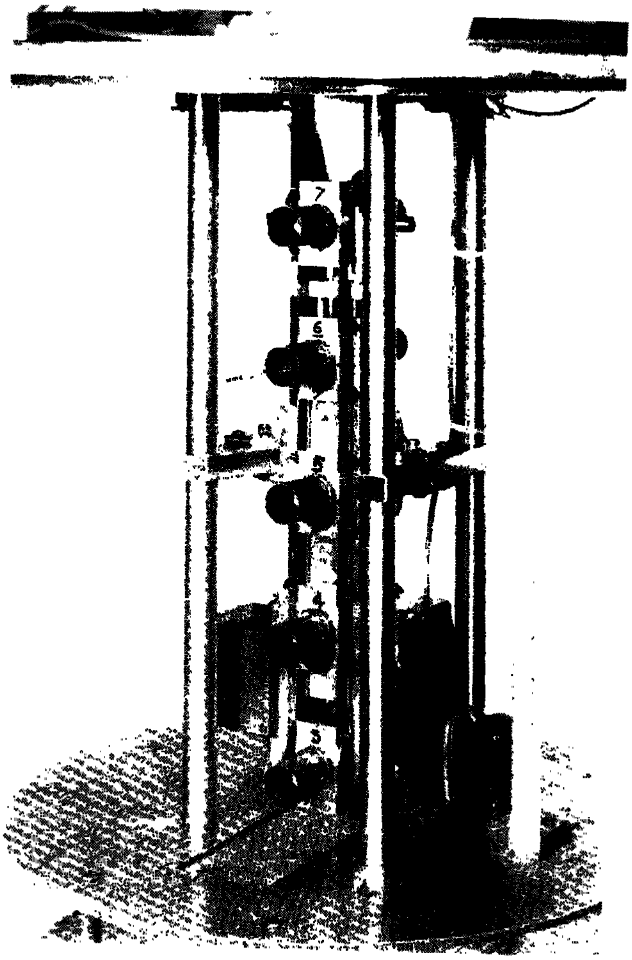

Fig. 6 - Geneva Drive Sample Changer

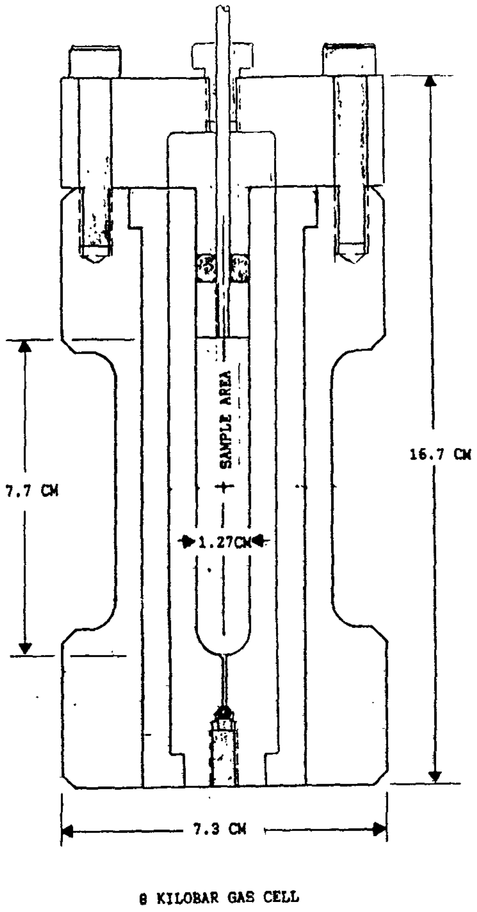

Fig. 7 - High Pressure Helium Gas Pressure Cell

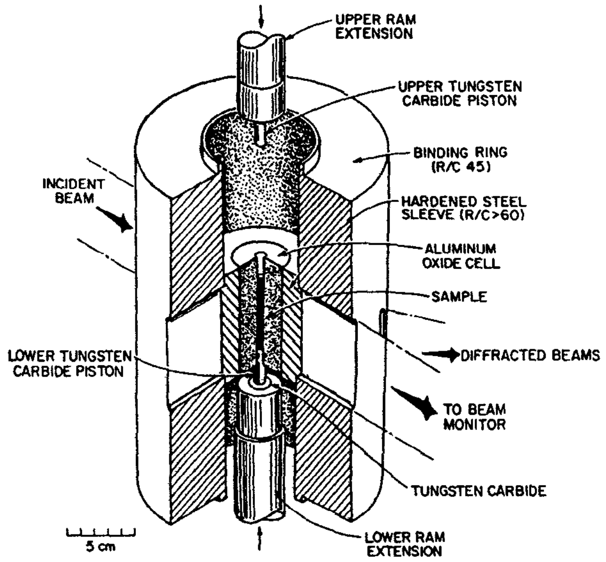

Fig. 8 - Room Temperature Piston-Cylinder

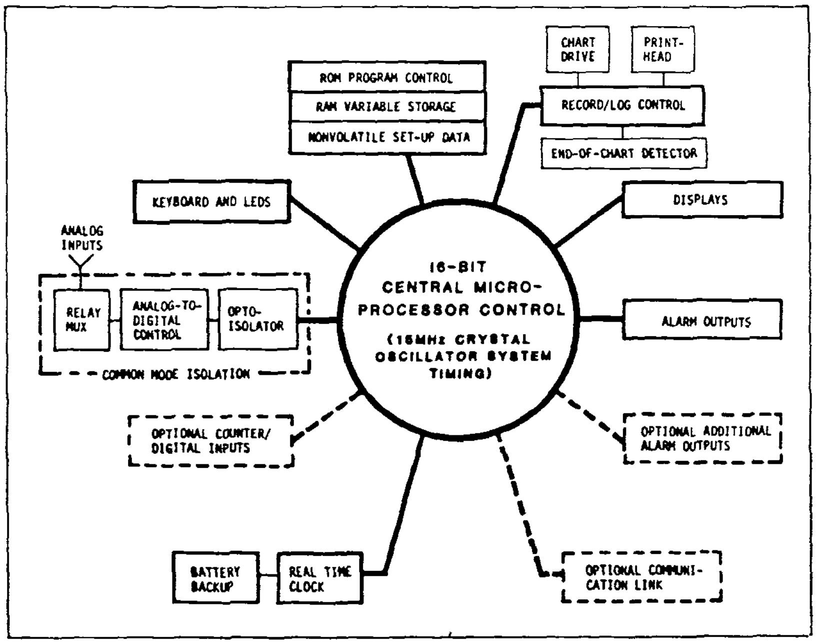

Fig. 9 - Chart Recorder/Data Logger

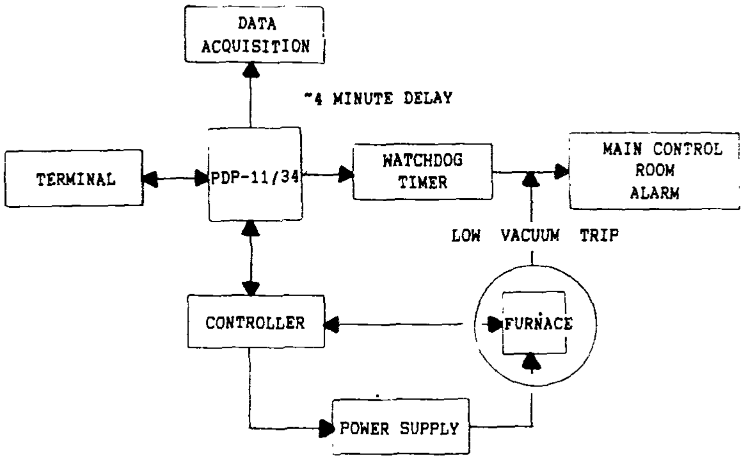

Fig. 10 - Furnace Controller Interface

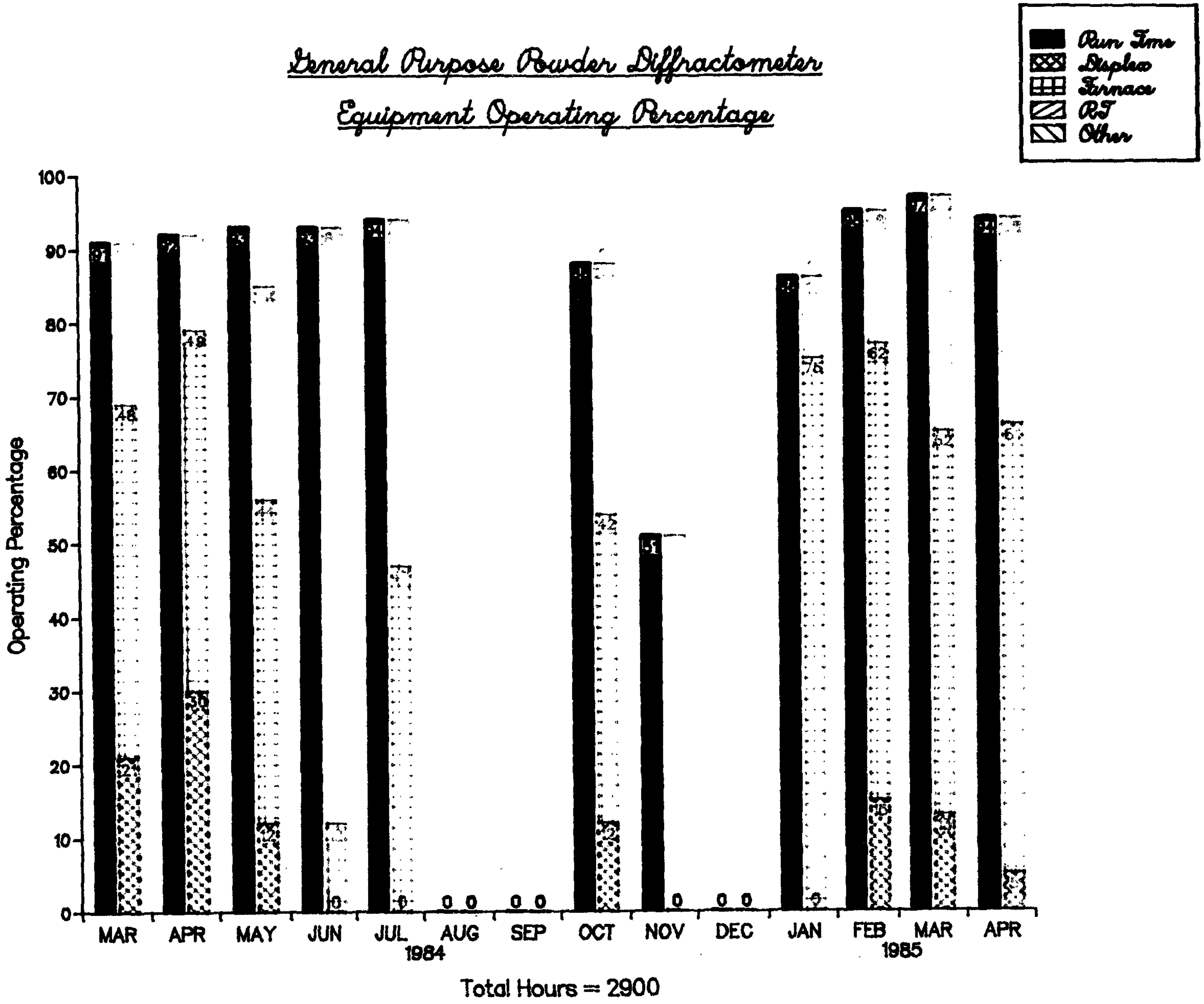

Fig. 11 – Stack Bar Graph of Peripheral Equipment (Displex, Furnace, Room Temperature, Other & Run Time) Usage as Compared with Monthly Operating Percentage