

Automated sample exchange and tracking system for neutron research at cryogenic temperatures

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An automated system for sample exchange and tracking in a cryogenic environment and under remote computer control was developed. Up to 24 sample “cans” per cycle can be inserted and retrieved in a programmed sequence. A video camera acquires a unique identification marked on the sample can to provide a record of the sequence. All operations are coordinated via a LABVIEW™ program that can be operated locally or over a network. The samples are contained in vanadium cans of 6–10 mm in diameter and equipped with a hermetically sealed lid that interfaces with the sample handler. The system uses a closed-cycle refrigerator (CCR) for cooling. The sample was delivered to a precooling location that was at a temperature of ~ 25 K, after several minutes, it was moved onto a “landing pad” at ~ 10 K that locates the sample in the probe beam. After the sample was released onto the landing pad, the sample handler was retracted. Reading the sample identification and the exchange operation takes approximately 2 min. The time to cool the sample from ambient temperature to ~ 10 K was approximately 7 min including precooling time. The cooling time increases to approximately 12 min if precooling is not used. Small differences in cooling rate were observed between sample materials and for different sample can sizes. Filling the sample well and the sample can with low pressure helium is essential to provide heat transfer and to achieve useful cooling rates. A resistive heating coil can be used to offset the refrigeration so that temperatures up to ~ 350 K can be accessed and controlled using a proportional-integral-derivative control loop. The time for the landing pad to cool to ~ 10 K after it has been heated to ~ 240 K was approximately 20 min. © 2007 American Institute of Physics. [DOI: 10.1063/1.2426878]

I. INTRODUCTION

Control of sample environment is required in many research applications at neutron beamlines facilities.^{1–4} Development and integration of controlled sample environments with beamlines is an important aspect of facility operations and user support. Neutron techniques are widely used to investigate physical, chemical, and biological phenomena in materials.^{5,6} Cryogenic sample environments are mainly used in two applications: (i) to minimize thermal motion of atoms to enable precise measurements of structure and (ii) to investigate low temperature phase behavior. In order to minimize thermal motion, the requirement is simply achieving a low temperature. Investigation of low temperature phase transitions also requires control of temperature in the cryogenic range. The availability of high flux neutron sources such as the Spallation Neutron Source^{7,8} (SNS) at Oak Ridge National Laboratory creates the need for remote and fast sample handling to enable a high throughput of samples in controlled environments.

Shah⁹ described an automated sample exchange system that can place up to 12 samples in a programmed sequence in a thermally controlled environment. The system can control sample temperature in the range of 20–150 °C. Sample exchange is accomplished by using a 12-sample carousel in which each sample position is equipped with a lead screw-driven linear actuator. The carousel is rotated with a stepper motor until the desired sample is above the neutron beam; the sample is then translated downwards into the beam location. Sample position is monitored by an optical sensor and slits in the carousel. Temperature control is achieved with a dedicated heater/sensor assembly attached to each sample.

An automated room temperature sample changer is installed at the glass liquids and amorphous materials diffractometer (GLAD) beamline at IPNS.¹⁰ The exchanger uses a chain-driven mechanism to move up to ten samples into the neutron beam in a programmed sequence. The system can operate unattended and allows the use of calibration samples as well as loaded sample vials of various diameters.

Volin and Bohringer¹¹ employed a sapphire “thermal switch” for a sample environment system that can control temperature in the range of 20–800 K using a closed-cycle

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refrigerator (CCR) and resistive heating. A sapphire link isolates the cooling stage from the heater at high temperatures where the thermal conductivity of sapphire is relatively low. At low temperatures when the heater is not deployed, the sapphire link provides a high conductivity connection to the cooling head.

The present work was focused on developing, testing, and integrating a system for remote control of sample exchange and logging of the sample sequencing in a cryogenic environment. The instrument was characterized in the laboratory. Tests were performed on the bench and in a mock up beamline configuration. The objective of developing the instrument is to achieve a short cycle time for sample exchange and cooling in a beamline environment. The instrument is being installed at the POWGEN 3 beamline¹² at the SNS where one goal is "round the clock" beam utilization without the need for continuous operator presence.

II. METHOD

A. Instrument concept and design

Requirements for the instrument were developed in consultation with beamline scientists and engineers at the SNS and through discussions at beamline user group meetings. The main requirements that were used to guide the instrument design are the following.

- High throughput of samples with remote computer monitoring, user-selected sequencing, recording of sample identification, and thermal history.
- Automation components that do not significantly compromise cryostat performance.
- Ability to sequence up to 24 samples in an unattended operation cycle with potential for increasing the sample load.
- Ability to probe samples over a range of temperatures from a few Kelvin to ~ 400 K.
- Compact, crane-compatible package including beamline "insert," cooling head, and sample exchange device.
- Simple load-and-seal sample encapsulation system.

An important design concept in the instrument was to decouple the sample from the relatively large mass actuator components that move the sample into the sample well. This concept was realized by placing the sample can on a "landing pad," a small aluminum structure that was directly coupled to the cold stage of the CCR. The landing pad has a cut out that engages with the lid of the sample can to provide a stable location that also defines the sample position relative to other components in the neutron beamline. In addition to direct heat transfer to the landing pad, the can transfers heat via helium exchange gas. After the sample was placed on the landing pad, the actuator was moved vertically upwards so its residual heat was transferred into the more powerful first stage of the CCR. In conventional cryogenic systems, the sample actuator or stick is fixed onto the sample and must be cooled along with the sample itself. The relatively large heat burden on the cooling system reduces the cooling rates that can be achieved.

The overall instrument concept was to integrate commercially available components using custom mechanical hardware and computer control software. The instrument design is illustrated in Fig. 1. The annotation in Fig. 1(a) defines the components of the system as used in the description. Samples were held in thin-walled vanadium tubes¹³ that were sealed under an atmosphere of helium. An identification number was marked on the lid of the can. Up to 24 samples can be loaded into an aluminum "carousel" [see Fig. 1(b)] that was located on the top plate of a 24 in. diameter stainless steel chamber that houses the sample handler and cryogenic components. Samples were loaded into the carousel using a tool that latches onto the lid of the sample can.

For beamline operations, the chamber can be installed as an "insert" that interfaces with a standard vacuum flange on the beamline. Components of the cryogenic system were made from Oxygen-free High Conductivity (OFHC) copper or aluminum alloy. Vacuum seals were made using indium. Surfaces to be sealed were "tinned" with indium and the seals were made by compressing indium wire or sheet between surfaces that were clamped together with bolts. A total of eight aluminized Mylar baffles were placed in the sample well to shield the cold landing pad from thermal radiation and to suppress convection of gas in the sample well. Two sets of the baffles are located between the first and second stage cooling rings, so that they effectively isolate the two stages when the sample handler is retracted. Cooling was achieved with a CCR (Ref. 14) that is specified to extract 35 W at the first stage at 41 K and 0.8 W at the second stage at 4.2 K. After the sample well was assembled, it was wrapped in aluminized Mylar and installed into a vacuum chamber. Temperature was measured at several points in the system using silicon diode sensors¹⁵ that were attached with thermally conducting epoxy resin.¹⁶ The output from the diodes was acquired using a Lakeshore model 340 controller. Temperature control was implemented by operating the CCR at full cooling power and energizing an electric heating coil that was wound onto the sample well near the landing pad. Operation of the sample handling, temperature control, and data acquisition were performed from a rack-mounted system via a computer.

To load a sample into the sample well, the carousel was rotated to align the selected sample with a screw-driven linear actuator that engages with sample can via a bayonet connector. After locking onto the sample, the selected can was raised out of the carousel and slowly rotated while a video camera reads the identification number. The can was then translated vertically downwards into the sample well where it was either held adjacent to the first stage cooling ring or placed directly on the landing pad. After delivery to the landing pad, the sample was released from the actuator by rotating the actuator rod. The sample remained on the landing pad until the actuator was re-engaged and the sample was moved vertically upwards and returned to the carousel. The sample insertion and retrieval sequence, duration of precooling, the time on the landing pad, and temperature control can be programmed from a LABVIEWTM-based computer program.

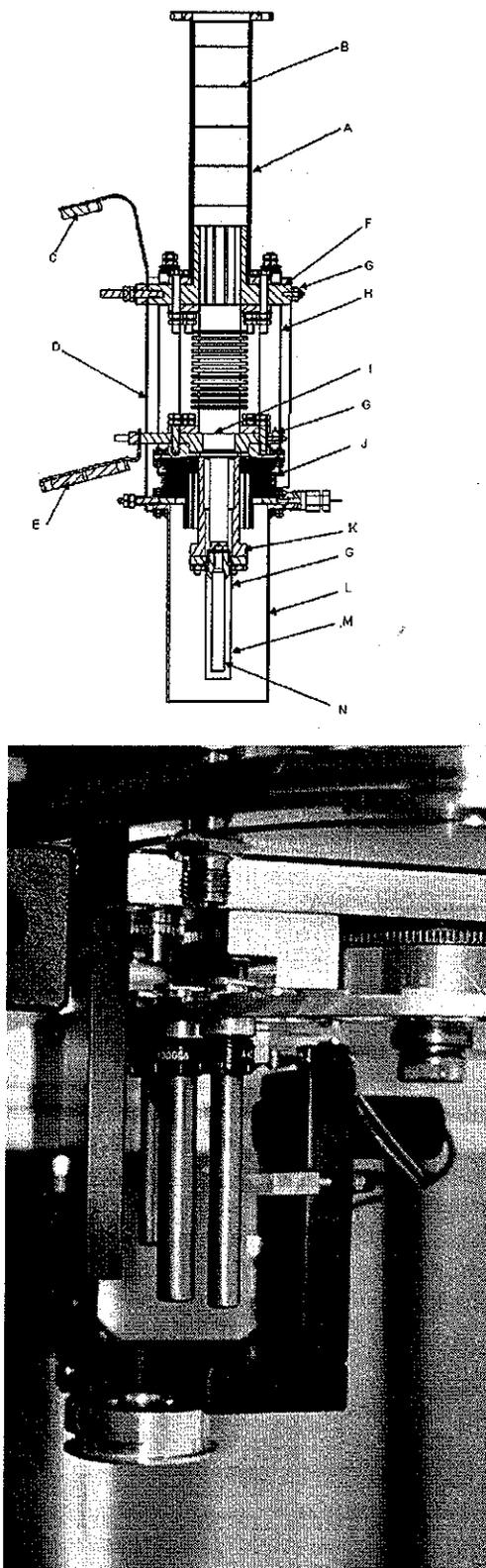


FIG. 1. (a) Diagram of the sample well. The main components are the following: A—sample well tube (stainless steel), B—baffles (8 in total), C—connection to the CCR first stage (~ 25 K), D—connection to the sample well heat shield, E—connection to CCR second stage (~ 4 K), F—first stage cooling ring, G—temperature sensor (3 in total), H—nylon/Pyrex standoff, I—second stage cooling ring, J—welded bellows, K—landing pad, L—sample well outer heat shield, M—sample well inner heat shield, and N—sample tube. The heater is wrapped around the area of the well adjacent to the landing pad. (b) Photograph of the sample carousel with 0.8, 1.0, and 0.6 cm diameter sample cans visible near the center of the image.

B. Sample can sealing and tracking system

Sample cans were sealed under dry helium that provides an inert sample environment and a heat exchange medium to increase heat transfer between the sample and the wall of the can. Sample cans are typically loaded in a helium-filled glove box. The use of a glove box makes the loading and sealing procedure time consuming and requires a relatively high degree of operator skill. In this work, a simple benchtop can sealing system was developed.

Diagrams of a sample can and the canning system are presented in Fig. 2. The vanadium cans were secured to an aluminum lid with high vacuum grade thermally conducting epoxy adhesive.¹⁶ The lid was sealed with an indium gasket that was formed *in situ* by a ridge on the inside surface of the can lid. The canning device can be used to evacuate and backfill the can with helium.

To operate the canning device, a can was loaded with sample and the gasket, pressure plate, and lid were assembled but not tightened. The can was placed in the canning device and the clamp was closed to seal the top nut against an o-ring. The can was evacuated by selecting the pump out position on the three-way valve. After evacuation, the valve was switched to backfill position so that the can was filled with helium to a positive pressure of ~ 30 kPa (5 psig). The evacuation and backfill process was repeated three or four times to remove residual air from the can. The can was filled with helium and the lid was sealed by rotating the top nut to compress the indium gasket. Additional indium gaskets were made by punching ca. 8 mm diameter disks from indium sheet.

Samples were tracked using a unique identification number that was engraved onto the side of the lid of the cans. Part of the operating sequence of the sample handling program was to read the identification number with a video camera. The output of the camera was processed using an optical character recognition software that stores the sample ID with the thermal history and other sample information in a data file.

C. Instrument testing

Instrument testing was performed in several stages. Throughout the testing and setup, data from the temperature probes were logged using a software package, "SEDAN LITE," that was developed at the SNS for data acquisition from cryogenic sample environments.¹⁷ The program provides up to four channels of data on a common time base and it allows acquisition at user-selected rates and for periods of several hours. The first stage of testing was performed on the bench to validate operation of the actuator mechanism and sample exchange. Preliminary cooling rate measurements were made using a liquid helium cryostat.¹⁸ Results of these tests are reported elsewhere.¹⁹

Initial tests of the cryogenic sample exchange instrument were made using a CCR at the SNS. These tests used a rig developed for the evaluation of sample environment components [SNS Multiple Adaptive Sample Housing (SMASH)]. The layout of the SMASH rig is illustrated in Fig. 3. The SMASH uses an ARS model DE210 CCR. The cooling rate

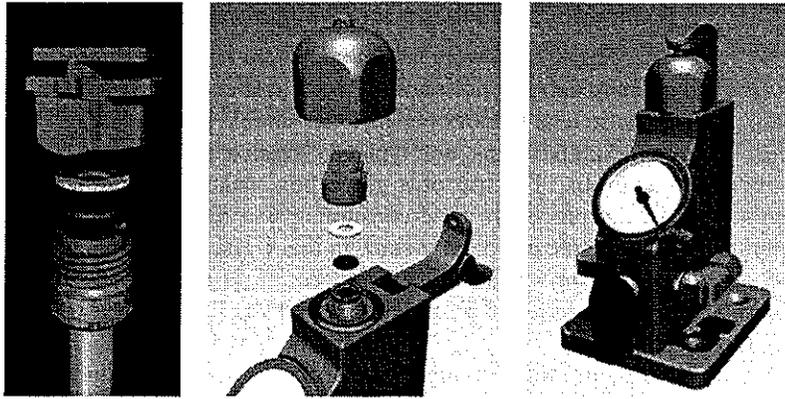


FIG. 2. Sample canning system and sample can components. Left to right, sample can components cap with bayonet engagement slot, pressure plate, indium gasket, and can with threaded top with identification number engraved on the outer surface. Center diagram of can closing device showing the can and lid components and the nut that is used to make the seal. Right, view of the can closing device. Gas enters the device through the port on the right and a vacuum pump is connected to the port on the left.

of selected samples was measured. The sample materials were chosen to bracket a range of thermal properties (heat capacity and thermal conductivity), density, and form (fine powder and 1–3 mm diameter shot) that would represent a variety of sample types that might be encountered in a general user facility. Information about the samples is summarized in Table I.

Sample materials were loaded into a 0.8 cm diameter vanadium can that was instrumented with a silicon diode temperature sensor (Lakeshore model DT-670B-SD silicon diode) that extended a distance of ~ 1 cm into the sample can. The mass of the can, sensor, and aluminum cap was approximately 12 g. The mass of sample ranged from ~ 0.5 to 4 g and it is given in the fourth column of Table I. The can was backfilled with helium to a positive pressure of ~ 20 kPa (3 psig) at *NTP* and sealed.

The output of the temperature sensors was acquired using a CryoCon model 34 controller. Three measurement points were used: (i) inside the sample can, (ii) the stick that was used to manipulate the sample and was located ~ 10 cm above the variable temperature insert (VTI) during the measurements, and (iii) the VTI that is equivalent to the landing pad. Cooling experiments were performed with the sample well filled to a positive pressure about 20 kPa (3 psig) of helium throughout the experiment. A few runs were made with the system evacuated to a pressure of 10^{-1} Pa.

Subsequent tests were performed using the complete sample exchange system with integrated CCR as described at the beginning of this section. Measurements were made with 0.6, 0.8, and 1.0 cm diameter sample cans. In some cases the cans were loaded with copper shot. Cooling rate tests were performed under a variety of conditions including with pre-cooling of the sample at the first stage of the cryostat. The

TABLE I. Sample materials. The mass presented in column 4 is based on 0.8 cm diameter sample can filled to a depth of 2 cm.

Material	Bulk density (g cm^{-3})	Mass (g)
Alumina powder	4.0	1.48
Alumina shot	4.0	1.92
Copper powder	8.9	2.15
Copper shot	8.9	4.32
Nylon pellets	1.2	0.54

use of temperature control by using the resistive heating coil powered by the Lakeshore controller was also investigated.

III. RESULTS

A. Sample exchange

The time required to acquire the video image of the sample identification was approximately 1 min. An additional 1 min was required to translate the sample into the sample well and release it on the landing pad and retract the actuator.

B. Measurements with SMASH rig

Temperature-time data were acquired from three points in the system: the sample can using an embedded sensor, the stick that was used to move the sample into the system, and the variable temperature insert that was attached to the cold stage of the CCR. Data were collected for samples of copper powder, alumina powder, and nylon pellets. Temperature versus time data for copper powder are plotted in Fig. 4. The samples were introduced into the system at a temperature of approximately 300 K and the initial temperature of the stick was approximately 300 K. The sample was moved into the system and placed on the VTI in a period of approximately 1 min. The forms of the temperature-time data for the three

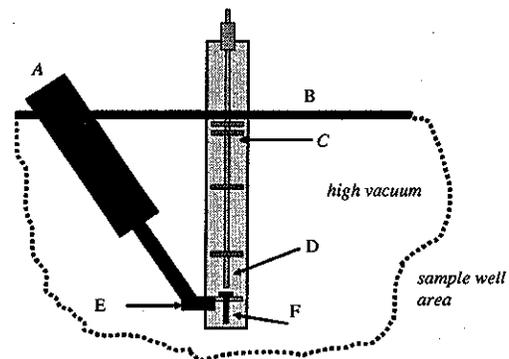


FIG. 3. Diagram of the SMASH rig illustrating the main components of the system. A—CCR (ARS model DE210), B—sample well top plate, C—sample well tube, D—stick with baffles mounted along its length, E—variable temperature insert (VTI), and F—sample can and landing pad. The temperature sensors were located at the stick, VTI, and sample can.

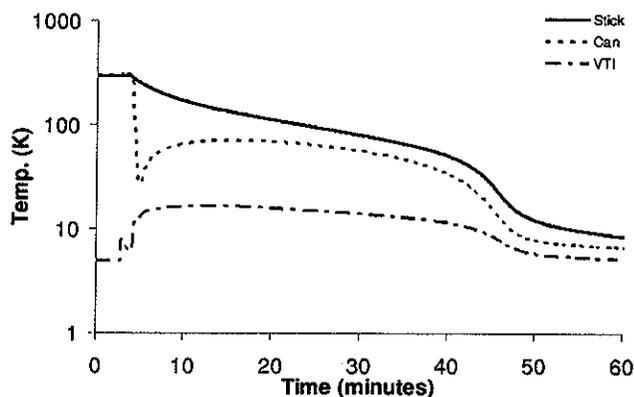


FIG. 4. Temperature-time data measured for copper powder in the SMASH rig with an early development model of the sample management system. This test demonstrated that loaded sample cans can be cooled very quickly but thermal coupling between the stick and sample can must be eliminated to achieve high cooling rates.

sample materials are similar with only slight differences in the time for equilibration. The trends in the cooling rate data are the following.

- (1) Almost immediately upon introduction of the sample, the temperature of the VTI rises to ~ 15 K, the temperature of the can decreases to ~ 25 K, and the temperature of the stick starts to decrease steadily.
- (2) After 2–4 min, the temperature of the can rises by 30–40 K over a period of a few minutes. Then the temperature decreases over a period of 30–50 min and equilibrates with the VTI. During this period, the temperature of the stick continues to decrease and it equilibrates with the VTI after approximately 50 min.

Differences in cooling rate between different samples were relatively small. All the samples cooled rapidly to ~ 40 K, then a slower cool down to base temperature (~ 6 K) occurs. The sample with the largest heat content (copper) takes slightly longer to cool than those with lower heat content.

A cooling rate measurement was performed with the system evacuated to a pressure of approximately 10^{-1} Pa. After a period of 2 h, the sample was at 50 K and the stick was at 170 K.

C. Measurements with automated sample exchange system

Measurements in the automated sample exchange system were made using a system that was instrumented with sensors on the sample well and the first and second stages of the CCR. The effects of precooling the sample at the first stage of the CCR were investigated in detail. Data shown in Figs. 5(a) and 5(b) show results obtained for cooling rate measurements. In all the measurements, the sample well was filled to a positive pressure of approximately 20 kPa (3 psig).

The results of initial cooldown of the sample well are shown in Fig. 5(a). From 300 to 120 K, the temperatures of the first and second stage cooling rings track each other closely. The sample well cools at a similar rate to the cooling rings, but its temperature is ~ 20 K higher. At approximately

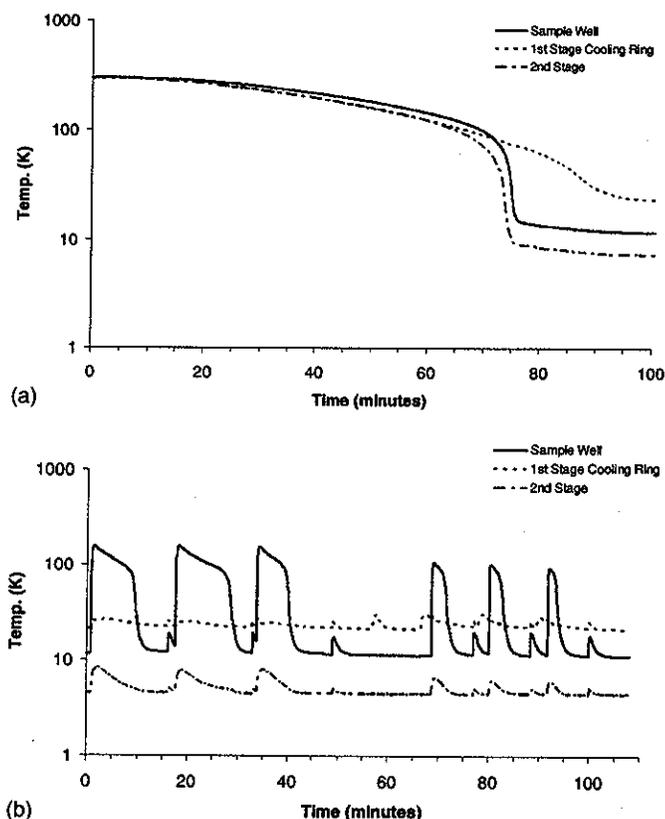


FIG. 5. Temperature-time data for cooling experiments in the sample handling system shown in Fig. 1. (a) Cooldown of empty system. Note the rapid decrease in temperature starting at ~ 100 K due to the decreased heat capacity of copper at low temperature. (b) Effect of can size on cooling rate. Note that the sample temperature is measured indirectly by sensors in close proximity. When a room temperature sample is lowered into the first stage precool zone, the nearby sensor shows a temperature spike and recovery. A similar spike and recovery is observed when the sample is delivered to the landing pad. The stick is retracted to the precool position immediately after sample delivery, and the sample well returns to base line (about 10 K), indicating that the sample is fully cooled.

100 K, the cooling rates of the sample well and first stage increase sharply and approach their stable values about 75 min after the CCR is first turned on. The temperature of the first stage cooling ring continues to decrease until about 90 min have elapsed. Stable temperature values of approximately 25, 10, and 4 K are reached for the first stage cooling ring, sample well, and second stage cooling rings, respectively, after 100 min.

The cooling rate measurements on cans were all made after the system had been cooled for several hours and reached its stable temperature condition. The sample well was filled with helium to a positive pressure of 20 kPa (3 psig). Information about the mass of the sample cans is given in Table II.

The effects of precooling on the total time to cool three sizes of sample can, 0.6, 0.8, and 1.0 cm in diameter, are shown in Fig. 5(b). The total time to cool decreases as the sequence continues and the sample manipulator cools.

The effects of precooling were investigated using sample cans containing copper shot. The shortest times for the total cooling cycle were obtained in cases where the sample was precooled for a period of 1–3 min. Longer precooling times decrease the time to cool from 40 K, but increased the over-

TABLE II. Dimensions and mass of the sample cans with lids and filled to a depth of 2 cm with copper shot. (Results for a can filled to a depth of 2 cm with copper shot. The payloads will be correspondingly smaller for lower density sample materials. Calculated values of payload for cans filled with a similar volume of aluminum oxide are shown in parentheses in the fifth column of the table.)

Diameter of can (cm)	Mass of empty sample can plus lid (g)	Mass of sample (g)	Total mass (g)	"Payload" (%)
0.6	9.0	2.6	11.6	22 (12)
0.8	9.1	4.3	13.4	32 (18)
1.0	9.1	6.6	15.7	42 (24)

all cycle time to cool to the temperature of the landing pad. The results of the cooling experiments are summarized in Table III. Note that the small increase in temperature was seen a few minutes after the sample is placed on the landing pad due to the heat input from the sample manipulator when the sample is retrieved. During the period that the sample is on the landing pad, the manipulator tip is located at the pre-cooling position, close to the first stage cooling ring.

Results of experiments in which the electric heater was operated are shown in Fig. 6. The data were obtained using a 50 Ω heating coil. The maximum temperature reached was ~ 240 K with the CCR operating at maximum cooling capacity. When the power to the heater was turned off, the sample well re-cooled to its base temperature in a period of 20 min. Work is in progress to optimize proportional-integral-derivative (PID) control algorithms needed to control temperatures at user-selected set points. The heating power will be limited to prevent heating of the indium seals above 350 K, about 80 K below the melting point of indium.

IV. DISCUSSION

A. Sample exchange

Exchange of samples under remote computer control was achieved using a carousel containing 24 samples. The sequence of samples and the sample identification was recorded by computer. Samples were exchanged into a cryogenic environment and the cooling rates were measured. The present system was demonstrated using a single carousel that held 24 samples. Additional samples can be loaded into the carousel using a manual exchange tool. For very high

TABLE III. Cooling time with precooling at the first stage of the CCR. (Results for a 0.8 cm diameter can containing 3.3 g of copper shot. Total time is the time to cool from room temperature to approximately the temperature of the landing pad, 12 K.)

Duration of precool (min)	Cooling time on landing pad (min)	Total time 300–12 K (min)
0	10.7	10.7
1	6.9	7.9
1.9	4.9	6.8
3	4.2	7.2
7	4	11

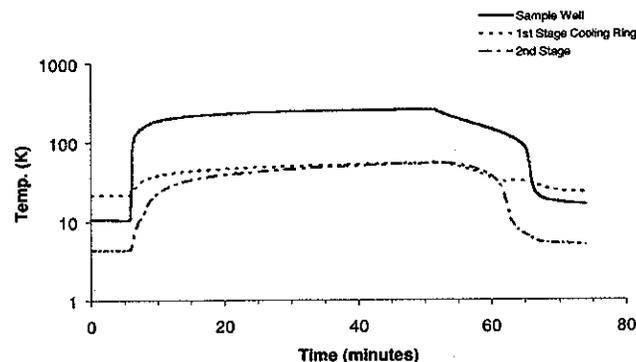


FIG. 6. Temperature-time data for experiments using a 50 Ω heating coil.

throughput of samples, automated reloading of the carousel would enable a virtually unlimited number of samples to be used.

B. Cooldown rates

1. Measurements in the SMASH rig

When the sample is introduced, the temperature of the VTI rises to ~ 15 K and the temperature of the can rapidly decreases to ~ 25 K. The temperature of the stick decreases steadily. After several minutes, the temperature of the can rises by 30–40 K over a period of ~ 5 min and then decreases over a period of 30–50 min before it equilibrates with the VTI. During this period, the temperature of the stick continues to decrease and it equilibrates with the VTI after approximately 50 min.

The "bounce" in the temperature of the can shortly after it is introduced is attributed to heating of the can by energy transfer from the relatively large mass of the stick. The precise mechanism of heat transfer between the stick and can was not conclusively identified. The stick is introduced into the system when there is a light positive pressure of helium in VTI. The downward motion of the stick will displace cold gas, tending to push it up to the sides of the sample well as the stick moves downwards. After the stick is introduced, the cold gas will warm up as heat diffuses into it, at the same time the displaced gas tends to move downwards, displacing warmer gas from the bottom of the well. In addition, conduction moves heat from the stick toward the cooler sample can. A detailed analysis of the heat transfer mechanisms that result in the temperature "bounce" is beyond the scope of this article and is being investigated in separate work at the SNS.

The cooling rate measurements in SMASH showed that the sample can cooled at a much higher rate than did the stick. This result confirms the idea that decoupling the large mass of the sample manipulator from the sample itself allows for fast cooling by decreasing the heat burden on the CCR.

2. Automated sample exchange system

The data presented in Fig. 5(a) show that the system reaches its equilibrium temperature approximately 100 min after the start of cooling. The data presented in Fig. 5(b) and in Table III indicate that precooling at the first stage of the CCR where there is ~ 35 W of available cooling power re-

duces the total cooling time from 300 to 12 K from approximately 11 to 7 min. The total time to cool a sample from 300 to 12 K is achieved by using approximately 2 min of precooling with the configuration that was used.

The beneficial effects of precooling are not strongly influenced by the sample can size or the size of the sample itself. We attribute this to the following: (i) at the precooling location, the can is still attached to the manipulator stick and so that both the can and stick are being cooled, and (ii) the sample cans are of similar mass regardless of their size and the sample adds only a small extra heat load (see Table II). The idea that the heat load from the stick can be a significant heat burden is further confirmed by the data from runs where empty cans were cooled. After the first cycle when the manipulator has started to cool, the excursion in the sample well temperature when the sample was introduced is significantly smaller than on the first run.

By decoupling the sample can from the actuator, the mass of material that must be cooled to low temperatures is reduced. As a result, relatively fast cooling rates can be achieved. Cooling from room temperature to ~ 12 K in 7 min is accomplished. In the measurements in SMASH, even though the sample was decoupled from the stick, the cooling time from 300 to 12 K was on the order of 50 min. In the present system, baffles shield the landing pad from the stick and this appears to have a large influence on the cooling time, reducing it by almost a factor of 5 without precooling and a factor of 7 when precooling is used.

From the data presented in Table II, it is apparent that even with the decoupled system, most of the mass that is cooled is the can. Payloads are from about 10%–40% of the total mass of the can plus sample depending on material and can size. As a result of the rather small payloads, the differences in cooling rates of different sample materials and forms of material are quite small. Efficient heat transfer between the sample and the can is achieved with a helium exchange gas. The cooling rate could be increased slightly by reducing the mass of the can and lid assembly.

C. Future developments

Work is in progress to optimize PID control algorithms to allow the system to operate at user-selected temperature set points up to 350 K.

There are two methods that may be used to increase the cooling rate of samples. The first is to increase the "payload" of the can by reducing the mass of the can and lid assembly. The current arrangement weighs ~ 9 g, mainly contributed by the aluminum lid. A reduction in the mass of the can lid would decrease the amount of energy that needs to be removed to cool the sample. An alternative method of increasing cooling rate is to increase the capacity of the cooling engine either by using closed-cycle refrigerators or adding a liquid helium cooling system. The use of additional refrigerators would be preferred if unattended operation is needed.

As requirements increase, it is expected that there will be

a need to increase the operating temperature range of the system above the current upper limit of ~ 400 K. In order to increase the limit, indium seals would need to be replaced with higher temperature materials such as lead or aluminum. Additional heating capacity would be needed in the system. Increasing the temperature range that can be accessed would decrease the sample throughput.

Increased automation would require development of capabilities for automated reloading of the sample carousel. A potential method of accomplishing this is to employ a simple robot "pick and place" device to move samples from a large capacity storage area into the sample carousel. With automated sequencing and an air lock on the chamber that houses the carousel, continuous operation could be accomplished.

Sample exchange in a cryogenic environment was demonstrated using an automated system that can be operated under remote computer control. The time required to cool samples from 300 to 12 K was 7–12 min. The cooling rate was insensitive to sample composition or the mass of the sample. The use of precooling of the sample decreases the total cooling time by almost 50%. Filling the sample well and the sample can with low pressure helium is essential to provide heat transfer and to achieve useful cooling rates.

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- ¹⁷http://www.sns.gov/instrument_systems/sample/SE_science.shtml
- ¹⁸Janis Research Company, Wilmington, MA, model SVT-XG-400T.
- ¹⁹J. E. Rix, J. K. R. Weber, L. J. Santodonato, S. E. Hammons, J. Hodges, M. Rennich, and K. J. Volin, *17th Meeting of the International Collaboration on Advanced Neutron Sources, Santa Fe, NM, 25–29 April 2005*, edited by G. Russell, J. J. Rhyne, and B. V. Maes (LANL, Los Alamos, NM 2006) Vol. 3, pp. 1086–1095.