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ISIS Spectroscopy and Support Division Cryogenics Review - June 1997

Prof. Dr. M Meissner

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ISIS Spectroscopy and Support Division

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Contents

1 General Remarks

At ISIS the user-based research programme is structured by a scheduled beam time of six 4-week-cycles in a year with about ten days of maintenance in between [R1]. Within this dense schedule the task of ISIS is to organise and to perform user-controlled experiments on - an as close as possible - a perfect "nose-to-tail" basis [R2]. From this outline, it is already clear that the efficiency and the reliability of both the experimental equipment and the technical staff must both be of high standards.

The support to visiting researchers and experimental apparatus, is in addition provided by the sample environment (SE) section headed by Ian Bailey within the User Support Group and formed by four persons for the cryogenic equipment and three persons for high temperature and pressure apparatus (plus one person for electronics) [R3]. In addition, three mechanical engineers of the Project Engineering Group collaborate with the SE group in designing new equipment for both in-house and external manufacturing.

The present status of the SE group could best be acknowledged by the broad range of experimental apparatus which has been used successfully in around five hundred experiments per year serving more than twenty instruments [R4, R5]:

- 17 Orange-type Cryostats
- 22 Closed Cycle Refrigerators
- 3 He^3 Sorption Cryostats
- 2 He^3/He^4 Dilution Stick Inserts
- 1 Cryomagnet
- 6 High-Temperature Furnaces
- 5 High-Pressure Systems
- a variety of rotating centre sticks for cryostats and for closed cycle refrigerators
- a variety of high-pressure centre sticks and cells for cryostats and for closed cycle refrigerators
- a variety of sample changers for cryostats and water-bath regulated systems

With this equipment the ISIS SE section provides an outstanding service to experimenters where the sample environment parameters can be controlled over a wide range (including positioning of the sample in respect to the neutron beam):

- low temperatures T = 50mK - 300K
- high temperatures T = RT - 2000°C
- high pressure up to p = 150kbar from 77K to RT; p=10kbar at T=700°C
- high magnetic field (vertical) up to B = 7.5Tesla

It is the policy of ISIS managers to keep and to develop this high standard as a 100% service for the external users. However, from this "full service" it follows that the visiting scientists - which in the majority are Ph.D. students or post-docs in their twenties from university labs - are no longer involved in the advanced or even basic experimental techniques. In cases of succesfully performed experiments the users are naturally very happy with this procedure. On the other hand, in cases when sample environment equipment fails to function, the users will complain about the reliability of cryogenic apparatus on the basis of standard consumer arguments rather than as collaborators [R6]. The move to tickets further emphaises this trend. At ISIS one should keep an eye on this development since it becomes evident that young scientists increasingly perform "keyboard experiments" only.

Sampling the Comments from the User Satisfaction Forms [R6] and the Instrument Lost Time Reports [R7] of year 1996 indicates some common problems in cryogenics (liquid coolant transfers, LHe-capillary blockages, temperature stability, time-scale of cool-down) which eventually constitute a one-third share of overall instrument loss time. Instrument loss time averages at 4.7% over the past two years. Losses of ~1.5% (ascribed to cryogenic problems) on 2200 instrument days p.a. represent 33 days at a nominal cost of ~£330,000 p.a.

However, from a cryogenic point of view - depending on the level of vacuum and cryogenic hardware and supporting electronics presently used - it will be impossible to run such equipment expecting almost 100% reliabilty. A majority of the problems arise from the status of technology for the various metallic joints in a cryostat (see section 3.3) and from the applied LHe-technology (see section 2 and 3.2). Also, it should be noted that cryogenic success depends on the training and the expertise of all the experimenters including SE staff, instrument responsibles and users (see section 5).

Before discussing these aspects in detail, two technical remarks will be given here:

- In the experimentall hall at many instrument sites pressurized gas bottles of nitrogen and helium can be seen. Thinking about the manhours to maintain the supply and the safety aspects as well, today's laboratory standard would be matched by the installation of pipe lines (served from liquid supply) with valve outlets at some strategic points and then using flexible lines to the instrument site.

- In the experimental hall many vacuum pumps (continuously running!) are not connected to a central gas exhaust system. A non-expensive plastic-pipe installation would benefit the health of the people working there.

2 Helium Quality

The impurity contamination of LHe in the transport vessels and/or the cryostats is a difficult problem and, in my opinion, impossible to solve. I would not recommend to ISIS a full-cycle LHe-management with liquefier and recovery and compressor system, mainly because of the additional manpower needed for this. However, for a well-maintained ISIS-owned LHe-management system one cannot exclude contamination by both basic physics and human errors.

My argument is that one has to live with a certain level of frozen atmospheric gases in the LHe-vessel. So, effort must be concentrated on clean LHe-transfer into a clean cryostat.

At HMI-BENSC we now have good experience with a second generation of syphons (made by Abingdon Scientific) which consists of two parts. One part for the cryostat (2m straight and rigid in the case of ISIS) which stays in place all the time (albeit raised up) and is closed with a blank. The other part belongs to the LHe-vessel (1.50m straight and a 90° 2m flexible part) and can be linked (under slow LHe-flow from both sides) for a transfer. At the bottom of the vessel-side syphon a filter is located; a similar filter can be used on the bottom end of the cryostat-side syphon.

After many days of needle valve (NV) usage and transfers, the cryostat will become contaminated, thus endangering the NV and capillary throughput. It should be discussed with AS to install another filter which is permanently mounted on the NV housing tube of the orange-cryostat and is the only intake for the liquid in the direction of NV and capillary.

Presently at ISIS the orange-cryostats are mostly operated in the warm-NV-mode. This means that the cold-NV is open all the time, which gives maximum chance for frozen gas particles to move into the flow impedance region. As proposed below, the operation with cold-NV mode would have the NV mostly closed all the time which diminishes the chance for blocking.

It is difficult to estimate how much the "open He-recovery system" of ISIS contributes to a LHe-contamination in the orange-cryostats (LHe-bath, sample tube). However, also from environmental arguments, it may be worthwhile thinking about the advantages of a full GHe-recovery and compressor-system (another question remains on the re-selling of the GHe to the factory).

3 Design Aspects

3.1 Cryostat Design

The orange-cryostats at ISIS are by now about ten years old. Like at HMI-BENSC an increased number of defects (zero in earlier years) is now observable. On the other hand, the design and manufacture of these cryostats is the same style at AS. For instance, Oxford Instruments manufactures the VARIOX-BL, a cryostat very similiar to the ORANGE, which has holding times for the liquids of up to four days! If one wishes to stay with AS's orange-maxi one should ask for an improved design of lower boil-off (superinsulation) and for increased volumes of the two baths (for ISIS it would be preferable to increase overall height since the handling of the cryostat is then much easier on the instrument site).

3.2 Needle Valve Design and Automation

In the past, the controlling of LHe-flow by a needle valve proved to be a major source of problems. Basically, at low temperatures the flow of liquid helium is controlled by changes in the needle opening of about a tenth of a millimeter. Comparing this order of magnitude to the rough mechanical design of a cryostat, it is evident that long term stability cannot be expected from the original cryostat design. Many improvements have been tested and, at present, AS sells a shared ILL-design which regulates the pumping pressure according to a set-point with a PID-controller which actuates the needle via a 6bar pressure transducer.

After a year of testing and modifications, 14 Auto-NV's have been manufactured by AS, and it now seems that our users (at HMI) are familiar with the device and accept (and understand) the operation. Once it works, the benefit is great since the pumping pressure (for cold NV-operation, see below) remains constant and unattended over many days. The PID-controller can be serial interfaced to the computer in order to open the NV for fast cool-down and close it for set-point operation by software routines.

The investment for automated AS NV's is substantial (ca. £1,500 each). However, the orange-cryostat can be operated more smoothly over the whole temperature range T from 1.5K to 300K.

3.3 Material Junctions

As pointed out in the General Remarks section a persistent problem in cryostats is the status of technology for material junctions. With the aim of increasing reliability, the following techniques for metallic material junctions (which were intended to provide vacuum isolation) are used:

Method	**Characteristic**
adhesive seal	thermal stresses and ageing introduce leakages
soft-(Pb)-solder joint	thermal stresses and chemical acid reactions introduce leakages
vacuum grease tapered seal	good where no mechanical strength is needed
In-sealed flange	thermal stresses and mechanical hysteresis introduce leakages
hard-(Ag)-solder joint	good for years
Al-SS friction junctions	perfect, when made by specialists
Al-welding	perfect, when made by specialists
SS-welding	standard and perfect

An example of inadequate technology is the status of ISIS's He^3/He^4 Dilution insert, where both the age of the device and the number of epoxy and soft-solder junctions are sources of He^4 leakages. In today's technology Oxford Instruments manufacture their He^3 HELIOX and He^3/He^4 KELVINOX sticks solely with SS-welding and a small number of hard-solder joints.

During the last five years at HMI-BENSC we have had many failures due to epoxy joints when operated at low temperatures. I do not recommend the use of this technique. In respect of thin foil material which is intended to provide reliable vacuum isolation at low temperatures it is necessary to develop better metallic junction techniques (laser-beam welding, e-beam welding).

4 Operational Aspects

4.1 Standard Cryostat Operation

Service

At ISIS a high degree of experience with the orange-maxi cryostat has been accumulated over the years. I am content that following my observation, it can be accepted that all standard procedures like preparation, evacuation, GHe-purging, leak testing and LHe-filling are performed with careful expertise.

With respect to possible LHe-blockages in the NV and capillary I would recommend to checking - on a regular basis - the throughput at RT by pumping GHe through the open and closed needle valve and measure both steady-state pumping pressures (NV open: $p > 10\text{mbar}$, NV closed: $p < 0.1\text{mbar}$). When a NV has to be changed, great care has to be taken in order to prevent pieces of the sealing PTFE-tape falling into the capillary intake (pressurise VTI-pumping port and clean the bottom of the LHe-bath with a tube connected to a vacuum cleaner).

In addition, for service intervals, the capillary inside the OVC should be opened (i.e. cut and re-sealed with a small indium-sealed brass flange or copper-tube soldered with hard silver joint). Then the capillary can be cleaned mechanically (wire technique) from grease which may accumulate during an experimental period.

The indium seals in the cryostat (lower part of aluminium-sample tube; capillary) can leak below superfluid temperatures. Therefore, on service, a 1.5K-test over 24h is sometimes helpful to clarify irregular situations such as fluctuating low temperatures and sudden high boil-offs. In particular, a test with 1bar He into the sample tube under these conditions (simulating users' sample changes) is a crucial test. A He-leak tester monitoring the OVC has to be installed and a data logger set-up (matching OVC during warm-up).

Preparation

At ISIS the cryostats will be prepared in the cryogenics laboratory for the experiment and then moved with a vehicle and then craned to the experimental site. There is agreement that the mechanical vibrations and stresses arising from this procedure are not good for the LN_2 and LHe reservoirs and supporting tubes.

It should be assessed whether a ground floor area in R55 could be arranged (fenced, about 5m x 4m) for the preparation and cool-down of the cryostats. Also, in some cases the cryostat could stay all the time at the instrument site (for example the muon facility). Another and easier possibility is to improve the cryostat transport trolley with anti-vibration mounts. In addition cryostats should always be lifted using ropes and slings, which are flexible, and never with chains alone.

Cold Valve Mode

At ISIS for orange-cryostat experiments in a temperature range T from 4.5K to 300K (600K) the warm-valve mode is presently used. In contrast to the cold-valve mode there are some disadvantages in using the warm-valve mode:

1) The forced flow of liquid helium for cooling is due to a small over pressure $\Delta p = 0.1$bar, only.

2) The NV is fully open all the time which increases the probability of blocking.

3) For experiments below 4.5K the user has to change the set-up to the pumped cold-valve mode. If one operates the orange-cryostat in the overall range T = 1.5 to 300K (600K) in the cold-valve mode solely, evidence suggests better and safer operation by the user:

a) A faster cooling rate can be controlled with the wide dynamic range of over pressure $\Delta p = 1$bar.

b) The NV is almost closed for 95% of all operation time, reducing contamination and blockage risks.

c) The cold-valve operation provides continuity over the whole temperature range.

However, it is clear that the operation of the NV in the cold valve mode is complicated when compared to the warm-valve mode. Roughing vacuum equipment is needed all the time including a pressure gauge for monitoring the pumping pressure between 0.1mbar and 1bar. Also, an Auto-NV-controller is needed for long-term unattended and stable operation. I recommend trying out the prototypes of AS at ISIS, where AS should provide a separate housing vessel for easier and fast tests in a LHe-vessel.

Once the function of the Auto-NV is acceptable, the performance, especially below 4.5K, is perfect. Operation at higher temperatures will benefit from lower LHe-consumption at a given (and for increasing) set-point(s). For faster cool-downs (and increased LHe-consumption) the NV can be opened for a short time-span. It should be noted that for the maxi-orange-cryostat the internal relaxation times of the VTI and the sample stick are fairly long above 50K (0.1 - 1h magnitude) due to the metallic masses involved. To speed up the time between temperature changes the thermal masses must be reduced by redesign.

4.2 Special Cryostats

Operating special cryogenic systems at very low temperatures (i.e. 30mK to 1K) and/or at high magnetic fields remains complicated even today and is far removed from performing standard cryogenics. In contrast to closed cycle machines and orange-cryostats there may be as many as ten discrete vacuum-/liquid-isolated volumes. A defect occuring in only one volume can ruin the whole set-up. Identifying a defect during an experimental set-up might take many hours or even days. Therefore, increasing the reliability of these systems is possible only by keeping a second system as a back-up. Like the orange-cryostat concept the use of two or even three special cryostats of the same type may concentrate the efforts for a better performance. A good example at ISIS is the three He^3 sorption cryostats of OI which function in identical ways and - despite the complicated cryogenics involved for condensation and cooling of the LHe^3 - work very reliably.

ISIS is in an exceptional geographical situation with two cryogenic companies (OI and AS) located nearby. With respect to the manpower available at ISIS for cryogenics, on these special cryostat systems it is recommanded that maintenance and repair of these systems should be done at OI or AS. As an important side-effect the nearby company responsible will benefit by improving a prototype into a standard system from the operational experience it gains at ISIS in realilstic conditions.

Cryomagnet

The OI cryomagnet with a 7.5 Tesla vertical magnetic field accommodates large sample sizes and good horizontal access for neutron scattering. It is about five years old. As the cryostat was under repair for new wiring in the ISIS SE laboratory during my visit, I was able to get a good impression of the quality of the inner bodies.

In the past two years AS has manufactured two vertical magnet cryostats (for HMI-BENSC) and one horizontal magnet cryostat (for the ILL/HMI/Dubna co-operation) where the medium field split-pair magnets were bought from OI. The prices of these magnet cryostats were very reasonable since standard Maxi-orange-cryostat bodies were used. This gives full advantage of the orange-concept where the VTI and all the top-plate ports of the cryostat are the same. For future purchases of cryomagnets from AS or other companies, one should adhere to this approved design since operation by ISIS staff and users will benefit from their experience in the past with basically the same cryogenic system (with design improvements to the NV and the cryogenic liquid consumption as mentioned above).

He^3 Sorption Cryostat

As already mentioned above, the new He^3 sorption cryostats (OI-HELIOX) at ISIS should have a high degree of reliability for performing user experiments. The He^3 system is completely sealed into the stick design and there is no external gas handling system. The He^3 cooling and condensation is managed by electronic devices. The He^4 precooling is standard NV plus rotary-pump-technique, which is a weak link in the system. However, OI's NV-technique with a fine SS-thread and stepper motor control proved to be very stable over several weeks during tests at HMI-BENSC.

Although the ITC-503 hardware for electronic control of temperature and long-term cooling power is both very reliable and user friendly, the complete status of operation can be visualized by OI's PC-based software product ObjectBench which provides a simple and intuitive data logger plus full remote operation for the user. At BENSC I found that users, especially from the UK, are familiar with this software and can run temperature and magnetic field variations on the spot. Considering the price of a PC plus the software licence (ca. £2,000) it is of some value for the user to operate the HELIOX/VARIOX-system by remote control. For instance the data log files can be very helpful in the case of troubleshooting. On the other hand, it should be considered that running a PC parallel to the cryogenic system needs additional skilled efforts for both staff and users.

He^3/He^4 Dilution Fridge

The arguments used above for operation of the He^3 sorption cryostat can be applied in the same way to a dilution system. However, in the past no He^3/He^4 sorption pump system has been sucessfully used or even offered by cryogenic companies. Thus, dilution systems had to be operated with external gas handling systems which circulate the mixture outside the cryostat. Specific to small dilution sticks (as for the TBT-system or the OI KELVINOX) is the fact that during circulation a substantial quantity of the mixture is at RT (and LN_2 with the cold trap). This can influence the performance (i.e. cooling power) of the small mixing chamber significantly. When the outside condensation pressure increases, the amount of the inside liquid decreases which eventually shifts the He^3/He^4 phase separation line out of the mixture cell, and hence the cooling power decreases rapidly (i.e. the base temperature can no longer be reached.

At OI a prototype version of a twin sorption pumped dilution unit has been built and tested successfully and has been shipped to their first customer. In my opinion this new technique (which was published ca. 10 years ago) marks a new step in very low temperature technology offering the same sealed system as for the He^3 sorption pumped HELIOX and the same easy-to-use but sophisticated electronic management. It will be worthwhile for ISIS to keep in contact with to this development at OI and observe the reliability of this system. As a by-product I expect that the price of this system would be considerably lower than that for a conventional system with external gas handling (see below).

At HMI-BENSC we now have a good one-year experience with the first KELVINOX dilution stick adapted to the orange-cryostat concept (i.e. 1.5K-cooling by static He^4 exchange gas only). Problems during initial cool-down originated from the external gas handling system which introduced partial blockages in the cryogenic part of the condenser line due to the migration of oil-vapour during initial high circulation rates. Also, we cannot reach the earlier base temperature of 30mK which we believe is due to fluctuations of the phase separation line due to different condensation pressures and due to an effect not fully understood of a part of the LHe^3 which collects on top of the dilution unit (instead of equalising levels with the LHe^3/LHe^4 in the still). The dilution stick itself is - up to now - very reliable and operation is very intuitive using the PC-based LabView software which controls the VARIOX, the gas handling system and the various thermometers and heaters. As mentioned above, the advantage of the remote control technique is that the operational parameters are constantly monitored and their graphic presentation provides useful information on the status of the dilution process.

In any case, a dilution system will always be an expert system where additional effort on each separate experiment must be invested into operation, service and testing. This is further emphasised by the fact that the dilution system will be operated in different experimental areas or on different instruments, with the consequence that the system has to be taken apart and put together frequently. Beam time allocation needs to recognise this aspect when determining periods required for experiments.

Temperature Control

At ISIS the low temperature thermometry of most of the equipment is based on the RhFe thermometer which is read by the Eurotherm-controller and which subsequently is connected to the instrument computer. This technique is proven and seems to work with good results both in performance of temperature regulation and read-out.

There are two small disadvantages: i) on the instrument itself the Eurotherm reads only Ohms, so that one cannot read the temperature near to the orange-cryostat. This is inconvenient on set-up and when troubleshooting; ii) the 1.5 Ohm-range for T<4K requires high, ~1MOhm resolution for accurate thermometry. One is committed to item i) without changing to a new electronic device; but item ii) would require individual calibration curve testing with high-precision low resistance calibrations from time to time.

5 Information

5.1 Training Procedures

The staff of the sample environment section obviously have a good deal of long-term experience in vacuum and cryogenic techniques. However additional training courses on the basic theory of thermodynamics, solid state physics and materials science would support that practical knowledge.

I am not however proposing mainstream lectures on an engineering school level. I recommend instead a lecture and practical course on the construction and function of a LHe-cryostat operating with a VTI and the related electronic components (thermometers/heaters/wires and subsequent electronic devices). There should be an expert lecturer from a nearby university or possibly from Oxford Instruments to give these courses.

5.2 Documentation

It is certainly necessary to provide all staff members and users with a condensed description of the construction, function and specifications of all sample environment equipment. The documentation could be in the form of a printed brochure or as hardcover information sheets or on web-pages. In either case it supports the needs of the user for their experiment and is useful for troubleshooting as well.

I have added to this report [note: kept by ISIS SE section] a CD-ROM with all data files (WORD 6.0) of our sample environment brochure (plus some new files on the function of the Auto-NV) where you might use the orange-drawings and ILL-HTF-drawings for your own documentation projects. Also the documentation on sample cells for cryogenic systems, furnaces and high-pressure cells (similar to those in the ISIS User Guide) is helpful for the users in order to prepare an experiment.

A final word on publishing documentation on web-pages: if one considers the many modifications to the equipment in a high-class user-oriented facility it becomes necessary to up-date documentation material on an annual basis. For printed material this issue is more difficult and both time and money consuming than is the case for the updating of electronic data files. Today it is fairly easy to produce documents with appropiate software and then transform them into html-format. By this method one can have an up-to-date version on the web and can develop a data source for a professionally produced brochure which can be issued as hard copy documentation on a longer time-scale.

6 Summary of Recommendations

As a summary I have grouped my recommendations below into four classes of interest. The relevant remarks can be found on the page number given in brackets. I trust that the recommendations within the first two classes should support the "orange-business" for smooth and safe operation at ISIS. The options listed in the last two classes are more difficult for me to judge since investment of effort and money and, subsequently, new technology is involved. I find that for these items collaboration between ISIS and BENSC is helpful: like ISIS's experience with the HELIOX and at BENSC with the KELVINOX or in using a shared data base for documentation of similar equipment at both institutions.

Improvements with respect to handling gases and liquids for cryostats:

- Service gases (GHe and GN_2) should be supplied from lines rather then bottles (p.2)
- Exhaust gases from the rotary pumps should be collected by an exhaust line (p.2)
- LHe-transfer is improved with two syphons which stay with the cryostat/vessel (p.3)
- LHe-transfer into the cryostat and NV and capillary should pass through filters (p.3)
- Use Cold Valve Mode together with Auto-NV device of ILL/AS (pp 3 + 4 + 6)
- Cryostats should be optimised for low LHe-consumption (p.4)

Increasing the reliability of cryostat function:

- Material joints which isolate cryogenic vacuum should be purely metallic (pp 4+5)
- The NV and capillary system should be regularly tested and serviced (p.5)
- Indium-seals should be tested and serviced for superfluid leaks (p.5)
- Cryostats should not be shocked by vibrations during transport particularly when fully loaded with cryogenic fluids (p.5)

Special Cryostat options:

- Use the expertise of AS and OI (p.7)
- AS can manufacture good price/value Magnet-Maxis (p.7)
- He^3 HELIOX operation can be remotely controlled by PC/ObjectBench (p.7)
- He^3/He^4 KELVINOX operation is less complicated with new twin-sorb (p.8)

Documentation for staff and users:

- Training of staff by specially designed courses on the equipment (p.9)
- Publishing of manuals on web-pages with yearly update (p.9)
- Use SE-BENSC files (p.9)

Reference to ISIS-Documentation

R1 ISIS Schedule 1997/98 (version of Jan. 1997)
R2 Instrument-specific SE schedule for cycle 2/97 (daily schedule list)
R3 ISIS Spectroscopy and Support Divison: Personnel SE (version of Dec. 1996)
R4 ISIS User Guide: Sample Environment (version of April 1997)
R5 Instrument specific SE requests for cycle 1/97 (pie chart statistics)
R6 Comments from Users Satisfaction Forms - ISIS Operational Year 1996
R7 Instrument Lost Time Faults for cycles 3,4,5/96