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An Automatic Liquid-Nitrogen Filling System For Use With Solid-State X-Ray Detectors

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Abstract

An automatic system for the filling of liquid nitrogen-cooled X-ray detectors is described. A novel feature of the system is the pre-cooling of the transfer tube from the nitrogen source to the detector when filling is initiated. This avoids some problems inherent in commercially-available systems. The control electronics center around an inexpensive microcontroller chip. This allows for the inclusion of safety features, whilst keeping the circuit construction very simple and straightforward.

Introduction

Experimental techniques using synchrotron radiation at X-ray energies have steadily increased in precision and sophistication over the years; consequently the use of energy-dispersive X-ray detectors, based mainly on high-purity germanium crystals, has become widespread. This type of device has now practically become the standard type of detector used in most techniques where energy resolution is a prime requirement, and particularly in X-ray fluorescence spectroscopy.

At the Daresbury SRS three multi-element detector arrays [1] and a number of single-element detectors are in regular use and these numbers are likely to grow. Such detectors need to operate at liquid nitrogen temperatures, and have their own cryostats built into them which need to be regularly topped up with liquid nitrogen. Any partial warming of a detector can seriously degrade its performance, and occasionally can result in expensive damage. At best, filling by hand is time-consuming and potentially hazardous. In addition, an experimental set-up can require a detector to be positioned such that access to it for filling is difficult.

There appear to be very few remote-control filling systems on the market at the present time. Those that are available are expensive to buy, and on investigation none of them appear to be suitable for use on the X-ray spectroscopy facilities at the SRS. The system described in this report has been developed primarily as a response to this situation. A prototype has been constructed and successfully installed at the materials science facility on beamline 9.3, where it is presently in routine use. The detector system here uses a Canberra type 7935-7 "big MAC" cryostat, and is permanently mounted in a downward-pointing vertical position. Access to the filling port is very restricted, so that in-situ filling by hand is practically impossible, and a remote-control method a real necessity.

System Requirements

For any filling system a large pressurized storage dewar is commonly used as the source of liquid nitrogen. Liquid is conducted to the detector cryostat through a length of insulated tube, termed a "transfer line", under pressure from evaporation in the storage dewar itself; no pumping is

required. The manual outlet valve of the storage dewar is left permanently open, and the flow of liquid from the dewar into the transfer line is controlled by a solenoid-operated valve. Ideally, the detector cryostat would contain a liquid nitrogen sensor placed so as to indicate the level of liquid in its own reservoir, and signal when this was full. However, this is hardly ever the case, and the fill-state must be estimated or determined externally. In commercially-available filling systems, the control valve is usually opened for a fixed length of time, empirically determined as that necessary to completely fill the detector cryostat. After this time the cryostat is deemed full and the valve is automatically shut. Alternatively, a liquid nitrogen sensor may be placed at the venting outlet of the cryostat. In this case liquid may be allowed to flow until an overflow is sensed, or until a safety limit on the filling time has been exceeded.

One obvious problem with such simple systems is that the transfer line and any gas in it will be at room temperature prior to the valve being opened. Upon opening the valve this relatively hot gas is inevitably blown through the detector cryostat before being followed by progressively colder gas, and then liquid. The transfer line itself will take some time to cool to liquid nitrogen temperature, which it must before any liquid can traverse the whole length of it. This can result in liquid being boiled off from, or even literally blown out of, the cryostat for up to several minutes before it can begin to fill with liquid from the storage dewar. There is thus the possibility of provoking a partial warm-up by the very process of refilling. In addition, after the valve is closed, liquid remaining in the transfer line will boil off through the detector cryostat, and may force some liquid out of it. The longer the transfer line, the worse these problems become.

If the filling system operates by opening the valve for a fixed time, there is a problem caused by the fact that the rate of liquid transfer will vary with the pressure in the storage dewar, which in turn is largely governed by how full it is. Opening the valve for a fixed length of time, unless that time always exceeds the maximum time required to actually fill the detector cryostat, cannot guarantee a complete fill every time the system is operated. On the other hand, using the method of filling to overflow inevitably ensures that when the filling system shuts off, the detector cryostat is full (unless automatically stopped in response to a fault condition or time limit).

Physical Description of Prototype

The filling system which has been developed is based on the overflow-sensing technique. It has, however, been elaborated on so as to avoid the problems mentioned earlier, and also draws on experience gained at Daresbury using a commercially-made autofill system with a large array of gamma-ray detectors [2]. The mechanics of the system and its control electronics are shown in block form in figure 1. For the prototype system, a storage dewar of 160 litres capacity, and working pressure 3.0 bar, has been used. The transfer line to the detector is a 5 metre length of flexible low-temperature-resistant plastic tubing (proprietary name "Tygothane") of internal diameter just under 10 millimetres, encased in foam rubber insulation which is in turn wrapped in braided nylon. The flow of nitrogen is controlled by two solenoid-operated valves, located one at each end of the transfer line as shown in the figure. In the original set-up, a pressure-relief valve, operating at approximately 1.5 bar (not shown in the figure) was included in the transfer line in case of any ice blockages. Operational experience has shown this to be unnecessary for this type of detector-dewar, and it is no longer included. Liquid nitrogen sensors are placed one at the open end of the transfer line (sensor A) and one on the overflow from the detector cryostat (sensor B). The sensors themselves are simply carbon resistors. Each is housed in a drilled block of nylon, and mounted so as to be directly in the nitrogen flow without impeding it. Extra lengths of tubing are attached to the downstream sides of the sensor blocks to direct the overflow nitrogen safely away from personnel and equipment.

The Control Program

All monitoring and control is from a central electronic unit. This is built around a microcontroller chip running a program written specifically for this application, plus interfacing circuitry to the sensors and to the solenoid valves. The control program monitors the status of the sensors' output-circuits, and generates the appropriate control signals to operate the valves. Two pushbutton switches are the only manual controls required to operate the system. A "start" switch initiates the filling sequence, which then runs under the control of the program until the detector cryostat is

full, unless a fault condition causes the sequence to abort. A "reset" switch is used to set the system to an initial "ready" state and can also be used to manually abort a filling sequence that has already been started.

When a normal filling sequence is initiated the following operations and processes take place:

1. Valves 1 and 2 are both opened. Nitrogen from the pressurized storage dewar flows through the open-ended transfer line bypassing the detector (figure 2a). The nitrogen first leaves the storage dewar as cold gas; this purges the transfer line and starts to cool it. As liquid is first produced it boils off in the transfer line, which continues to cool. When the line is cold enough, liquid can reach the open end, to be detected by sensor 'A'.
2. When liquid is detected at sensor 'A', valve 2 is closed. Liquid then flows into the detector cryostat (figure 2b). When this is full, the nitrogen overflow is detected by sensor 'B'.
3. Once liquid is detected at 'B', valve 1 closes, shutting off the nitrogen supply. At the same time valve 2 is re-opened for a few minutes (figure 2c). This allows any liquid remaining in the transfer line to boil off and vent to air. Valve 2 is then closed and the system goes into a waiting state.

Whenever liquid is first detected by either of the sensors, the control program calls a timing subroutine which checks for the continuous detection of liquid over about five seconds, after which the appropriate valve operation is carried out. If at any time during this period the sensor does not detect liquid, then the routine exits to the main program and resumes monitoring the sensor at the normal intervals. This prevents false triggering of the valves by any small splashes of liquid that may precede the main flow.

The circuitry associated with the sensor resistors is designed to detect if either sensor has failed through becoming either open-circuit or short-circuit. Throughout the filling sequence the control program monitors the sensors for these fault conditions. If sensor 'A' fails during stage 1 of the sequence, or if sensor 'B' fails during stage 2, then the control unit immediately terminates the filling sequence and goes to stage 3; the nitrogen supply is shut off, and the transfer line is allowed to vent.

During both of the first two stages of the sequence the elapsed time from the operation of the valve, or valves, is monitored. If a preset time has elapsed without nitrogen being detected by the relevant sensor, then a fault condition is assumed by the control unit. This could be caused by partial failure of a sensor (e.g. a change in its temperature coefficient, such that it cannot cause its comparator circuit to trigger). Alternatively there could be a blockage in the system, or the storage dewar could run out of nitrogen. In any case the sequence is immediately terminated and the transfer line vented as before. It can happen that a time-out is caused by the storage-dewar pressure being low due to the level of nitrogen in it being low, although still sufficient to fill the detector. In such cases, the program allows the system to be reset and re-started even though the transfer line is being vented. Once liquid appears at sensor 'A' then valve 1 will close as normal and filling will resume.

The main part of the control program is shown in flow-chart form in figure 3. For the sake of clarity the operations which monitor for sensor faults or for the sequence being aborted have been omitted from the figure, as these are repeatedly executed between each of the main operations. Likewise the timing subroutine to prevent false triggering of the valves is not shown. However, all of the timing subroutines work in the same way, namely by counting down from an initial value and flagging when zero is reached. The program itself is written in a proprietary assembly language. All of the software and hardware necessary for compiling the program and downloading it to the microcontroller are commercially available and will not be detailed here.

Liquid Nitrogen - Sensing Circuits

The nitrogen sensors consist of 0.125W carbon resistors of the Allen-Bradley type. Their resistances measure 390 Ohms at room temperature, and rise to over 500 Ohms in liquid nitrogen. Effectively they are being used here as thermistors; figure 4 shows the complete circuit for one sensor. The temperature is sensed as the voltage drop across the sensor resistor, which is driven by a constant current of 15mA; this figure has been chosen as a trade-off between optimising the voltage drop across the sensor while keeping self-heating to an acceptable level. The voltage drop across the

sensor is fed to the positive input (pin 2) of an LM311 comparator, shown as IC1 in the figure. The negative (pin 3) input, essentially a threshold corresponding to liquid nitrogen temperature, is derived from a ZNREF100 voltage reference chip via a 10k-Ohm potentiometer. This voltage can be adjusted to compensate for minor changes in sensor characteristics with repeated temperature cycling. This adjustment also allows variations within resistor tolerance limits to be compensated for. The threshold voltage is set such that when the sensor is immersed in liquid nitrogen the voltage at pin 3 of IC1 will exceed that at pin 2. The comparator output, which is connected to one of the inputs of the microcontroller, and shown in the figure as the "liquid sensed" line, will then switch from 5V to 0V. Two other comparators, IC2 and IC3, compare the sensor voltage drop against thresholds corresponding to abnormally high (for IC3) or low (for IC2) sensor resistances. These in turn correspond to the sensor being open and short-circuit respectively. If either condition should arise, the 'sensor failure' line switches from 5V to 0V, and an indicator LED is illuminated.

Control Circuitry

The control circuitry for the whole system, shown in figure 5, is centred around an Arizona Microchip PIC16C55 programmable microcontroller. Up to twenty pins on this device can be individually user-defined as either inputs or outputs, the definition being made in the user's software. The use of a microcontroller allows for a substantial amount of sequential logic to be performed in a single chip, and also allows for some modifications to be implemented by reprogramming rather than by reconfiguring circuitry. For this application, the logic status of the sensor-circuit outputs, plus the "start" and "reset" switches, are read by the control program as single-bit digital inputs. Remote control is made available by having input sockets wired in parallel with the start and reset switches (not shown in the figure); taking the input to ground duplicates the action of the switch concerned. Those pins of the microcontroller that are defined as outputs provide the switching-signals for the solenoid valves, and for the indicator LEDs.

A 50Hz oscillator circuit provides an external clock for the microcontroller, which executes one program instruction for every four clock cycles. For the prototype a PXO1000 crystal-controlled oscillator chip

has been used, but precision timing is not really necessary here and any fairly stable oscillator circuit would be adequate. The initial number for the main timeout subroutines is provided by a set of DIL switches, read by the control program as a parallel 8-bit binary input to an on-board register. Once a suitable time limit to cover separately both nitrogen transfer and detector filling has been determined empirically the switches can be set accordingly.

The control signals to open each solenoid valve are stepped up through a relay-driver chip (CA3262E, not shown in the figure) to switch a solid-state relay, which in turn applies 240V a.c. power to the solenoid valve. An earlier version using conventional relays was found to suffer from noise radiated by the relay contacts. The control unit is located remotely from the solenoid valves themselves, so neon indicator lamps are connected across the relay outputs to show which valve is powered at any one time.

Performance and Further Developments

The prototype has been in routine use for several months now, and has proved both satisfactory and reliable. So far the system has always been used by manually starting the filling sequence. The "start" and "reset" input sockets would, however, allow it to be started and if necessary aborted, by some automatic device.

Several improvements to the original design are envisaged, such as increasing the number of diagnostic indicators, adding an audio alarm etc. More importantly, it is recognized that the timing method used, requiring the same time limit for both transfer-line cooling and for detector filling, is rather inflexible and could cause problems if significantly different limits were required. In the present set-up, with working pressure of 3.0 bar, a transfer line length of approximately five metres and a detector capacity of 7.0 litres, a time-out limit of about eight minutes for both stages of the filling sequence has been found to be adequate, so that there is no problem as yet. However it is intended to provide more flexible timing by using two independent timing circuits which the control program would start, and which would flag the program when a limit was reached, rather than doing the timing in the program itself.

At present the sensors are checked periodically for any drift in characteristics, and their comparator-threshold voltages adjusted as necessary. It is intended to incorporate a more robust type of sensor, such as a platinum resistance elements [3], or some form of capacitive sensing, so that such checks become unnecessary. Several possibilities are at present under consideration.

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Figure Captions

Figure 1. Block schematic of filling system.

Figure 2. Filling sequence operations.

Figure 3. Flow-diagram of main control program.

Figure 4. Schematic of liquid nitrogen sensing circuit.

Figure 5. Schematic of connections to PIC16C55 microcontroller.

Figure 1.

a. Both valves open. Transfer line purged and cooled.

b. Valve 2 closes. Liquid directed into detector cryostat.

c. Valve 1 closes and valve 2 re-opens. Transfer line vented.

Figure 2.

Figure 3.

*+12V supplies to LM311's not shown

Figure 4.

Figure 5.