

STANDARD OPERATING PROCEDURE

Procedure:	Using X-ray cryosystems
School/Department:	School of Molecular Bioscience
SOP prepared by:	Mitchell Guss, Nick Coleman
Version:	SMB037.3

Section 1 - Personal Protective Equipment

1. Lab coat or lab gown
2. Proper enclosed footwear
3. Hair tied back if long
4. Thick temperature-resistant gloves (when handling liquid N₂)
5. Full face shield when transferring liquid N₂

Section 2 – Potential Hazards + Safety precautions

1. **The X-ray equipment is hazardous. Do not use it unless you have been personally instructed by the equipment custodian.** Just reading this SOP is not sufficient to allow safe use.
2. **Liquid nitrogen poses a suffocation risk** – if a large quantity of liquid N₂ is spilled, it can displace the air in the room. Only use in well-ventilated spaces. In the X-ray lab, ensure both doors to the room are open when handling liquid N₂.
3. Large quantities of liquid N₂ or other liquefied gases should only be used in areas equipped with an oxygen sensor and alarm – check with your supervisor that this is the case, know what this alarm sounds like, and what to do if it goes off.
4. Liquid nitrogen poses a risk of cold burns. Wear thick gloves when handling.
5. Read the risk assessments for X-ray equipment and Cryogenics before starting work to ensure you understand the risks and take appropriate protective action.
6. Workers with pre-existing medical conditions (e.g. allergy, immunocompromised state, chemical sensitivity) and workers who are pregnant or expecting pregnancy must consult with their supervisor AND medical specialist AND the university's WHS services before performing this procedure. If there are any serious concerns expressed by any of these individuals, this task must not be performed.

Section 3 – Equipment and Procedure

1. Equipment: Cryosystems and dewars, room 283; Air handling systems, room 284.

Pre-experiment check list:

2. Check the Liquid N₂ levels in the operating dewar prior to commencing an experiment (if they need filling see below)
3. Check dry air flow to the appropriate instrument (Figure A, next page)

Filling the dewars:

4. Before filling or topping-up the dewars, put on the appropriate safety equipment including thick cold-resistant gloves and face shield.
5. Open the front and back doors to the laboratory.
6. In the event that the oxygen alarm sounds, turn off the supply dewar and leave the laboratory immediately until the oxygen level returns to normal.
7. Connect the delivery hoses and place the fill tube in the cryosystem dewar. Remove the other fill plug.
8. Start filling slowly using the control valve on the supply dewar until the tubes have reached low temperature. Avoid rapid boil off.
9. When filling is complete, shut off the fill valve and wait until the tubes have warmed so that they can be removed safely.
10. Place empty supply dewars outside the laboratory and inform the Service Centre so they can be refilled.

Operation:

11. Start-up Oxford cryosystem. Allow 30 minutes to reach the standard operating temperature of 100K.
12. When the experiment is concluded use the program knob on the cryosystem (see manual) to select END. The system will then warm up to room temperature and then shut off.



LEFT: Figure A – air control unit

This unit directs the dry air supply to the two beamlines “mama” and “papa”.

The dry air can come from the compressors located next to and below this unit OR from the nitrogen supply in room 201. The flow gauges show the actual flow to the instruments and should be between the red marks during operation. The flows can be adjusted with the two silver knobs in the centre near the bottom. The two top black switches (top is PAPA) control whether the air comes from the compressors (switches pointed to the right) or from the nitrogen supply (pointing to N₂). The third black switch from the top selects whether the nitrogen comes from 201 or a bottle in the corridor. The latter is only used for regenerating the drying tubes. The switch marked BENCH is used to direct nitrogen flow to the bench in 285 for drying tube regeneration. The picture above shows both lines operating with dry nitrogen from 201.

In this case the master flow valve from 201 (see Figure B below).

LEFT: Figure B –master control valve



The LN₂ master control valve from the boil off in room 201 is shown circled at the top of this figure. This valve should be off when the dry air is being sourced from the compressors. It is possible to have one system operating from the compressor and the other from nitrogen in room 201.

Section 4 – Disposal / Spills / Incidents

1. In the event of a spill of a large amount of liquid N₂, shut off supply lines or cylinders if safe to do so, and leave the lab immediately. Evacuate other nearby personnel also.
2. Any incidents that involve large spills of cryogenic agents (>1 L) or injuries to personnel need to be reported immediately to your supervisor and via the online reporting system. Near misses (situations that appear hazardous) should also be reported in the same way.

Section 5 – Repairs / Certification / Validation

1. The two instruments were purchased from Marr research in a bundle with the two image-plate detectors but are supported directly by:
Oxford Cryosystems Ltd
3 Blenheim Office Park
Lower Road
Long Hanborough
Oxford
OX29 8LN
United Kingdom
Tel: **+44 (0)1993 883488**
Web: <http://www.oxcryo.com/contact-us/>
2. The most common fault is loss of vacuum in the delivery tube as evidenced by a failure to reach the operating temperature of 100 K and/or by condensation on the outside of the tube. The vacuum can be restored by pumping down the system in the School of Chemistry. Contact the person in charge of the X-ray crystallography equipment in Chemistry for help.
3. The dry air pumps require periodic maintenance. They either fail to start or do not produce the desired air flow. These can be fixed by the Service Centre using kits ordered from Oxford Cryosystems. Spare kits should always be kept in the X-ray laboratory. While the air dryers are off-line the instruments may be used with N₂ gas from room 201.

Section 6 – Relevant safety data sheets

1. Be familiar with the SDS for liquid nitrogen, and for any other chemicals being used in the X-ray analysis.

Section 7 - References

1. Risk assessments and SOPs for X-ray equipment (SMB038) and Cryogenics (SMB010).

SOP Consultation, Training and Approval

Print names and enter signatures and dates to certify that the persons named in this section have been consulted/trained in relation to the development and implementation of this Standard Operating Procedure. WHS Representative (WHS Committee) certifies that consultation has taken place.

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