Sample Environment Equipment Handbook

Australian Centre for Neutron Scattering

Australian Nuclear Science and Technology Organisation

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Australian Government

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Preface

Please Read This First

This manual is designed to give you information about our sample environment equipment to help you choose the best fit for the parameters you want to achieve. We offer a large range of equipment across multiple ACNS neutron beam instruments which can make choosing difficult. There are a number sample environment options to achieve similar parameters on each neutron beam instrument. To help with your initial selection please consider the following table of the sample environment equipment most commonly selected for our neutron beam instruments. If your parameters are not here, please read on or get into contact with us on (sample_environment@ansto.gov.au) for advice.

Diffraction			
Echidna	CF-7	Top-loading cryostat	4-800K
Wombat	CF-8	Top-loading cryostat	4-800K
Koala	CF-2	Bottom-loading cryostat	4-800K
Kowari		Ambient temperature mount	
Spectrometers			
Taipan	CF-1	Bottom-loading cryostat	4-800K
Taipan: Be Filter	CF-8	Top-loading cryostat	4-800K
Sika	CF-3	Bottom-loading cryostat	4-800K
Pelican	CF-11	Top-loading cryostat	1.5-800K
Emu	CF-12	Top-loading cryostat	1.5-800K
Scattering			
Bilby	SC-8	12 position sample changer	253-353K
Quokka	SC-2	20 position sample changer	263-393K
Kookaburra	SC-9	6 position rotating sample changer	253-353K
Reflectometers			
Platypus	SL-1	Solid liquid cells	263-363K
Spatz	SL-1	Solid liquid cells	263-363K
Imaging			
Dingo		Ambient temperature mount	

Please read on to discover more about our magnets, dilution refrigerators, furnaces, etc. This manual is arranged in sections by equipment type. If you would like to see the list of equipment for a specific neutron beam instrument please go to chapter 7.

Contents

Chapter 1

Introduction

This document is intended to give a brief overview of the sample environment capabilities available at the Australian Centre for Neutron Scattering (ACNS). In particular, beamline users can use this handbook as a guide to selecting the optimal sample environment apparatus to use for your experiment on the various neutron scattering instruments. The different sample environment setups are considered standalone units which are compatible with many instruments and may also be used in conjunction with each other.

Please be aware that the contents of this document will be updated periodically and that a newer version may be available. Also, note that it is not possible to account for every setup configuration here, so if your requirements differ from what is described then please contact the sample environment group (sample_environment@ansto.gov.au) for more information as in many cases modifications can be made to existing setups.

Most of our equipment is integrated with SICS. SICS (SINQ instrument control system) provides command line instrument control. The user interface on the instruments in most cases is Gumtree. The exceptions are Koala , Platypus and Sika have unique user interfaces. In combinations these packages provides remote control and data logging of sample environment parameters and collection of neutron data. Hence in most cases data from sample environment equipment will be provided in the same data file as the neutron data. If this is not possible we attempt to provide timing trigger points in the neutron data file so the sample environment files and neutron data can be synchronised.

1.1 Considerations when selecting ACNS equipment

To aid with choosing the best sample environment setup for your experiment, it is worth considering the following points

- What is the simplest setup that will satisfy your requirements?
- What are the potential safety considerations for the experiment? Any potential hazards must be addressed in a risk assessment before the experiment begins.
- What temperature range and stability do you require ?
- How much time is required for the sample environment equipment to be set up, ramped between different set points, or for a sample change to be performed?
- How will the sample be mounted?
  - Is the sample environment setup appropriate for the size of your sample?
  - Will the sample be loaded into a can or cell, or mounted onto a holder and if so does a new holder need to be made?
  - Are the materials used for mounting the sample appropriate for neutron scattering and the temperature range to be investigated?
- Will maintenance of the setup be required during the experiment, for example replenishing cryogenic liquids in wet cryostats?

It is best to discuss your sample environment choices and any potential hazards related to your experiment with your local contact for the neutron instrument that you are using, and the sample environment group should be consulted if further information is required.
To assist you the table below gives an indication of the time, in hours, required for various steps in the preparation of our sample enviroment equipment. Pre-beam time is the time required for SE to pump down, pre-cool or otherwise prepare prior to your beam time. In some cases this will mean your selected piece of equipment will be scheduled to start mid week. Setup time is that period required by SE on the first day of your scheduled beam time to install the selected piece of equipment. Sample environment normally have access to the instrument enclosures from approximately 9.00am on your first scheduled day. Installation of equipment for your experiment cannot begin till the previous piece of SE equipment has been removed. This period is not required if the same piece of equipment is used for sequential experiments. In general equipment that does require long set up times will be scheduled as blocks of time by the instrument scientists. Heat/Cool time is the period required to reach the extreme temperature achievable immediately after install for that piece of equipment. Normally time to temperature decreases after the ramp to set-point so this is the longest period that you may need to wait. Sample change is the period required to change from base or maximum temperature (cool down of furnaces warm up of cryostats). Note bottom loading cryostat have long sample change times however measurements can still be undertaken during warm up. The compressor has to be turned off during warm up prior to sample change as the whole cold head has to be close to room temperature before breaking vacuum. Sample changes in bottom-loaders require the use of the enclosure jib crane where available. In

contrast a sample change in a top-loading cryostat can be undertaken with only minimal temperature change to the cryostat. Also the compressor remains running significantly increasing the cool down rate after the sample change. It is best to only use bottom loaders if you only have a small number of samples. Removal time is the period to remove the sample environment and clear the enclosure ready for the next experiment. By checking the schedule you can determine time lost by adding the removal of the previous equipment to the installation of your selected equipment. Instrument scientists doing the scheduling try to keep this lost time in mind when organising the order of experiments.

Ancillary code	Pre-Beam Time	Install	Heat-Cool	Sample Change	Remove	Notes
Cryostats						
CF-1 & CF-3	0	1	3	2	1	Compressor is off during sample change
CF-2	0	1	3	2	1	Compressor is off during sample change
CF-5	0	1	2	2	1	Compressor is off during sample change
CF-6	0	1	2	2	1	Compressor is off during sample change
CF-7 & CF-8	0	1	2	0.25	0.5	
CF-10	0	2	4	0.25	2	
CF-11 & CF-12	0	3	8	0.25	2	
CF-13 & CF-14	0	2	9	0.25	2	
Cobra	0	1	1	0.1	1	
OC-1	24	1	3	0.25	1	12 hourly liquid cryogen refills
Dilution fridges						
DL-1	24	4	20	12	4	includes install/removal of CF-10 or AVM-1
OS-1	12	4	12	12	4	includes install/removal of OC-1
DC-1	48	5	12	24	5	
Furnaces						
F-1	0	1	2	2	1	Temp < 100 for sample change
F-4	0	0.5	1	1	0.5	
F-5	0.5	1	1	1	0.5	
F-6	0.5	1	1	1	0.5	
Magnetic						
HM-1	72	3	3	2	3	times for use with bottom loader
HM-2	72	2	2	0.25	2	12 hourly liquid cryogen refills
HM-3	0	1.5	2	2	1	times for use with CF-6
AVM-1	0	3	3	0.25	3	may require cryogen fills
AVM-2	24	3	3	0.25	3	may require cryogen fills
Electric						
EF-1	0	1	_	_	0.5	
PG-1	0	0.5	_	_	0.5	
BT-1	0	0.5	_	_	0.5	

Ancillary code	Pre-Beam Time	Install	Heat-Cool	Sample Change	Remove	Notes
Pressure						
GD-1	0	2	_	_	2	variable depends on set up
GS-1	0	2	_	_	2	
VD-1	12	2	_	_	2	
P-2	0	4	_	0.1	4	
P-3	0	2	_	1	2	
Rheology						
RH-1	0	3	1	0.25	2	
RV-1	0	2	0.5	0.25	2	
Solvent delivery						
SP-1	0	0.5	_	0.1	0.5	
QP-1	0	0.5	_	0.1	0.5	
SF-1	1	1	1	0.5	1	Check operation before install
Monolayer Prep						
LF-1 & LF-2	2	1	1	0.5	1	clean troughs immediately before use
Thermal Analysis						
DSC-1	6	2	1	0.25	2	run indium calibration before use
Light spectroscopy						
IR-1	0	2	1	0.25	2	
OF-1	0	1	_	_	1	
Sample Mounting						
EC-1	0	1	_	_	1	Used with CF-5
EC-3	0	1	_	_	1	RT only used on Kowari
SC-1	6	1	2	0.5	1	Pre time if changing hot/cold config
SC-2	0	0.5	3	0.5	0.5	
SC-3	2	1	2	0.5	1	clean troughs immediately before use
SC-4	0	1	_	0.25	1	calibrate positions during setup
SC-5 & SC-10	0	2	_	0.25	1	calibrate positions during setup
SC-7	0	0.5	_	0.25	0.5	
SC-8	0	1.5	2	0.25	1.5	
SC-9	0	2	2	0.25	1.5	

1.2 Considerations before shipping samples to ACNS

Firstly samples are considered hazardous until shown to be otherwise. If a Safety Data Sheet (SDS) is available use this to determine if your sample is non-hazardous. If an SDS is not available contact the ACNS Lab Manager. If the sample is non-hazardous it can be shipped however please include a copy of the SDS on the outside of the box so the contents can be checked before the box is opened.
If the samples are hazardous, contact the ACNS Lab Manager before shipping. There will be special packing requirements particularly if the samples have to be shipped by air freight.
We strongly advise against carrying samples with you. If the samples are non hazardous and you have to bring them in your luggage be sure you have paperwork to show security and customs officials if necessary. Without this paperwork there is a chance your sample will be confiscated by customs officials.

1.3 Considerations when bringing your own sample environment

If you're bringing your own sample environment equipment with you, please let us know so we can ensure that it is appropriate for use with the neutron instrument. Considerations include

- Can it be mounted on the sample stage for the desired instrument? For example, see Figs. 1.1 and 1.2.
- Are approvals needed (such as electrical, laser or pressure vessel) before use?
- Are any modifications or extensions to existing sample environment required to accommodate your equipment?

1.3.1 User-supplied pressure or vacuum vessels

If you intend to bring any equipment that is either a pressure or a vacuum vessel, please contact us with at least eight weeks notice to get the vessel approved by our pressure approvals officer. It is best to start the conversation about pressure approval when you submit your proposal. Be aware that approval may require redesign or modification of your equipment to meet ANSTO standards. Refer to Approval process for use of pressure vessels at the Australian Centre for Neutron Scattering.

1.3.2 User-supplied non-ionising radiation sources (UV, laser or welding equipment)

If you intend to bring any sources of non-ionising radiation, the Sample Environment group must be consulted at least 8 weeks in advance to ensure that safety approvals can be processed before your experiment begins. Safety approval may require interlocks to our safety interlock system. At ANSTO we follow guidelines set down by our regulator ARPANSA. Please visit their website for more information .

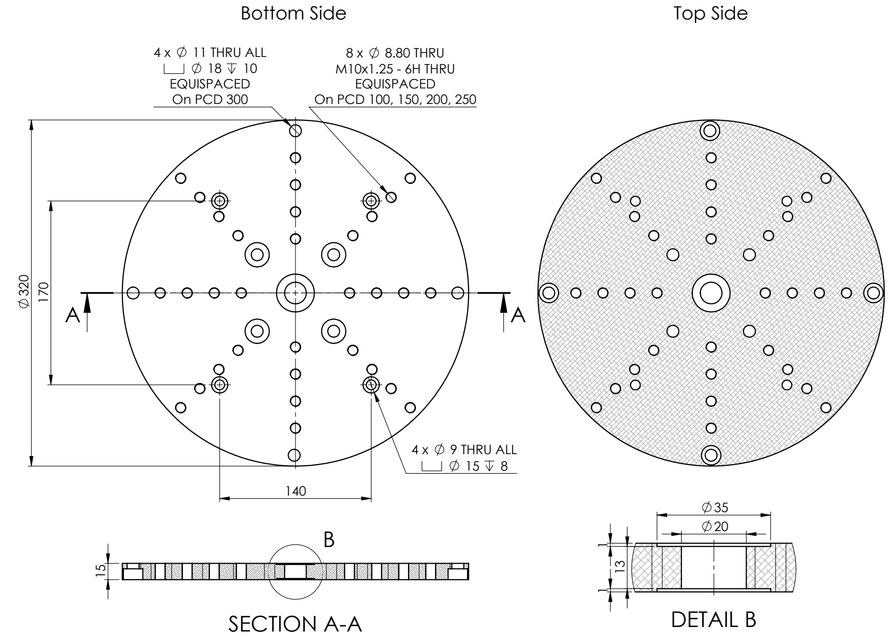


Figure 1.1: Schematic of the standard top plate for neutron instruments using a goniometer stage. Detail B shows the dimensions of the centering pin hole.

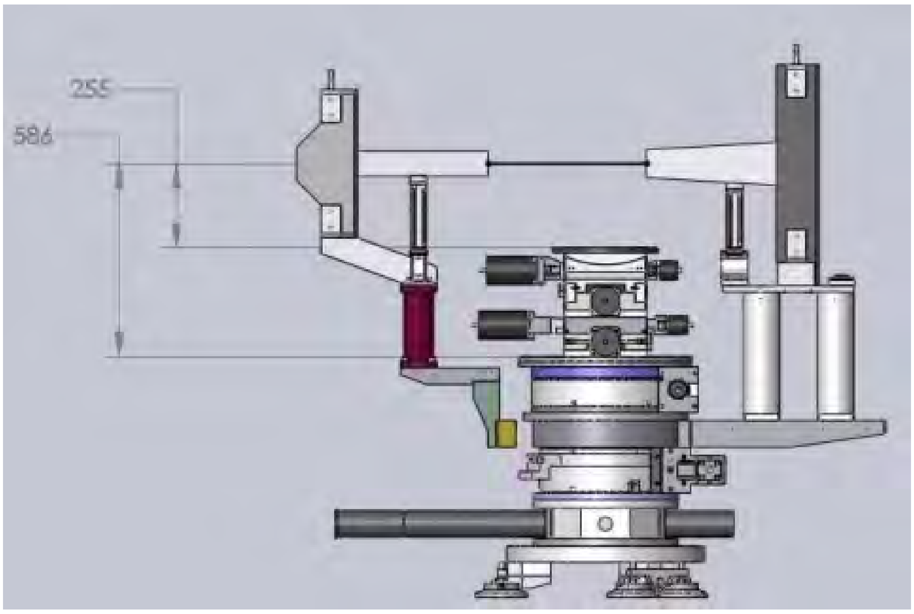


Figure 1.2: Schematic showing the neutron beam (dark horizontal line) height relative to the goniometer stages typically used on diffractometer and triple-axis spectrometer instruments. Other instruments incorporate vertical translation stages hence have variable beam height

1.3.3 Compatibility of ACNS SE equipment with neutron instruments

The following tables illustrate the existing compatibility of sample environment setups with the different neutron instruments. Note that it may be possible to extend the compatibility of some setups to more instruments than is shown here (if such a combination is technically feasible and enough time is given to complete commissioning tests), so please discuss such requests with the sample environment group. Recommended sample environment for a particular instrument is indicated by a green tick ✓a white tick ✓merely indicates mounting is possible. Please contact your local contact prior to booking. More information concerning neutron instruments is supplied in chapter 7. Detailed sample environment user instructions are supplied with the equipment where required. Extra instruction can always be obtained from our sample environment team.

1.3.4 Temperature

Ancillary code	Echidna	Wombat	Koala	Kowari	Sika	Taipan	Taipan: Be Filter	Emu	Pelican	Bilby	Quokka	Kookaburra	Platypus	Spatz	Dingo
Cryostats															
CF-1 & CF-3	✓	✓	_	✓	✓	✓	_	✓	✓	_	_	_	_	_	_
CF-2	_	_	✓	_	_	_	_	_	_	_	_	_	_	_	_
CF-5	✓	✓	_	_	✓	✓	_	_	_	_	_	_	_	_	✓
CF-6	_	_	_	_	_	_	_	_	_	✓	✓	_	✓	✓	_
CF-7 & CF-8	✓	✓	_	_	✓	✓	✓	_	_	_	_	_	_	_	_
CF-10	✓	✓	_	_	✓	✓	_	_	✓	_	_	_	_	_	_
CF-11 & CF-12	✓	✓	_	_	✓	✓	✓	✓	✓	_	_	_	_	_	_
CF-13 & CF-14	✓	✓	_	_	✓	✓	_	_	_	_	_	_	_	_	_
Cobra	_	_	✓	_	_	_	_	_	_	_	_	_	_	_	_
OC-1	✓	✓	_	_	✓	✓	_	_	_	_	_	_	_	_	_
Dilution fridges															
DL-1	✓	✓	_	_	✓	✓	_	_	✓	_	_	_	_	_	_
OS-1	✓	✓	_	_	✓	✓	_	_	_	✓	✓	_	_	_	_
DC-1	✓	✓	_	_	✓	✓	_	_	✓	_	_	_	_	_	_
Furnaces															
F-1	✓	✓	_	_	✓	✓	_	_	✓	_	_	_	_	_	✓
F-4	_	_	_	✓	_	_	_	_	_	_	_	_	_	_	_
F-5	✓	✓	_	_	✓	✓	_	_	_	_	_	_	_	_	_
F-6	✓	✓	_	✓	✓	✓	_	_	_	_	_	_	_	_	_

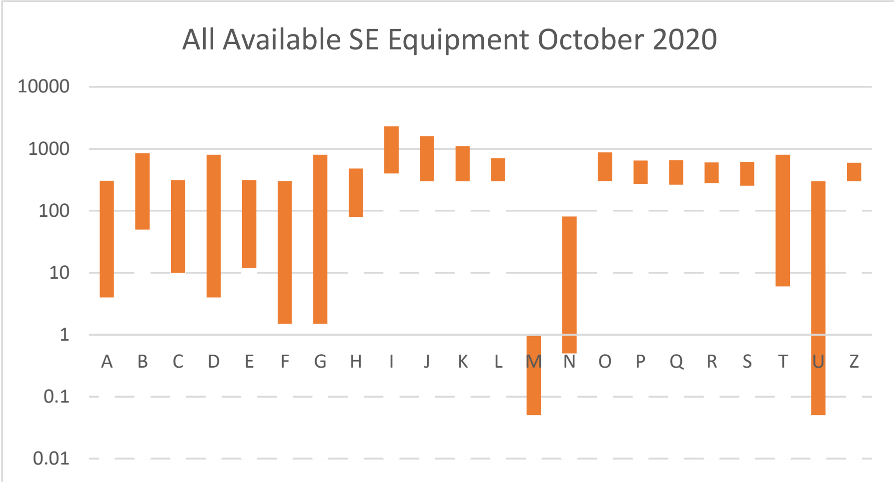
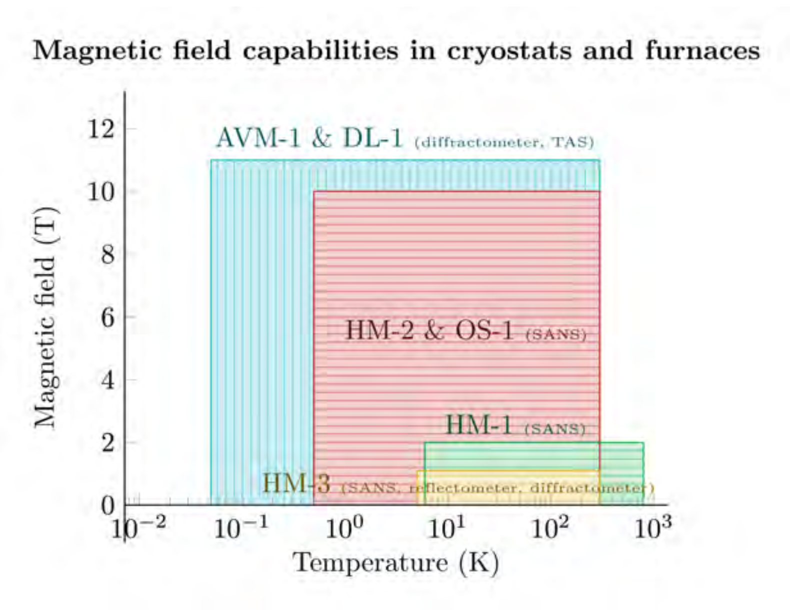


Figure 1.3: Temperature Ranges of all Equipment. Table below relates code letter to ancillary

Code									
A	CF1	CF2	CF3	CF6	CF7	CF8	HM3-CF6		
B	CF1-HT	CF2-HT	CF3-HT						
C	CF5	EC1-CF5							
D	CF7-HT	CF8-HT							
F	CF10	CF11	CF12	OC1	HM2	AVM1	AVM2	CF13	CF14
G	CF11-HT	CF12-HT	CF13-HT	CF14-HT					
H	Cobra								
I	F1	F1-HTS1							
J	F4								
K	F5								
L	F6								
M	CF10-DL1	CF11-DL1	CF12-DL1	AVM1-DL1					
N	OC1-OS1								
O	SC1-HT								
P	SC1								
Q	SC2	DEC1							
R	SC3								
S	SC8	SC9	SL1						
T	HM1-CF1								
U	DC1								
Z	EC1	EC3	SC4	SC5	SC6	SC7	SC10	HM3	HM1

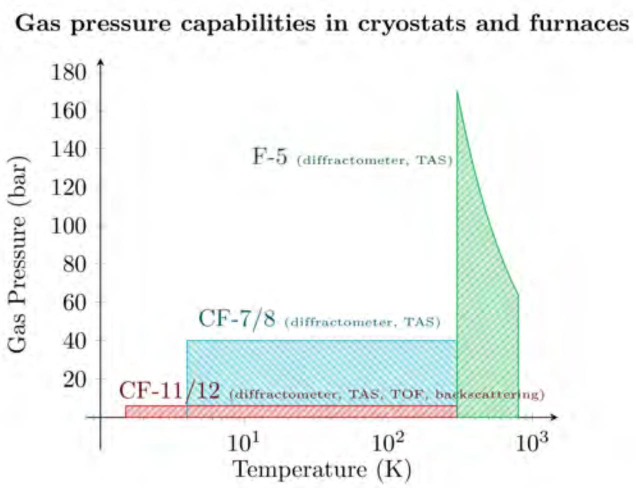
1.3.5 Magnetic and electric fields

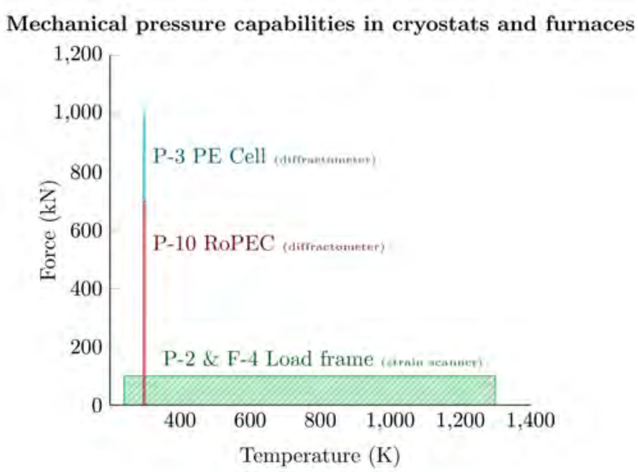
Ancillary code	Echidna	Wombat	Koala	Kowari	Sika	Taipan	Taipan: Be Filter	Emu	Pelican	Bilby	Quokka	Kookaburra	Platypus	Spatz	Dingo
Magnetic															
HM-1	–	–	–	–	–	–	–	–	–	✓	✓	✓	–	–	–
HM-2	–	–	–	–	–	–	–	–	–	✓	✓	–	–	–	–
HM-3	–	✓	–	–	–	–	–	–	–	✓	✓	–	✓	✓	–
AVM-1	✓	✓	–	–	✓	✓	–	–	–	–	–	–	–	–	–
AVM-2	✓	✓	–	–	✓	✓	–	–	✓	✓	✓	✓	–	–	–
Electric															
EF-1	✓	✓	✓	✓	✓	✓	–	✓	✓	✓	✓	✓	✓	✓	✓
PG-1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
BT-1	✓	✓	–	✓	✓	✓	–	–	–	✓	✓	–	✓	✓	✓



1.3.6 Pressure

Ancillary code	Echidna	Wombat	Koala	Kowari	Sika	Taipan	Taipan: Be Filter	Emu	Pelican	Bilby	Quokka	Kookaburra	Platypus	Spatz	Dingo
GD-1	✓	✓	–	–	✓	✓	✓	✓	✓	–	–	–	–	–	–
GS-1	✓	✓	–	–	✓	✓	✓	–	✓	–	–	–	–	–	–
VD-1	✓	✓	–	–	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
P-2	–	–	–	✓	–	–	–	–	–	–	–	–	–	–	–
P-3	✓	✓	–	–	✓	✓	✓	–	–	–	–	–	–	–	–





1.3.7 Techniques

Ancillary code	Echidna	Wombat	Koala	Kowari	Sika	Taipan	Taipan: Be Filter	Emu	Pelican	Bilby	Quokka	Kookaburra	Platypus	Spatz	Dingo
Rheology															
RH-1	–	✓	–	–	–	–	–	–	–	✓	✓	✓	–	–	–
RV-1	–	–	–	–	–	–	–	–	–	–	✓	–	–	–	✓
Solvent delivery															
SP-1	–	–	–	–	–	–	–	–	–	✓	✓	✓	✓	✓	–
QP-1	–	–	–	–	–	–	–	–	–	–	–	–	✓	✓	–
SF-1	–	–	–	–	–	–	–	–	–	✓	✓	–	–	–	–
Monolayer Prep															
LF-1 & LF-2	–	–	–	–	–	–	–	–	–	–	–	–	✓	–	–
Thermal Analysis															
DSC-1	–	–	–	–	–	–	–	–	–	–	✓	–	–	–	–
Light spectroscopy															
IR-1	–	–	–	–	–	–	–	–	–	–	–	–	–	✓	–
OF-1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

1.3.8 Sample changers and manipulators

Ancillary code	Echidna	Wombat	Koala	Kowari	Sika	Taipan	Taipan: Be Filter	Emu	Pelican	Bilby	Quokka	Kookaburra	Platypus	Spatz	Dingo
EC-1	✓	✓	–	–	✓	✓	–	–	–	–	–	–	–	–	–
EC-3	–	–	–	✓	–	–	–	–	–	–	–	–	–	–	–
SC-1	–	–	–	–	–	–	–	–	–	✓	✓	–	–	–	–
SC-2	–	–	–	–	–	–	–	–	–	✓	✓	–	–	–	–
SC-3	–	–	–	–	–	–	–	–	–	–	–	–	✓	–	–
SC-4	✓	✓	–	–	–	–	–	–	–	–	–	–	–	–	–
SC-5 & SC-10	✓	✓	–	✓	–	–	–	–	–	–	–	–	–	–	–
SC-6	–	–	–	–	–	–	–	–	–	–	–	✓	–	–	–
SC-7	–	–	–	–	–	–	–	–	–	✓	✓	–	–	–	–
SC-8	–	–	–	–	–	–	–	–	–	✓	✓	–	–	–	–
SC-9	–	–	–	–	–	–	–	–	–	–	–	✓	–	–	–

Chapter 2

Temperature

Temperature control equipment, such as thermal baths, cryostats, furnaces and cryofurnaces (which allow both cryogenic and furnace temperatures within the same setup) are listed below. Please note that some other types of sample environment equipment such as magnets (Chapter 3) and sample changers (§6.1) are also capable of independent temperature control.

Each cryostat or furnace is provided with a closed-loop temperature controller, typically a Lake Shore 336/340 or Oxford Instruments Mercury iTC, which interfaces with our SICS software to provide instrument control.

- Lake Shore 336 manual user instruction (PO-UI-073)
- Lake Shore 340 manual user instruction (PO-UI-040)
- Oxford Instruments Mercury iTC manual user instruction (PO-UI-141)

2.1 Thermal baths

We are also able to provide temperature baths such as Huber Unistat and Julabo FP40, FP45 and FP50 units, with either ethylene glycol or silicone oil. These baths are provided with sample environments requiring their use, such as temperature controlled sample changers, but can also be used independently. Please discuss your requirements with the Sample Environment group.

Figure 2.1: Photos of some temperature baths.

2.2 Cryostats and cryofurnaces

A variety of cryostats and cryofurnaces (cryostats with the ability to reach elevated temperatures) are available for use with most neutron instruments. The key characteristics of these are

- Temperature range
- How the cryostat/cryofurnace is mounted on the neutron instrument (for example, with a vacuum tail or inside a detector well)
- Whether samples are bottom-loading (fixed directly to a cold finger) or top-loading (mounted on sample probes which can be changed at temperatures below ambient)
- Whether a cryostat is closed-cycle (i.e. "dry" and without need for liquid cryogens) or "wet" (requiring consumption of liquid helium and/or nitrogen)
- Whether they are compatible with other ancillaries

2.2.1 Bottom-loading closed-cycle cryofurnaces CF-1 & CF-3

Ancillary codes	CF-1 & CF-3
Compatible instruments	Echidna, Kowari, Pelican, Sika, Taipan, Wombat, Emu
Temperature ranges	Cold configuration: 4 K to 300 K Hot configuration: ∼50 K to ∼800 K
Time to base temperature	∼2.5 hours
Thermometry	Cold stage: DT-670 silicon diode Hot stage: E-type thermocouple, platinum resistor PT100/1000
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can Bottom-loading cryostat exchange gas can
Complementary ancillaries	EF-1: 10 kV electric field system
Project folder	BIP-0264
User instruction	PO-UI-049: Janis Cryofurnace Changing User Instruction
SICS control	Lakeshore 340/336 (100W heater up to 2 temperature sensors)
Equipment Number	167210, 220172

These cryofurnaces mount samples on a stage directly attached to a cold head (typically a Sumitomo RDK-408D2) inside an evacuated chamber. They have aluminium vacuum tails and radiation shields, and can be mounted on goniometer stages to allow for up to 20° of tilt and 360° of rotation. The sample height in the neutron beam can be adjusted by adding spacers to the vacuum tail. Different temperature ranges are possible, depending

on whether the cold stage (copper link between the sample and cold head) or hot stage (stainless steel is used to thermally isolate the sample from the cold head) is installed. Please specify which stage is required for your experiment.
Because the diffraction instrument have no vertical translation the sample height is adjusted by using a range of adapters and spacers between the cryofurnace and the sample stage. To determine the required adapter use the following steps. This must be completed before assembling the vacuum tail.

- 1. fit sample to cryofurnace using M6 mounting thread
- 2. measure from the **underside** of the cryofurnace sealing flange to the centre of the sample
- 3. transfer this measurement and mark it onto the vacuum tail. Measuring from the **top** of the sealing flange on the tail down toward the base. This will be the sample position when the cryofurnace is assembled.
- 4. measure from the base of the vacuum tail up to the mark from step 3.
- 5. subtract the measurement from step 4 from 255mm and the result is the required height of the adaptor.

Note: Sample changes take several hours as they require the cryofurnace to first be brought to room temperature.

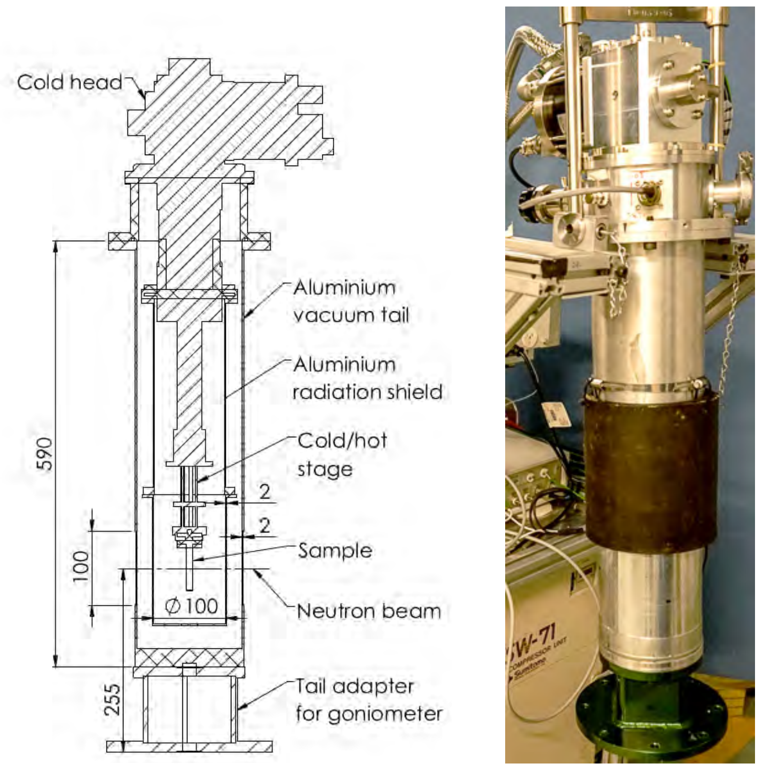


Figure 2.2: Schematic (left) and photo (right) of CF-1/3.
Note: use steps above to determine required adaptor height

2.2.2 Bottom-loading closed-cycle cryofurnace CF-2

Ancillary code	CF-2
Compatible instrument	Koala
Temperature ranges	Cold configuration: 4 K to 300 K Hot configuration: $\sim$50 K to $\sim$800 K
Time to base temperature	$\sim$2.5 hours
Thermometry	Cold stage: DT-670 silicon diode Hot stage: E-type thermocouple
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can
Project folder	BIP-0174
User instruction	PO-UI-049: Janis Cryofurnace Changing User Instruction
SICS control	Lakeshore 340 (100W heater up to 2 temperature sensors)
Equipment Number	167360

This cryofurnace is equivalent to CF-1 and CF-3, however it has a different vacuum tail which is unique to fit the rotation stage on Koala.

Note: Sample changes take several hours as they require the cryofurnace to first be brought to room temperature.

Figure 2.3: Photo of CF-2.

2.2.3 Bottom-loading closed-cycle cryostat CF-5

Ancillary code	CF-5
Compatible instruments	Echidna, Sika, Taipan, Wombat
Temperature range	10 K to 300 K
Thermometry	DT-670 silicon diode
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can
Complementary ancillary	EC-1: Eulerian cradle
SICS control	Lakeshore 340 (100W heater 1 x temperature sensor)
Equipment Number	220173

This cryostat has a narrow aluminium vacuum tail which can be mounted on the Eulerian cradle EC-1. A short cylindrical tail is available for this cryostat. It has been used on both Dingo and Kowari. Where a full-sized cryostat was not appropriate.

The vacuum tail mounts by sliding over two captive o-rings. Ensure the sealing surface is clean and thin layer of vacuum grease applied before assembly. The mounting incorporates a x-y translation stage to allow the sample to be aligned when used in EC-1. Z adjustment is via the four studs supporting the cold head. When mounting in EC-1prior to assembly mark the position of the groove in brass locating ring on aluminium collar. This allows the groove to be aligned with the pin in small rotation stage on EC-1. If this groove is misaligned damage can occur to the cryostat mount that will need to be repaired before attempting to mount it a second time.

Note: Sample changes take several hours as they require the cryofurnace to first be brought to room temperature.

Figure 2.4: Photo of CF-5.

2.2.4 Bottom-loading closed-cycle cryostat CF-6

Ancillary code	CF-6
Compatible instruments	Quokka, Platypus, Bilby
Temperature range	4 K to 300 K
Time to base temperature	∼2 hours
Thermometry	DT-670 silicon diode
Mount required for sample	Flat horizontal plate or M6 male threaded post
Sample containers	none required for reflectometry SANS cell holder Vanadium can
Project folder	BIP-0071
Complementary ancillaries	EF-1: electric field system HM-3: 1 T horizontal magnet
SICS control	Lakeshore 340 (100W heater 2 temperature sensors)
Equipment Number	220174

This cryostat has two aluminium vacuum tails compatible with the 1 T horizontal magnet HM-3. The first tail is diamond shaped an fits between the poles of HM-3. It is used on Platypus within the magnet and on the scattering instruments without the magnet. On the scattering instruments the tails is rotated 90 degrees so the neutron beam travels the shortest path through the tail. The second tail is cylindrical and also fits within the HM-3. This tails is used on the scattering instruments where the magnet is mounted so the magnetic field is vertical and the cryostat is horizontal. A bracket and special set of studs are required to mount the cryostat. This configuration should only be used if HM-1and CF-1 & CF-3or HM-2is are not available. The cryostat mount includes a manual rotation stage for sample alignment.

CF-6It also has high-voltage feedthroughs which are compatible with the electric field system EF-1.

Note: Sample changes take several hours as they require the cryofurnace to first be brought to room temperature.

Figure 2.5: Photo of CF-6 (left) and the vacuum tail used for HM-3 (right).

2.2.5 Top-loading closed-cycle cryofurnaces CF-7 & CF-8

Ancillary codes	CF-7 & CF-8
Compatible instruments	Echidna, Sika, Taipan, Taipan: Be Filter, Wombat
Temperature ranges	Cold probe: 4 K to 300 K Hot probe: 4 K to ∼800 K
Time to base temperature	< 1 hour for sample change
Compatible sample probes	Cold probe with DT-670 silicon diode TL-1 & TL-3: full range probe with Rhodium iron sensor TL-5 & TL-9: gas/vapour delivery sample probes
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can
Complementary ancillaries	GD-1: gas sorption/desorption system VD-1: vapour delivery system
Project folder	BIP-0097, BIP-0254, BIP-0258
User instruction	PO-UI-072 Top Loading Cryofurnace Sample Changing User Instruction
SICS control	2 x Lakeshore 340/336 control VTI and sample temperature (sample probe up to 2 heaters and 2 temperature sensors)
Equipment Number	168749, 171010

These cryofurnaces have aluminium vacuum tails and radiation shields. They are mounted on goniometer stages to allow for up to 20° of tilt and 360° of rotation. Attocube miniature piezo sample goniometer stages can also be used (please contact SE group for more information). Sample probes have ∼25 mm of height adjustment, while spacers for the VTI are available to achieve further height changes. When below room temperature, the sample can be mounted on a cold probe and exchange gas is added to the sample space to achieve cryogenic temperatures. For elevated temperatures, full range probes TL-1 & TL-3 is used in vacuum. Gas/vapour delivery probes TL-5 & TL-9 can also be used with these cryofurnaces. Modified probes for *in situ* optical spectroscopy and use power supply or potentiostat are available upon request.

Note: CF-7 typically used on Echidna, and CF-8 typically used on Wombat.

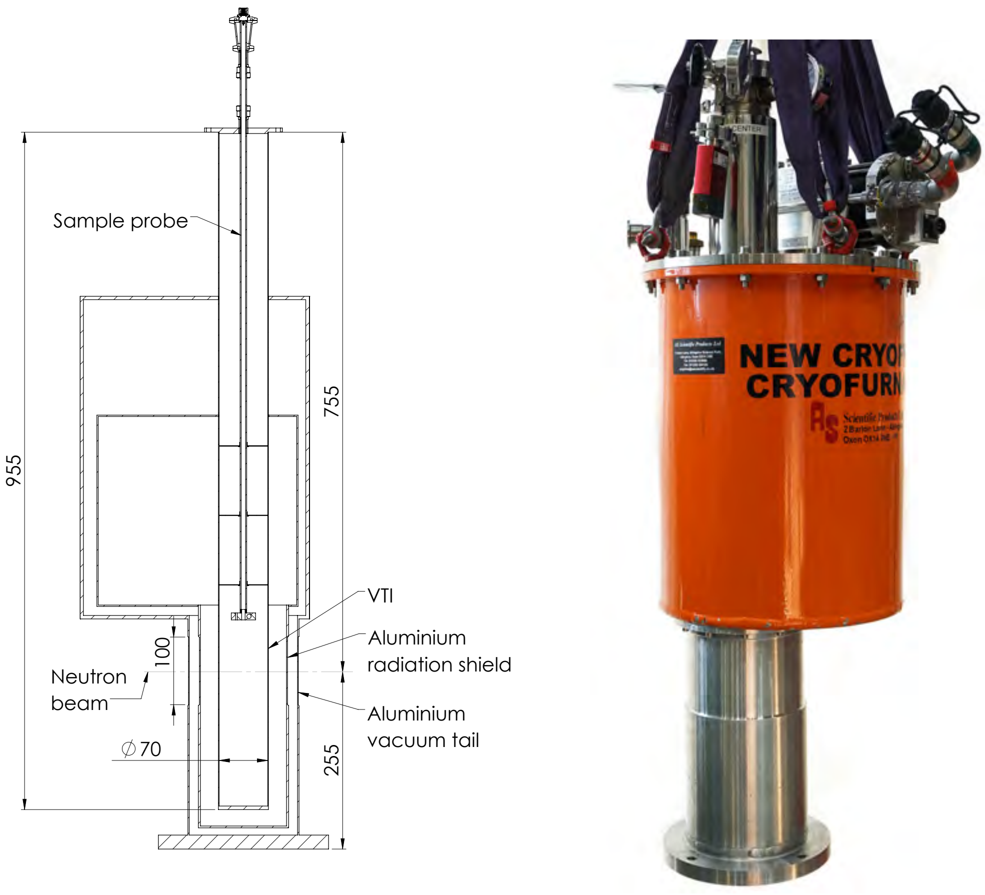


Figure 2.6: Schematic (left) and photo (right) of CF-7/8.

2.2.6 Top-loading closed-cycle cryostat CF-10

Ancillary code	CF-10
Compatible instrument	Echidna, Pelican, Sika, Taipan, Wombat
Temperature range	1.5 K to 300 K
Compatible sample probe	Cold probe with Cernox
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can (small flange only)
Complementary ancillary *SICS control*	DL-1: dilution refrigerator 2 x Mercury control VTI, sample temperature and needle valve (sample probe 1 heater, 2 temperature sensors)
Equipment Number	220175

This cryostat has an aluminium vacuum tails and radiation shields, and is fitted with a rotation stage to allow 360°of motion. The bore for the sample probe is ϕ 50 mm, while the sample space is ϕ 36 mm. This cryostat can be used with polarisation coils. Sample probes have $\sim$ 25 mm of height adjustment. Where this is insufficient a range of spacers available that raise the sample probe relative to the VTI flange. To mount this cryostat requires the removal of the tilt and translation states from the neutron instrument. Hence single crystal samples must be well aligned before mounting (book time on Joeyif required). Once mounted only sample rotation is available via the stage mounted on the VTI.

This cryostat is designed to accept the dilution insert DL-1. Please note when installed the rotation of the sample is limited due to the cabling and design of the dilution stick. Discuss your needs with SE prior to arrival and ensure your sample is well aligned if it is a single crystal.

Note: When used with DL-1, the narrow tail with sample space ϕ23.6 mm ID must be used.

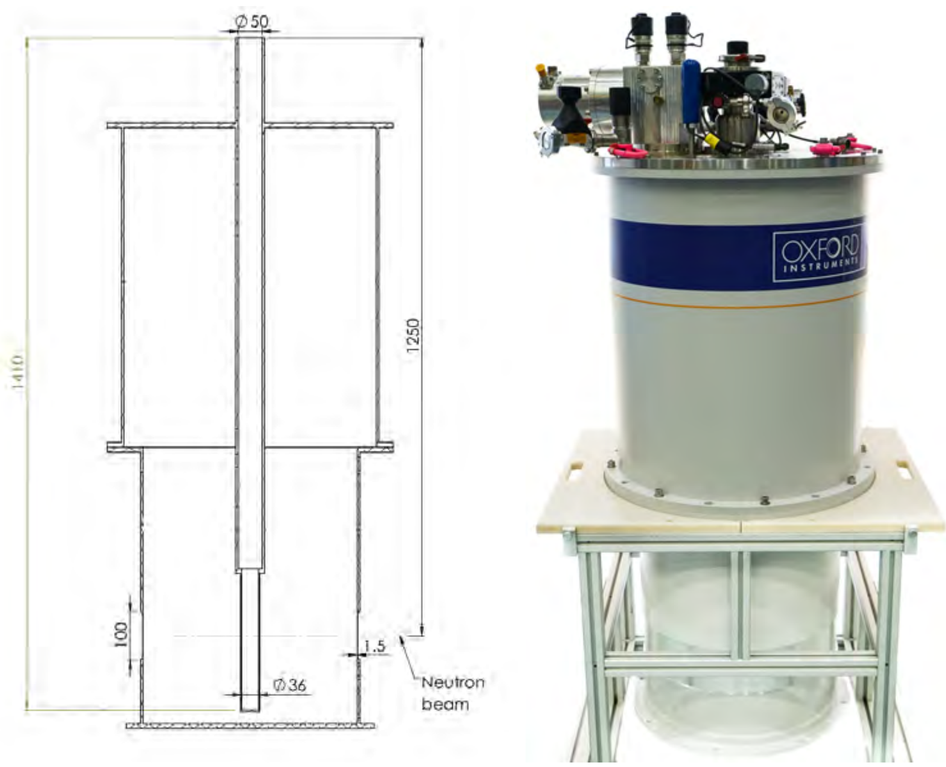


Figure 2.7: Schematic (left) and photo (right) of CF-10.

2.2.7 Top-loading closed-cycle cryofurnaces CF-11 & CF-12

Ancillary codes	CF-11 & CF-12
Compatible instrument	Echidna, Emu, Pelican, Sika, Taipan, Wombat
Temperature ranges	Cold probe: 1.5 K to 300 K Hot probe: 1.5 K to $\sim$800 K
Compatible sample probes	Cold probe with Cernox Hot probe with Rhodium iron sensor Gas/vapour delivery probe with DT-670 silicon diode
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can 100 bar stainless steel furnace can
Complementary ancillaries	DL-1: dilution refrigerator GD-1: gas sorption/desorption system VD-1: vapour delivery system
User instruction	PO-UI-146: Usage of Top Loading Cryostats CF-11 and CF-12
SICS control	2 x Mercury control VTI and sample temperature (sample probe 1 heater, 2 temperature sensors + needle valve)
Equipment Number	220176, 220177

These cryofurnaces have aluminium vacuum tails and radiation shields. They are mounted on goniometer stages to allow for up to 10° of tilt (at reduced cooling efficiency), and have 360° rotation stages for the sample probes. Sample probes have $\sim$25 mm of height adjustment. Where this is insufficient a range of spacers available that raise the sample probe relative to the VTI flange. The sample probes are hollow line of sight design allowing the centre of the stick to be used for complimentary techniques. For instance an optical can be introduced through the sample stick to illuminate the sample *in situ*. A specialised annual can is used with a conical reflector in the base and a quartz inner cylinder. This fits into a standard aluminium annular outer can.

Note: CF-11 typically used on Pelican, and CF-12 typically used on Emu, where the standard vacuum tail is removed to mount the cryofurnace in the detector well of these instruments. When used with DL-1, the wider radiation tail with a ϕ44 mm ID sample space can be used.

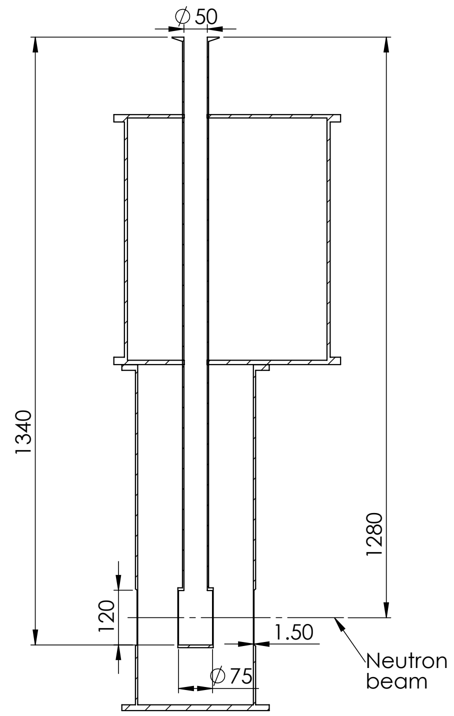


Figure 2.8: Schematic of CF-11.

Figure 2.9: Photo of CF-12 (left) and its gas delivery probe (right).

2.2.8 Top-loading closed-cycle cryofurnaces CF-13 & CF-14

Ancillary codes	CF-13 & CF-14
Compatible instrument	Echidna, Sika, Taipan, Wombat
Temperature ranges	Cold probe: 1.5 K to 300 K Hot probe: 1.5 K to ∼800 K
Compatible sample probes	Cold probe with Cernox Hot probe with Rhodium iron sensor
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can 100 bar stainless steel furnace can
Project folder	BIP-0350
Complementary ancillaries	DL-1: dilution refrigerator GD-1: gas sorption/desorption system VD-1: vapour delivery system
User instruction	PO-UI-146: Usage of Top Loading Cryostats CF-11 and CF-12
SICS control	Lakeshore 336 being commissioned VTI, Sample, Needle valve (sample probe 1 heater, 2 temperature sensors + needle valve)
Equipment Number	220178, 220179

These cryofurnaces have aluminium vacuum tails and radiation shields. They are mounted on goniometer stages to allow for up to 10° of tilt (at reduced cooling efficiency), and have 360° rotation stages for the sample probes. Sample probes have ∼25 mm of height adjustment. Where this is insufficient a range of spacers available that raise the sample probe relative to the VTI flange.

Note: These cryostats are currently being commissioned .

Figure 2.10: Photo of CF-13 in SE Area.

2.2.9 Cryostream Cobra

Ancillary code	Cobra
Compatible instrument	Koala
Temperature range	80 K to 400 K
Mount required for sample	M6 male threaded post
Project folder	BIP-0096
User instruction	PO-UI-005: Usage of COBRA on KOALA
Manufacturer website	Oxford Cryosystems
SICS control	Cryosystems controller 1 heater 1 temperature sensor
Equipment Number	220180

The Oxford Cryosystems Cobra is a non-liquid nitrogen cryostream which uses a cold head to cool nitrogen flowing over a small sample to temperatures as low as 80 K. The nitrogen flow is arranged as two coaxial laminar flows the inner flow is temperature controlled and cools the sample while the outer flow is at room temperature and prevents frost forming on the sample.Samples or structures larger than the inner flow diameter (about 8mm) cause turbulence and frost will form as surrounding air is mixed with the cooled nitrogen gas. Hence a heater on the mounting plate prevents condensation or ice from forming on the supports. Nitrogen gas can also be heated to 400 K. This system has the advantages of minimising the amount of unwanted material in the neutron beam (such as the vacuum tail and radiation shields found on most cryostats) and allows samples to be changed very quickly.

The Cobra system is used on Koala with a mounting plate incorporating a bearing to allow rotation of the sample, while the cryostream nozzle remains fixed. Samples are fixed to a small aluminium pin or enclosed in a 4mm diameter quartz tube. A continuous source of dry nitrogen (approx 99.5%) is derived from air using a Parker Nitroflow nitrogen generator. The system can also be run from the nitrogen supply on the services panel.

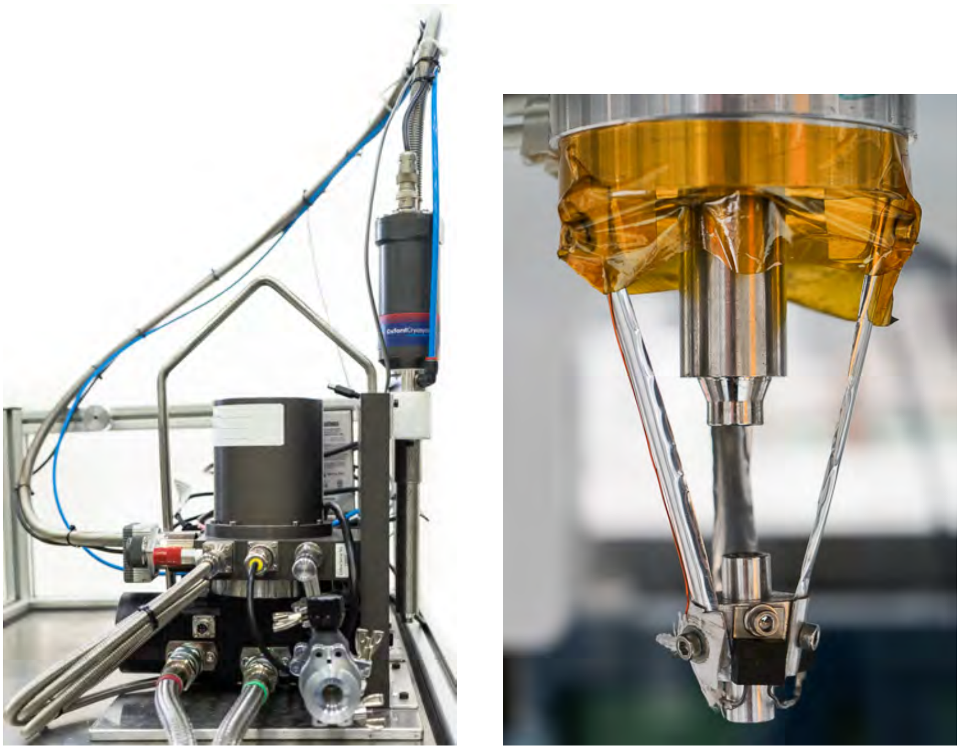


Figure 2.11: Photo of the Cobra cryostream (left), and the sample mounting position under the cryostream nozzle (right).

2.2.10 Orange cryostat OC-1

Ancillary code	OC-1
Compatible instrument	Echidna, Sika, Taipan, Wombat
Temperature range	1.5 K to 300 K
Compatible sample probe	Cold probe with Cernox
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can Aluminium annular can
Complementary ancillary	OS-1: one-shot ^{3}He refrigerator
Project folder	BIP-0004, BIP-0112
User instruction	PO-I-028: Orange Cryostat Operating Instructions
SICS control	LakeShore 340/336 and needle valve (sample probe 1 heater and 2 temperature sensors)
Equipment Number	172423

This ILL-designed orange cryostat is cooled using liquid helium. It can be mounted on a goniometer stage allowing 20° of tilt and 360° of rotation. It is compatible with the one-shot ^{3}He refrigerator OS-1. The height from the sample position to the base of the vacuum tail is shorter than the 255mm required and hence an adapter stand and long threaded rods are used to mount OC-1on the sample stage.

Note: This cryostat must be refilled with liquid nitrogen and liquid helium periodically during use, which must be done by personnel who have completed an ACNS induction in the handling of cryogenic liquids. Approximately 24 hours are required to prepare the cryostat before use.

Figure 2.12: Photo of OC-1.

2.2.11 Triton dilution refrigerator/cryostat DC-1

Ancillary code	DC-1
Compatible instrument	Echidna, Wombat, Taipan, Sika, Pelican, Emu
Temperature range	50 mK to 300 K
Thermometry	Cernox NTC sensors Ruthenium oxide NTC sensors
Mount required for sample	TBC Oxford Puck
Sample containers	OFHC cans
Project folder	BIP-0350
SICS control	Oxford Labveiw VI (connect to cryostat control laptop)
Equipment Number	220181

Triton dilution cryostat manufactured by Oxford Instruments. Samples may be bottom or top loaded. Bottom loading is useful if only one sample is to be used during the experiment. The sample is loaded X days prior to beam time and cooling may commence either offline or once installed. Top loading allows sample changes during an experiment. However the top loading mechanism is not suited for safe operation on some instruments. Discuss your experiment with sample environment well before your beam time. Commissioning is continuing on this new piece of equipment. Powder samples should be loaded under a helium atmosphere and or a suitable deuterated solvent added (eg. d-propanol) to optimise thermal conductivity between the can and sample.

Note: Extra time is required for sample preparation and sample changes when using the dilution cryostat. There is a top loading option available for this cryostat when mounted on some neutron instruments. Please discuss your experimental requirements with the Sample Environment group before booking this piece of equipment. Initial samples should be mounted offline, ideally in advance so as not to reduce your beam time.

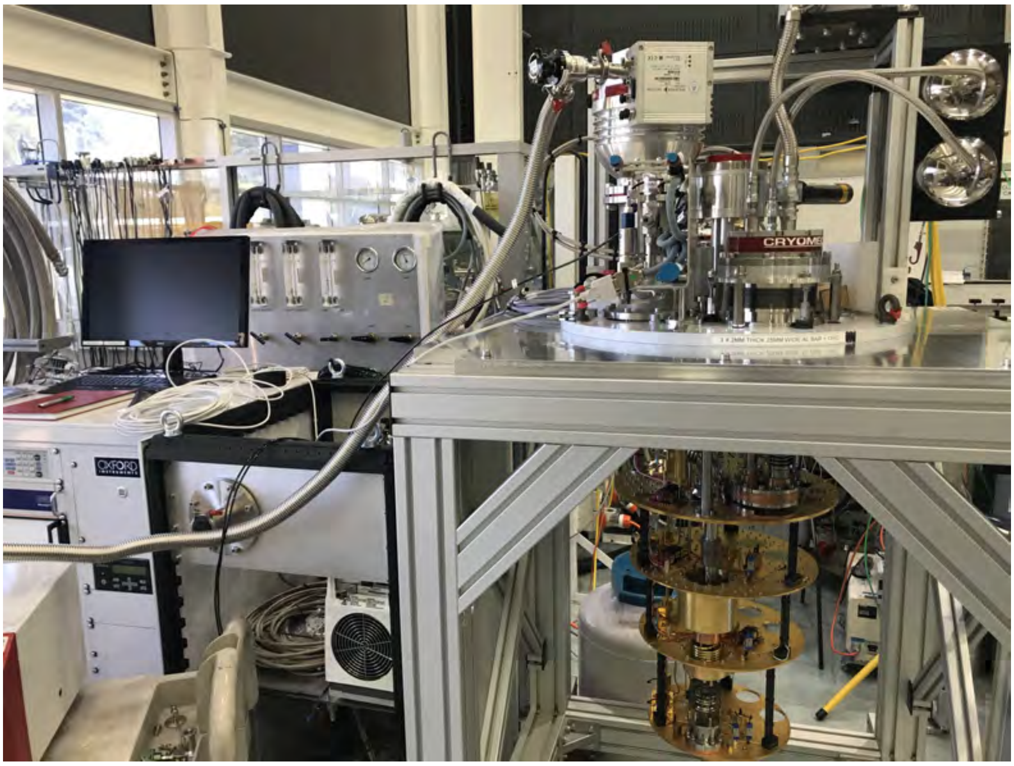


Figure 2.13: Photo of DC-1 in SE area.

2.3 Dilution inserts

Low-temperature inserts are used to extend the temperature range of cryocoolers and magnets to below 1.5 K. These inserts must be used in conjunction with a cryostat or magnet, and they also act as sample positioning probes.
Dilution cooling relies on the phase separation that occurs in a mixture of He^3 and He^4 at low temperatures to provide sample temperatures below 0.1K. Careful sample preparation is required to attain these low temperatures and discussion with sample environment prior to your experiment will be beneficial.
The preparation of the dilution fridge and sample takes several hours and generally the sample will be required by the sample environment team the day before the experiment. Cooling of the main cryostat has to be taken into account and generally a minimum of 2 days beam time should be allowed for to ensure sufficient time is available to collect data at base temperature. The table in the introduction gives an approximate set up time for dilution experiment with our different dilution options.

2.3.1 Kelvinox dilution refrigerator insert DL-1

Ancillary code	DL-1
Compatible instrument	Echidna, Emu, Pelican, Sika, Taipan, Wombat
Temperature range	50 mK to 900 mK
Thermometry	Cernox NTC sensors Ruthenium oxide NTC sensors
Mount required for sample	M6 male threaded post
Sample containers	OHFC cans
Must be booked with one of	AVM-1: 12 T vertical magnet CF-10: top loading cryostat CF-11 & CF-12: top loading cryofurnaces
User instruction	I-4449: Use of Oxford Instruments Kelvinox Dilution Insert PO-UI-126: Sample Can Preparation for the Dilution Insert
SICS control	Oxford Labveiw VI (connect to cryostat control laptop)
Equipment Number	220182

Kelvinox dilution insert manufactured by Oxford Instruments. Specialised oxygen-free copper sample cans including annular cans are provided upon request, or the sample may be attached to an OFHC pin using cryogenic-compatible hydrogen-free epoxy. Powder samples should be loaded under a helium atmosphere to optimise thermal conductivity between the refrigerator and sample. Temperatures above 1.5 K can be achieved by adding exchange gas to the insert and controlling the temperature with the VTI of the cryostat. Preparation of the insert takes several hours prior to cooling and preferably overnight evacuation. It is strongly recommended that the sample be made available to sample environment no later than the morning of the day before the start of the experiment beam time. This will ensure the minimum amount of beam time is lost. If the sample requires measurement beyond the upper stable temperatures for the insert discuss the order of measurements with sample environment to maximise your measurement time. Powder samples should be loaded under a helium atmosphere and or a suitable deuterated solvent added (eg d-propanol) to optimise thermal conductivity between the can and sample.

When using AVM-1 and CF-10, a narrow bore (ϕ23.6 mm ID) tail is used, while for CF-11 & CF-12 a larger (ϕ44 mm ID) tail can be fitted.

Note: Extra time is required for sample preparation and sample changes when using the dilution insert. Please discuss your experimental requirements with the Sample Environment group before booking this piece of equipment. Samples are mounted offline, ideally in advance. Several hours are required to change a sample, and ∼24 hours for the sample to reach dilution temperatures.

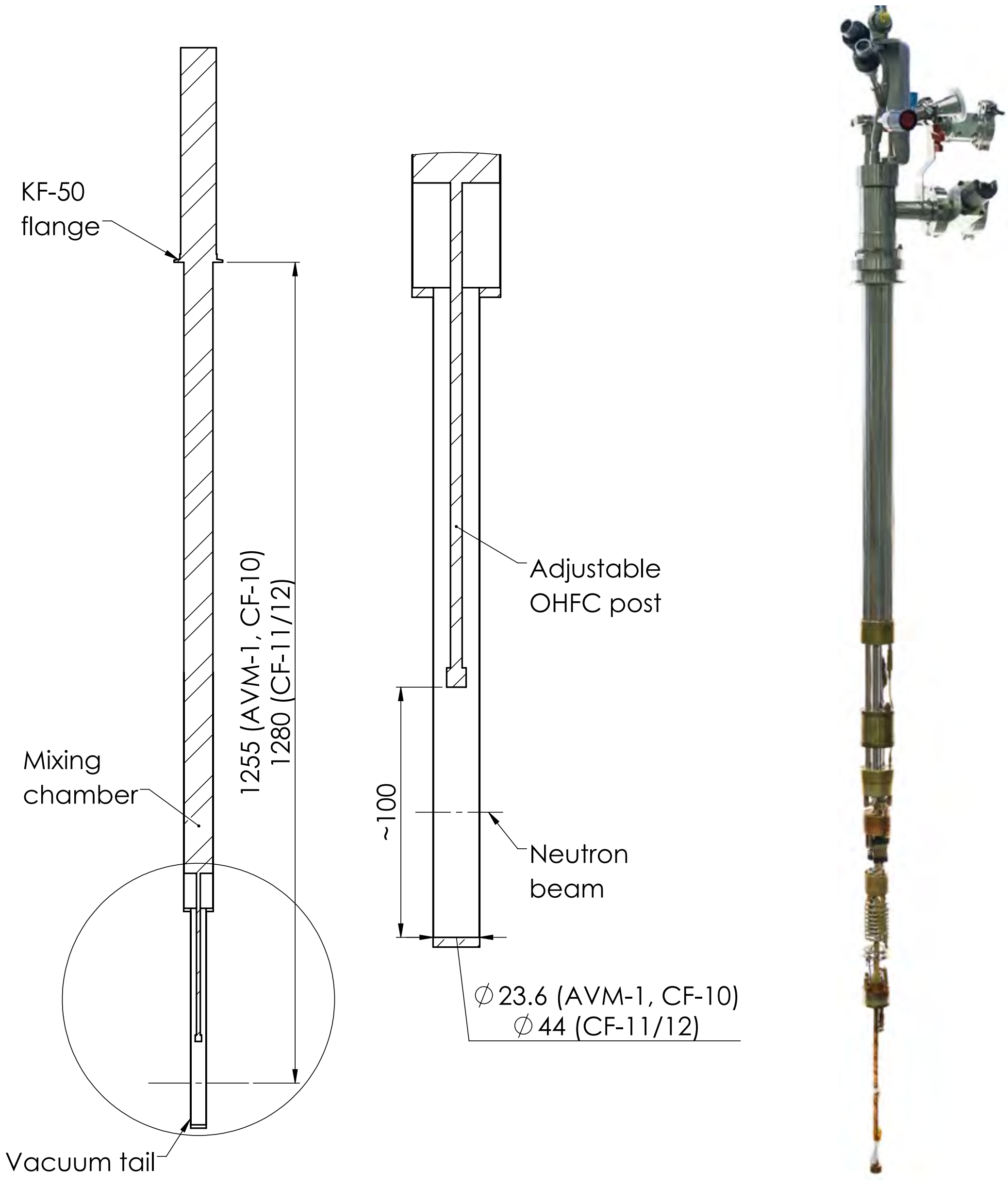


Figure 2.14: Simplified schematic (left) and photo (right) of DL-1.

2.3.2 He dilution one-shot insert OS-1

Ancillary code	OS-1
Compatible instrument	Bilby, Echidna, Quokka, Sika, Taipan, Wombat
Temperature range	0.5 K to 80 K
Thermometry	Cernox NTC sensors
Mount required for sample	M6 male threaded post
Sample containers	OHFC cans
Must be booked with one of	OC-1: orange cryostat HM-2: 10 T horizontal magnet
Project folder	BIP-0044
User instruction	I-4428 (PO-UI-127) ^{3}He One-Shot in Orange Cryostat User Instruction PO-UI-062: ^{3}Helium Fridge User Instruction
Manufacturer website	Oxford Instruments HelioxVT
Manufacturer instruction manual	Oxford Instruments HelioxVT operators handbook
SICS control	Oxford Labveiw VI (connect to cryostat control laptop)
Equipment Number	171472

Samples can be cooled to 0.5 K for up to 24 hours, and then a 30 minute regeneration is required. The sample space has an ID of 25 mm. Note that the one-shot refrigerator OS-1 is mounted within the orange cryostat OC-1with Echidna, Sika, Taipan and Wombat, while it mounted within the horizontal magnet HM-2 with Bilby and Quokka. It recommended to make the sample available to sample enviroment no later than the morning of the day before the start of the allocated beam time.

Powder samples should be loaded under a helium atmosphere and or a suitable deuterated solvent added (eg d-propanol) to optimise thermal conductivity between the can and sample.

Figure 2.15: Photo of OS-1.

2.4 Furnaces

A variety of furnaces are available to heat samples above room temperature under vacuum, atmosphere or with *in situ* gas delivery. Please note that devices which can reach both cryogenic and above ambient temperatures are listed in §2.2 as cryofurnaces.

2.4.1 High temperature vacuum furnace F-1

Ancillary code	F-1
Compatible instrument	Echidna, Pelican, Sika, Taipan, Wombat
Temperature range	300 K to 1900 K
Compatible sample probe	Vacuum probe with C-type thermocouple Gas flow probe with K-type thermocouple
Mount required for sample	M6 male threaded post
Sample containers	Vanadium can
Complementary ancillary	HTS-1: furnace gas delivery probes
Project folder	BIP-0002, BIP-0196, BIP-0278, BIP-0306
User instruction	PO-UI-137: 1600C Niobium Vacuum Furnace User Instruction
SICS control	1 heater Eurotherm controller
Equipment Number	167186

The 1600°C ILL-type high temperature vacuum furnace is heated by passing up to 300 A through a thin niobium element. The outer furnace body is made of aluminium and there eight layers of thin niobium radiation shields. The samples a introduced into the furnace via a standard KF40 vacuum fitting. and a variety of sample probes have been developed for sample to be measured in vacuum, in various atmospheres and a sample rotation stage is available.

Note: A second identical furnace F-7 is kept as a spare, and cannot be booked separately to F-1. The furnace requires several hours to cool from its maximum temperature to allow sample changes. The elements are attacked by oxygen above 100 degrees C and forms nitrides above 300C both reactions will shorten the life of the element by making it brittle it.

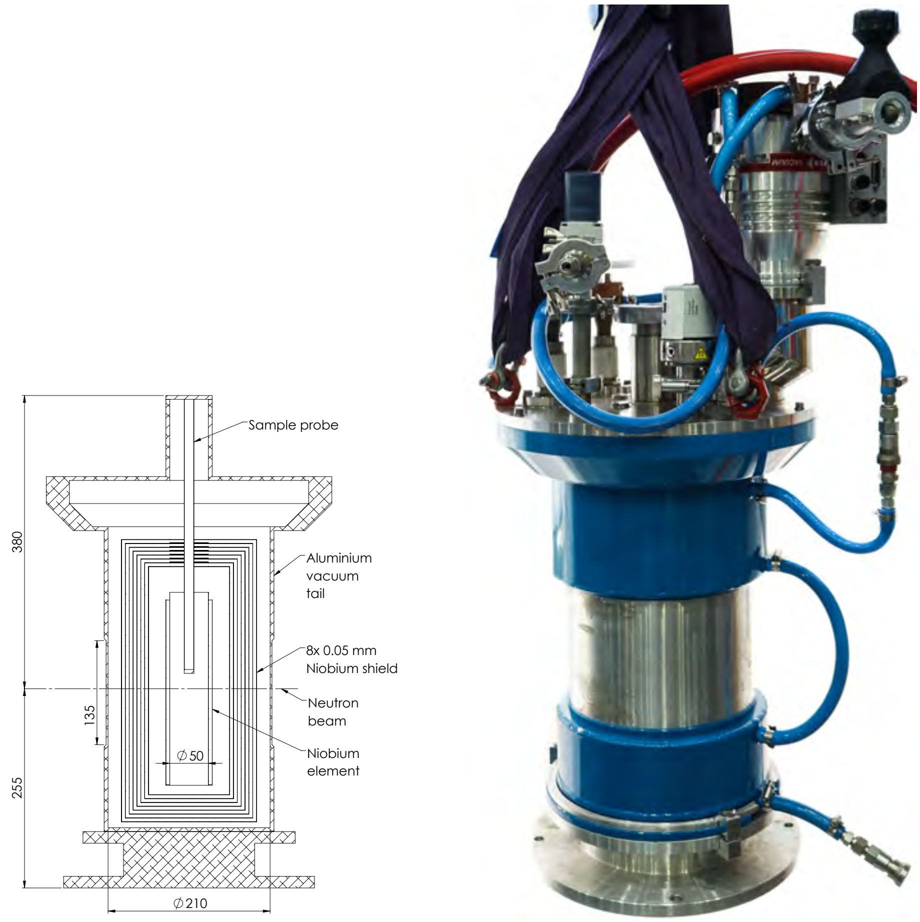


Figure 2.16: Schematic(left) and photo (right) of F-1.

2.4.2 Load frame furnace F-4

Ancillary code	F-4
Compatible instrument	Kowari
Temperature range	300 K to 1300 K
Mount required for sample	Load frame grips
Complementary ancillary	P-2: 100 kN load frame
SICS control	Proprietary heater controller, water cooled Halogen lamp
Equipment Number	220183

Halogen lamp furnace for use with the Kowari load frame P-2. The halogen lamps themselves are water cooled. Sample are mounted as normal in the load frame water cooled platens are required to protect the load cell from damage if the furnace is run at high temperatures.

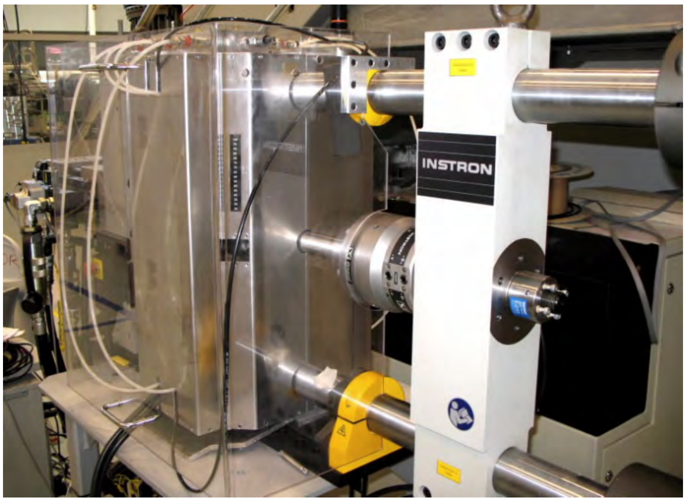


Figure 2.17: Photo of F-4 mounted on 100kN load frame P-2.

2.4.3 Low temperature vacuum furnace F-5

Ancillary code	F-5
Compatible instrument	Echidna, Sika, Taipan, Wombat
Temperature range	300 K to 800 K
Mount required for sample	M6 male threaded post
Inbuilt thermometry	K-type thermocouple
Sample containers	Vanadium can 100 bar stainless steel furnace can
Complementary ancillary	GD-1: gas delivery system
Project folder	BIP-0160, BIP-0260
User instruction	PO-UI-102: Low Temperature Vacuum Furnace User Instruction
SICS control	Watlow Controller 1 heater
Equipment Number	220184

This furnace is heated with a set of halogen lamps contained in an aluminium vacuum vessel, using a mirror to reflect radiation back to the sample position. Standard vanadium cans may be used. The furnace can be modified so gas dosing sample cans can be pressurised with gases (including hydrogen and deuterium) at elevated temperatures using a 100 bar stainless steel furnace can.

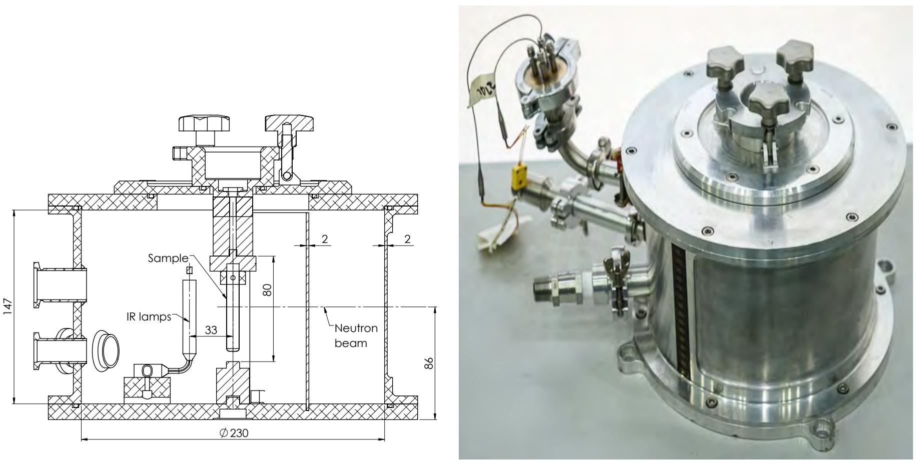


Figure 2.18: Schematic (left) and photo (right) of F-5.

2.4.4 Environmental chamber F-6

Ancillary code	F-6
Compatible instrument	Echidna, Kowari, Sika, Taipan, Wombat
Temperature range	300 K to ∼400 K
Mount required for sample	M6 male threaded post
Inbuilt thermometry	CernoxNTC sensor DT-670 silicon diode PT100/1000 platinum resistor
Sample containers	Vanadium can 100 bar Stainless steel furnace can
Complementary ancillary	P-2: 100 kN load frame
User instruction	I-2981 Operation of Environmental Chamber F-6 User Instruction
SICS control	Eurotherm 1 heater
Equipment Number	220185

This environmental chamber is used to keep physically large samples at a well-controlled temperature between room temperature and ∼100°C. A constant flow of nitrogen gas passes over two heater elements to warm the sample mounted inside the chamber. A sliding door provides easy access to the sample mount.

It can be used on Kowari by mounting it on the load frame P-2. Otherwise, it can be mounted on a base which fits the sample stage of Echidna, Sika, Taipan and Wombat.

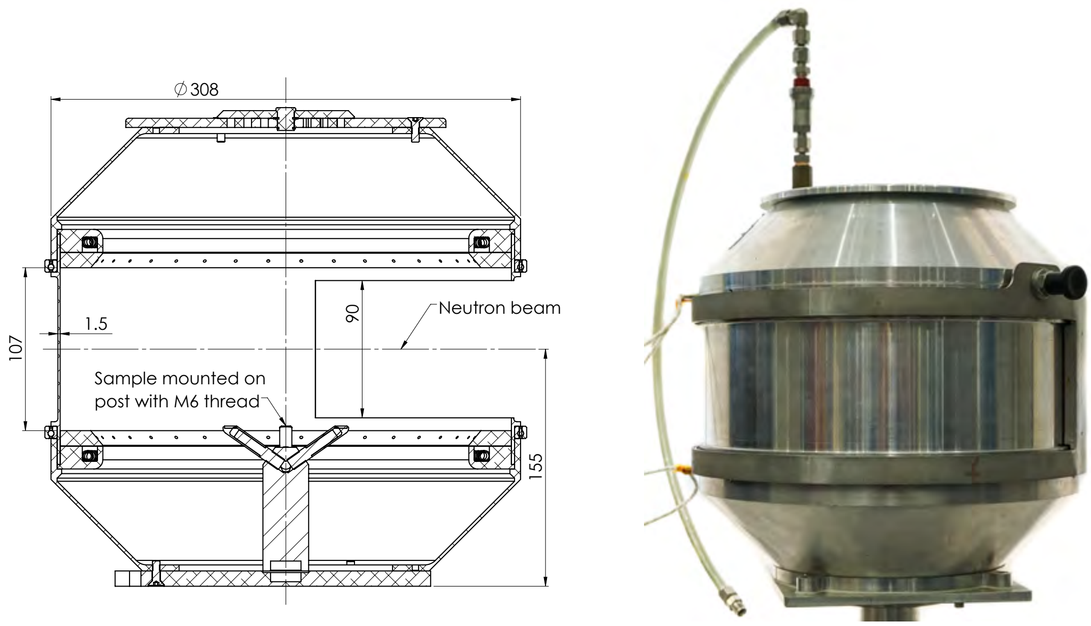


Figure 2.19: Schematic (left) and photo (right) of F-6.

Chapter 3

Magnetic and electric fields

The magnets available at ACNS come in two forms they either have an inbuilt temperature control unit (VTI), or can be fitted with a closed-cycle cryofurnace. The orientation of the magnetic field relative to the neutron beam and, if the sample is a single crystal, to a particular crystal axis is often important. The magnetic field orientations available are either perpendicular to the beam or parallel to the neutron beam. So all vertical magnetic are exclusively perpendicular to the beam. While all horizontal magnets may produce fields perpendicular to the beam, however only HM-1and HM-2can produce fields parallel to the beam. Just to confuse things further HM-3is mounted as a horizontal magnet on Platypusbut is rotated to act as a vertical magnet on Wombat, Bilby, and Quokka.
Superconducting magnets (all magnets except HM-3) are susceptible to "quenching" this occurs when part of the coil winding falls out of the superconducting state.In the non-superconducting state the energising current for the magnet now heats the coils due to I2R heating and the field rapidly collapses releasing even more energy. In wet cryostats AVM-1, AVM-2and HM-2a quench will result in loss of liquid helium and heating of the coils. Re-cooling and refilling the liquid helium may take 12 hours. HM-1is a dry high temperature superconducting magnet hence a quench results in the coils heating and the power supply tripping. The safety trip can only be reset once the temperature of the coils has returned to the safe range.
The most common reasons for a quench occurring are driving the magnet and cryostat beyond the parameters provided by sample environment (ramp rate, max current and max VTI temperature, etc) or the movement of ferromagnetic material near the magnet. To reduce the chance of a quench;

- do not increase field ramp rates without discussion with sample environment
- do not enter the enclosure till the magnet has ramped to less than 1T
- do not conduct a sample change until the magnet has ramped to zero field.
- use only non-magnetic screws and sample holders (no mild steel screws)
- ensure the sample holder is rigid so the sample will not move once the field is applied.

The safe use of high field magnets involves specific procedures. Ensure you are familiar with these before starting your experiment. Access to these procedures and training is provided onsite.

3.1 Horizontal magnets

3.1.1 1.5T Closed-cycle horizontal field magnet HM-1

Ancillary code	HM-1
Compatible instruments	Bilby, Quokka, Kookaburra
Temperature range	6 K to 800 K
Maximum magnetic field	5 T
Time to cool coils	∼60 hours
Thermometry	Dependant on insert
Mount required for sample	M6 male threaded post
Sample containers	Usually purpose built
Complementary ancillaries	CF-1 & CF-3, ambient temperature mount or sample changer EF-1
Project folder	BIP-0012
User instruction	PO-UI-051: 5 Tesla Horizontal Magnet User Instruction I-4522 (PO-I-035): 5 Tesla Horizontal Superconducting Magnet
Manufacturer website	Industrial Research Limited HTS-110
SICS control	Magnet current, Twickenham magnet power supply
Equipment Number	169000

High temperature superconducting magnet designed by HTS-110. Liquid helium free design allows a warm bore construction. Temperature control is via the selected ancillary. Maximum field is limited to 1.5T due to the large fringe fields generated by this magnet.

The sample is mounted either on a bottom-loading closed cycle cryofurnace under vacuum which allows the temperature to be varied from 6 K to 800 K, or the sample can be mounted in air at ambient temperature. Windows allow neutrons to pass through the cryofurnace in either the axial or transverse direction.
A dedicated sample changer is available for room temperature measurements. Two opposing windows are removed to allow the changer to slide into the magnet. translation is control is provided from an unused sample stage axis.

Note: When using the cryostat options Sample changes take several hours as they require the cryofurnace to first be brought to room temperature.

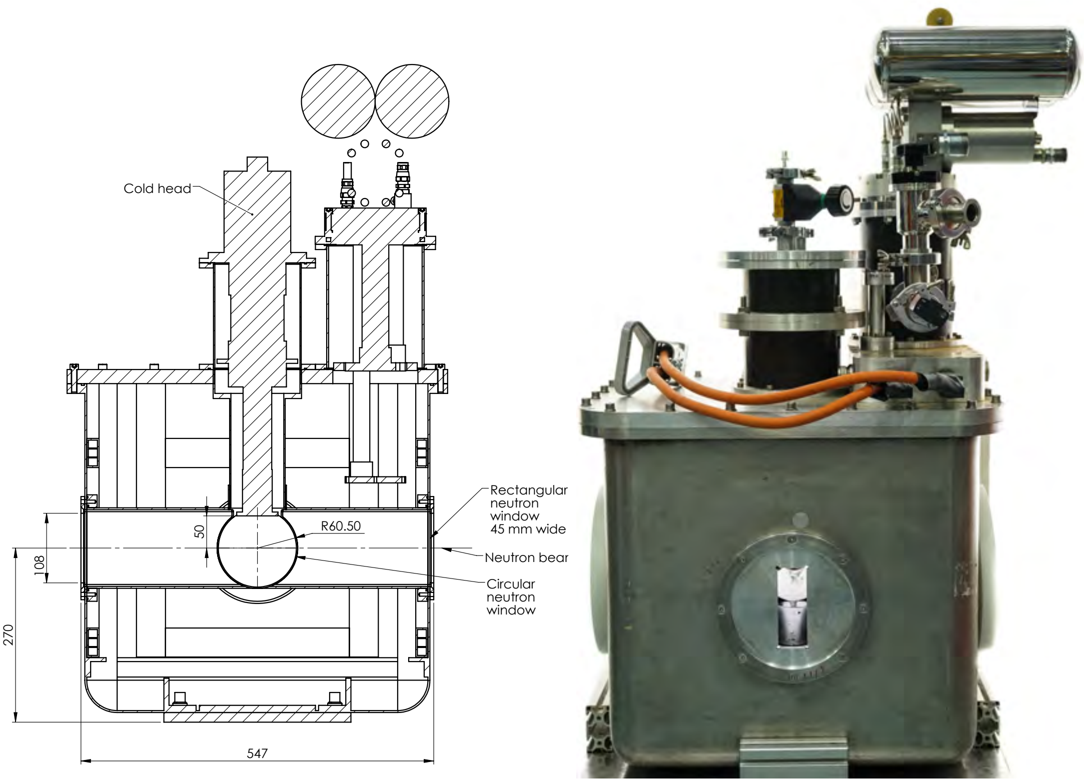


Figure 3.1: HM-1 schematic (Left) including CF-3 and photo (right) including room temperature mount

3.1.2 10 T Wet horizontal field magnet HM-2

Ancillary code	HM-2
Compatible instruments	Bilby, Quokka
Temperature range	1.5 K to 300 K
Maximum magnetic field	10 T (up to 11 T with lambda point refrigerator)
Thermometry	Cernox NTC sensor
Mount required for sample	M6 male threaded post
Complementary ancillaries	OS-1: ^{3}He one-shot refrigerator insert
Project folder	BIP-0039, BIP-0199
User instruction	I-4524 (PO-I-037): 11 Tesla Horizontal Superconducting Magnet
Manufacturer website	Oxford Instruments Spectromag
SICS control	Oxford Labveiw VI (connect to cryostat control laptop)
Equipment Number	171500

An Oxford Instruments Spectromag horizontal superconducting magnet with a peak field of 10 T (with coil temperature 4.2 K) is available for use on Quokka. Though fitted with a lambda plate to allow fields between 10T and 11T this has been disabled due to excessive liquid helium usage please contact the Sample Environment group for advice about this.

Figure 3.2: Photo of HM-2.

3.1.3 1 T Horizontal field electromagnet HM-3

Ancillary code	HM-3
Compatible instruments	Bilby, Platypus, Quokka, Wombat
Maximum magnetic field	1.1 T
Complementary ancillaries	CF-6: bottom-loading cryostat, Hellma cell holder
Project folder	BIP-0071
Manufacturer website	Bruker
SICS control	Magnet current Bruker magnet power supply
Equipment Number	220187

A Bruker B-EC1 electromagnet with a peak field of 1.1 T and a 50 mm opening between the adjustable pole pieces is available for use on Platypus. This magnet has been designed to be used in conjunction with the polarisation system on Platypusproviding a transverse horizontal field this magnet can also be mounted on Bilby, Quokka, or Wombat to provide a vertical transverse field. It is suitable for polarisation experiments.

Figure 3.3: Photo of HM-3.

3.2 Vertical magnet

3.2.1 12 T Re-condensing asymmetrical vertical field magnet AVM-1

Ancillary code	AVM-1
Compatible instruments	Echidna, Sika, Taipan, Wombat
Temperature range	1.5 K to 300 K
Maximum magnetic field	11 T (not currently capable of 12 T)
Thermometry	Cernox NTC sensors
Mount required for sample	M6 male threaded post
Sample containers	OHFC can
Complementary ancillaries	DL-1: dilution refrigerator
User instruction	PO-I-059 Routine Operation of the 12 Tesla Vertical Magnet
Manufacturer instruction manual	Superconducting magnet system operators handbook
SICS control	Oxford Labveiw VI (connect to cryostat control laptop)
Equipment Number	180411

This Oxford Instruments asymmetric re-condensing vertical magnet can reach magnetic fields of up to 12 T, with a maximum ramping rate of 0.5 T/min. The temperature range of the magnet alone is 1.5 K to 300 K, while in conjunction with the Kelvinox dilution insert DL-1 a base temperature of 50 mK can be achieved. A rotation stage allows the sample probe to be aligned *in situ*.

Note: When in persistent mode the magnet power supply will indicate zero current. Press magnet status to determine actual field applied to sample

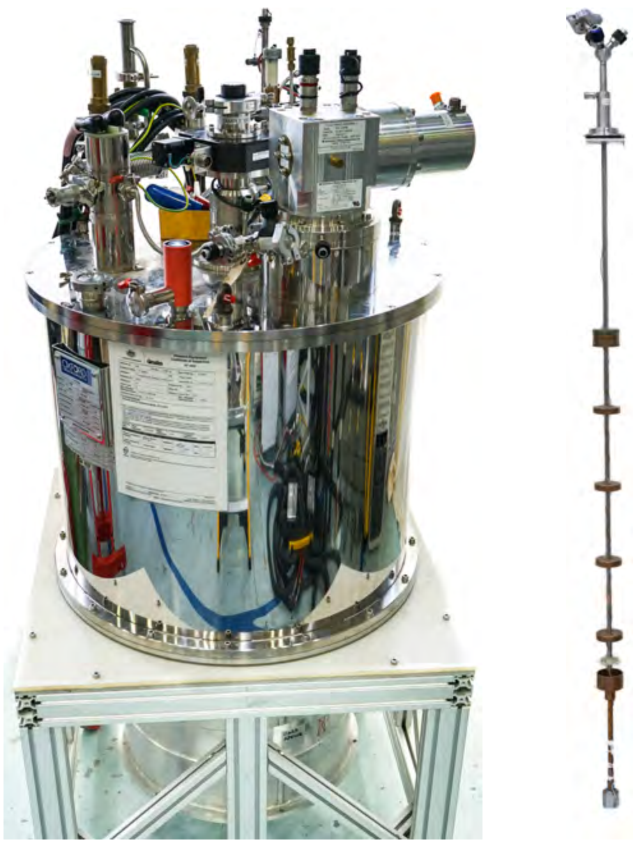


Figure 3.4: Photo of AVM-1 (left) and its sample probe (right).

3.2.2 7 T Re-condensing asymmetrical vertical field magnet AVM-2

Ancillary code	AVM-2
Compatible instruments	Echidna, Sika, Taipan, Wombat, Quokka, Bilby
Temperature range	1.5 K to 300 K
Maximum magnetic field	7 T (max field is reduced above 450K)
Thermometry	Cernox NTC sensors
Mount required for sample	M6 male threaded post
Sample containers	bespoke aluminium holders
Project folder	BIP-0350
SICS control	Oxford Labveiw VI (connect to cryostat control laptop)
Equipment Number	220188

This Oxford Instruments asymmetric re-condensing vertical magnet can reach magnetic fields of up to 7 T, with a maximum ramping rate of 0.5 T/min. The temperature range of the magnet alone is 1.5 K to 300 K. temperatures above 300K are possible using a high temperature stick, However the max field decreases with sample temperature. Discuss your high temperature requirements with sample environment before booking. A rotation stage allows the sample probe to be aligned *in situ*.

Two tail configurations are available to suit either diffraction or SANS. Changing between these tails requires warming the magnet chamber up to room temperature. This takes of the order of a week and consumes significant amounts of liquid helium to cool the winding down. Hence the magnet will be set up for blocks of experiments for each configuration to reduce the number of times this changeover occurs.

The unique design of the aluminum supports between the poles of this magnet provide a reduced background for both scattering and diffraction compared to the 12T magnet vertical AVM-1 which has the standard labyrinth designed supports.

Note: When in persistent mode the magnet power supply will indicate zero current. Press magnet status to determine actual field applied to sample

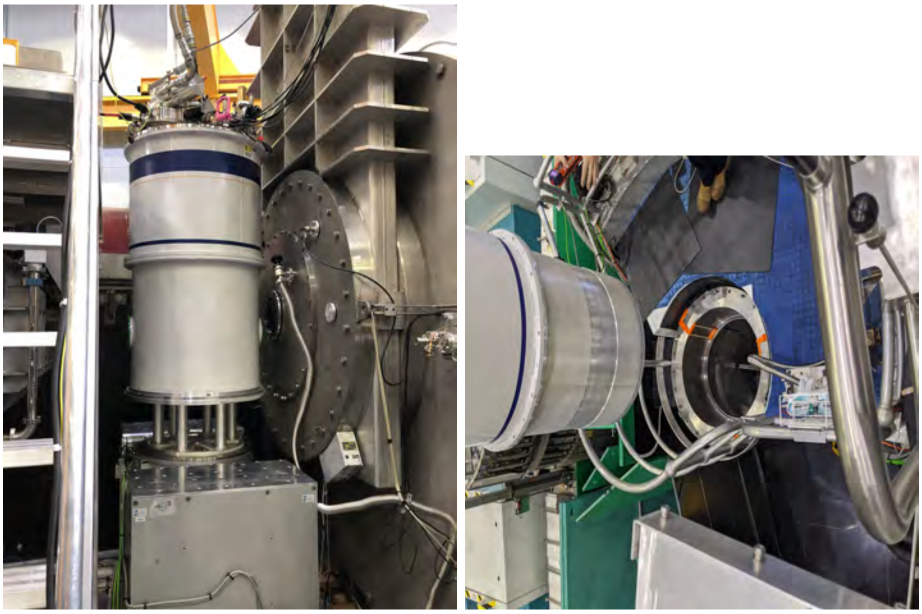


Figure 3.5: AVM-2 Mounted on Quokka (left) and being lowered into the Pelican Sample well (right). Note different tail for scattering and diffraction

3.3 Controlled voltage and current devices

3.3.1 10 kV Electric field system EF-1

Ancillary code	EF-1
Compatible instruments	Echidna, Kowari, Quokka, Sika, Taipan, Wombat
Maximum voltage	±10 kV
Complementary ancillaries	CF-1 & CF-3: bottom-loading cryostat (CF-1 only) CF-6: bottom-loading cryostat
SICS control	Arbitary waveform generator output HV Amp. 1000x gain
Equipment Number	167760

The 10 kV electric field system is used in conjunction with cryostats to provide a controlled electric field for samples. The output of the system is controlled with a waveform generator, which in turn is controlled via SICS. Several power supplies are available for voltages up to ±10 kV, at currents up to 60 mA and bandwidth up to 7 kHz.

Note: Design of electrode systems for high voltage experiments, especially in cryostats, is a very difficult undertaking. Please discuss with sample environment. We may already have a tested system that will suit your experiment.

Figure 3.6: Photo of EF-1.

3.3.2 Potentiostat/galvanostat PG-1

Ancillary code	PG-1
Compatible instruments	All instruments
Maximum voltage	30 V
Maximum current	2 A
Complementary ancillaries	CF-1 & CF-3, CF-6: bottom-loading cryostat SL-1 ambient temperature mount
Project folder	BIP-0186
Manufacturer website	Metrohm
SICS control	None uses Nova software VNC to laptop Can accept TTL trigger from PS-1
Equipment Number	174045

The Autolab 302N potentiostat/galvanostat, used for battery characterisation, cyclic voltametry and impedance spectroscopy, has a maximum current of 2 A, compliance voltage of 30 V, and a bandwidth of 1 MHz.
Can be configured to allow SICS to trigger the start of a sequence. This is very useful in CV as neutron data collection can be synchronised.

Figure 3.7: Photo of PG-1.

3.3.3 AC impedance spectrometer AC-1

Ancillary code	AC-1
Compatible instruments	Platypus
Project folder	BIP-0008
SICS control	none
Equipment Number	XXXXXX

A spectrometer capable of measuring the impedance of a sample in the range 10 kΩ to 100 MΩ.

Figure 3.8: Photo of AC-1.

3.3.4 Battery tester BT-1

Ancillary code	BT-1
Compatible instruments	All instruments
Maximum current	4 channels @10 A 2 channels @20 A
Project folder	BIP-0319
Manufacturer website	Cadex
SICS control	none VNC to Cadex laptop
Equipment Number	220189

The Cadex C8000 battery testing system has four channels which can be programmed to 10 A for charging up to 100 W per channel, and discharging up to 80 W per channel. Channels can be bridged to double current output. The unit incorporates battery temperature monitoring and ramping and cycling of charge/discharge cycles.

Figure 3.9: Photo of BT-1.

3.3.5 SICS controlled power supply PS-1

Ancillary code	PS-1
Compatible instruments	All instruments
Maximum voltage	60 V
Maximum current	10 A
SICS control	control voltage or current, output relay
Equipment Number	220190

These powers supplies are useful for applying low voltages to samples. They can also be used to produce TTL (5V) pulses to trigger sample environment equipment such as PG-1and LED light sources. As the voltage, current and output relay can all be controlled remotely by the SICS the operation can be included in the instruction issued during a neutron experiment.

Figure 3.10: PS-1.

3.3.6 SICS readable multimeter MM-1

Ancillary code	MM-1
Compatible instruments	All instruments
Maximum voltage	120 V
Maximum current	10 A
Project folder	BIP-0240
SICS control	monitor voltage, current or frequency
Equipment Number	167225

Keithley multimeters are available for monitoring voltage, current or frequency signals by SICS. For instance MM-1 can be used to monitoring the analogue output of RH-1related to rotational speed, analogue output from PG-1etc. Please be aware that the update rate for SICS can be quite slow.For rapidly changing signals talk to sample environment about data-logging options.

Figure 3.11: Photo of MM-1.

Chapter 4

Pressure

4.1 Gas pressure and flow

Gas delivery as a controlled pressure or flow can be achieved in a variety of ways. The simplest of these may be for example to plumb a gas supply into a gas delivery probe such as TL-5 & TL-9 or HTS-1. However, the following sample environment setups may be booked in conjunction with a gas delivery probe and compatible cryostat or furnace to allow greater control of such experiments.

Any experiment requiring high gas pressures, the use of flammable or otherwise dangerous gases, or user-supplied gas handling equipment must be discussed with the Sample Environment group in advance as safety approvals may be required.

4.1.1 Gas sorption/desorption system GD-1

Ancillary code	GD-1
Compatible instruments	Echidna, Pelican, Platypus, Sika, Taipan, Wombat
Maximum pressure	200 bar
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace CF-11 & CF-12: top-loading cryofurnace F-5: low temperature furnace GS-1: gas spectrometer
Compatible sample probes	TL-5 & TL-9: gas delivery probe for CF-7 & CF-8 Gas delivery probe for CF-11 & CF-12
User instruction	I-4473 (PO-UI-142): User Guide for Hiden Isochema IMI
Manufacturer website	Hiden Isochema
SICS control	VNC laptop running Hiden software
Equipment Number	220148

The Hiden Isochema IMI-HTP gas sorption/desorption system can be used to deliver up to four different gases (in addition to helium) to samples at pressures of up to 200 bar or flow rates of up to 1000 mL per minute. It can be used to pressurise a sample produce a flow of gases and mix up to four streams or is can be used for quantitative determination of adsorption isotherms.
Gas dosing experiments to determine absorption isotherms use manometric measure-

ments. This is equivalent to the more common gravimetric techniques. However pressure dosing has the advantage of being able to be carried out at higher pressures and temperatures. Prior to dosing experiment pressure measurements with helium (assumed to be non absorbing) is used to determine the system volume. With a know system volume measured changes in pressure can then be related to volumes absorbed or released during the experiment with the reactive gas. Experiments can be performed to measure gas uptake, to provide a constant gas pressure to the sample, or similarly a controlled input or exhaust flow rate of gas. The system is computer controlled and programmable to easily perform pycnometry of the sample, programmed sequences, and isotherm measurements.
The IMI system is frequently used on Echidna and Wombat with CF-7 & CF-8, which requires the use of the gas delivery sticks TL-5 & TL-9. It can also be used with the low temperature vacuum furnace F-5 with samples mounted in a 100 bar stainless steel furnace can, or with a gas delivery probe with CF-11 & CF-12 on Emu and Pelican.

Before booking this piece of equipment consider whether the use of a normal regulator and rotameter will achieve the requirements for your experiment.

Figure 4.1: Photo of GD-1.

4.1.2 Gas mass spectrometer GS-1

Ancillary code	GS-1
Compatible instruments	All instruments
Pressure range	0.1 to 2 bar
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace CF-11 & CF-12: top-loading cryofurnace F-5: low temperature furnace GD-1: gas spectrometer
Compatible sample probes	TL-5 & TL-9: gas delivery probe for CF-7 & CF-8 Gas delivery probe for CF-11 & CF-12
Manufacturer website	Hiden Analytical
SICS control	VNC laptop running Hiden software
Equipment Number	220200

The Hiden Analytical HPR-20 is a compact electrospray mass spectrometer using soft APSI ionisation an detect fragments from 1 to 200 AMU, It can be used as a residual gas detector, detect gas or vapour in any flow stream and can be used in conjunction with the Hiden IMI system GD-1 or the vapour delivery system VD-1for trace analysis of the downstream gas composition. The delivery capillary is heated and the ionisation chamber operates at 200°C. The inlet pressure must be between 200 mBar and 2 Bar.
Detection is not quantitative without calibration. Also as the analysis is carried out on charged fragments of the introduced gases there can be spectral overlaps that must be considered when selecting mass ranges. reference to the NIST ChemWeb Book is a source of mass spectrometer spectral patterns. Note the molecular weight of the starting material may not be the most sensitive peak in the pattern. for instance butane sensitivity is over 6 times great at 43 AMU however its molecular weight is 58 AMU.

Figure 4.2: Photo of GS-1.

4.1.3 Vapour delivery system VD-1

Ancillary code	VD-1
Compatible instruments	All Instruments
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace CF-11 & CF-12: top-loading cryofurnace Ambient temperature chambers
Compatible sample probes	TL-5 & TL-9: gas delivery probe for CF-7 & CF-8 Gas delivery probe for CF-11 & CF-12
Manufacturer website	Hiden Isochema
SICS control	VNC laptop running Hiden software
Equipment Number	220201, 220202

Two Hiden Isochema XCS systems are available for *in situ* static or dynamic vapour delivery. The flowing dynamic system allows for temperatures from room temperature up to 50°C, at a maximum flow rate of 500 mL per minute. The system incorporates 3 mass flow controllers and two vapour generators. the mass flow controllers set the flow and ratio of gases and the vapour generators (one for water and one for organic solvents) can be used in any combination to suit the experiment. The unit can be used without he vapour generators to provide a mixed gas flow.

The static system uses two computer control needle valve to control vacuum and vapour delivery to maintain a constant vapour pressure in the sample chamber. This means the sample itself must not out-gas when under vacuum and the chamber should hold a high static vacuum pressure. Hence the chamber and seals all must not out gas so no elastometric seals should be used in its construction. The vapour pressure can be ramped, regulated or dosed. So kinetics of vapour interaction with the sample can be investigated. The vapour delivery capillaries on both system are heated to prevent condensation. Please discuss your vapour delivery requirements with sample environment prior to your experiment so the appropriate system and sample chamber is used. In some cases a bespoke chamber may need to be developed before your arrival.

Figure 4.3: Photos of the dynamic (left) and static (right) vapour delivery systems.

4.2 Uniaxial loading

The following sample environment setups are available to apply mechanical pressure to samples during neutron experiments.

4.2.1 Load frame P-2

Ancillary code	P-2
Compatible instruments	Kowari
Maximum applied load	100 kN
Mount required for sample	Load frame grips
Complementary ancillaries	F-4: load frame furnace F-6: environmental chamber
Project folder	BIP-0038, BIP-0265
User instruction	I-4446: KOWARI 100 kN Load Frame
Manufacturer website	Instron
SICS control	VNC laptop running Instron
Equipment Number	168747

The Instron 100 kN horizontal load frame is used for for tension, compression and fatigue testing on Kowari. This load frame can fatigue samples at rates of up to 10 Hz. Temperature control is possible with use of furnaces F-4 or F-6. A variety of sample mounting grips are available depending on the sample temperature.

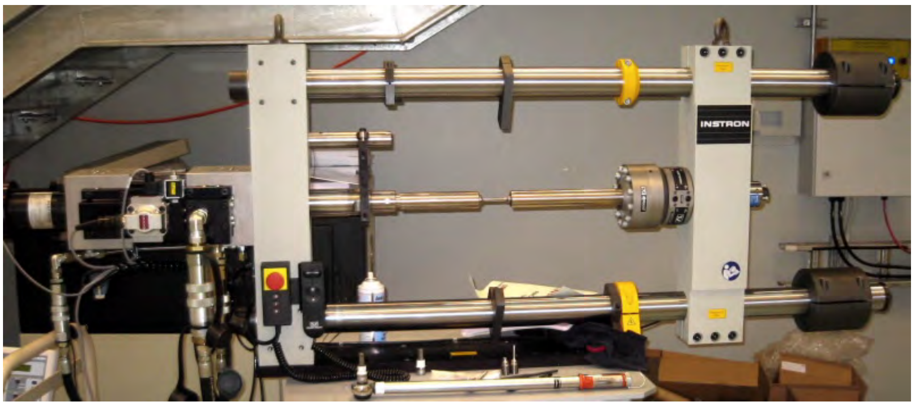


Figure 4.4: Photo of P-2.

4.2.2 Paris-Edinburgh cell P-3

Ancillary code	P-3
Compatible instruments	Echidna, Sika, Wombat, Taipan, Taipan: Be Filter
Maximum pressure	10 GPa
Sample containers	TiZr gaskets
Complementary ancillaries	EF-1: high voltage supply
Project folder	BIP-0015, BIP-0094
User instruction	PO-UI-059: Paris-Edinburgh Cell User Instruction I-4466 (PO-UI-125): Gasket Preparation for P-3
SICS control	Monitors pressure. Change in pressure are manual
Equipment Number	168704

Our VX-5 Paris-Edinburgh cell is manually operated with a hydraulic hand pump. An aluminium chamber has been designed to allow the PE cell to be operated in vacuum. With pyrophylite gaskets and mica and PTFE anvil supports voltages up to 5kV can be applied across the sample while pressurised.

Samples are loaded into null-scattering TiZr gaskets, typically along with a pressure calibrant such as lead, and a pressure transmitting medium such as a mix of deuterated methanol and ethanol. Although achieving sample pressures of around 5 GPa is relatively routine, attaining sample pressures of above 1 GPa is dependent on the correct sample and loading procedure. Particular attention to the gasket cleaning, selection and location on the anvils must be made to ensure the gaskets do not fail during loading.

Note: Before mounting on the Taipan: Be Filterthe PE cell has to be rotated on its stand. Positions for Wombat and Taipan: Be Filterare marked. The Wombat position is suitable for the other listed instruments.

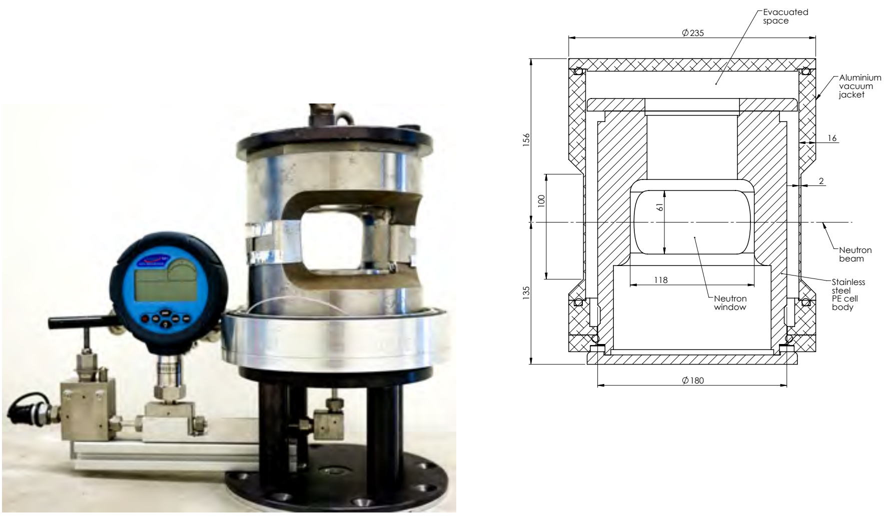


Figure 4.5: Photo of the Paris Edinburgh cell (left) without the vacuum jacket fitted and schematic (right)

4.2.3 Rotating Paris-Edinburgh cell P-10

Ancillary code	P-10
Compatible instruments	TBA (to be commissioned on Dingo and Wombat)
Maximum pressure	4 GPa
Sample containers	TiZr gaskets
Project folder	BIP-0329
SICS control	Monitors pressure. Change in pressure are manual
Equipment Number	220203

The rotating Paris-Edinburgh cell is based on a V7 Paris-Edinburgh cell. The modifications allow the anvils to be rotated relative to one another while the sample is under load. This imparts a torsional shear along with the uniaxial loading to simulate what might be expected in some geological settings.

Two sample configurations have been developed the first can achieve 3.2 GPa and 750 °C and contain four samples with a volume of 4 cm3 each, while the second can achieve 4 GPa and 900 °C and has a single sample volume of 11 cm3.

The Rotating Paris-Edinburgh cell (RoPEC) has completed offline testing and will be commissioned for use on Dingo and Wombat.

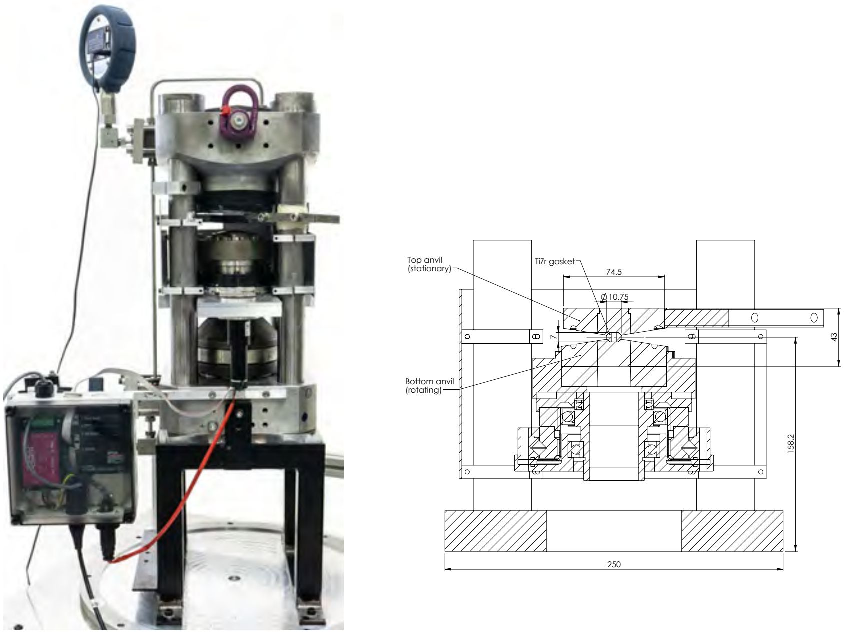


Figure 4.6: Photo of P-10 (Left) and Schematic (right).

4.3 Isostatic loading

The following sample environment setups are available to apply isostatic pressure. Generally this equipment can be used for both solid and liquid samples. The hydraulic fluid (gas or liquid) may also be the sample or a neutron transparent fluid (ie D2O) may be used to pressurise a solid sample. The pressure transfer fluid loads to samples during neutron experiments.

4.3.1 SANS high pressure rig SPC-1

Ancillary code	SPC-1
Compatible instruments	Quokka, Bilby, Kookaburra
Temperature range	15°C to 70°C
Maximum stress	450 MPa
Project folder	BIP-0042
SICS control	Monitors pressure. Change in pressure are manual
Equipment Number	220204

The SANS high pressure rig uses a manual pressure generator manufactured by HIP to control the pressure applied to the sample. The sample may be liquid or solid. Mounted in the high pressure cell. Use only the high pressure cell designed for this rig.

Figure 4.7: Photo of SPC-1 on Quokka (right) and pressure intensifier(left).

Chapter 5

Techniques

We offer a range of sample environments for experiments involving complementary techniques . This equipment is available to all instruments though some techniques may not be suitable without considerable modification. Please discuss with sample environment if you'd like to use these on instruments other than those listed in the descriptions.

5.1 Rheology

5.1.1 MCR-500 rheometer RH-1

Ancillary code	RH-1
Compatible instruments	Bilby, Kookaburra, Quokka, Wombat
Temperature range	-40°C to 80°C
Geometries provided	Cylinder in Cylinder Cone Plate (Silicon wafer)
Project folder	BIP-0023
Manufacturer website	Anton Paar
SICS control	None uses Rheoplus software VNC to laptop can produce analogue coded voltages monitor using MM-1 can produce trigger to sync for oscillatory rheology
Equipment Number	168661

This rheometer is capable of shear rates up to 2500 s^{-1}, and a temperature range of -40°C to 80°C. Both Continuous and oscillatory shear is available. In the oscillatory mode a trigger is provided to synchronise the neutron data collection so post-binning of the data can be carried out. The RheoSANS is based on the Anton-Paar MCR 500 rheometer modified for *in situ* SANS measurements. It is controlled by Anton Paar RheoPlus software where SANS data may be acquired under different shear stress, strain and temperature conditions at the same time rheology measurements conducted. Temperature is logged at the sample position.

The cylinder in cylinder measurement system (referred to as CIC or Couette) consists of two concentric quartz cylinders separated by a 0.5 mm gap where up to 6 mL of sample is loaded. The static outer cylinder is covered by temperature jacket which does not obstruct the neutron beam, while the inner cylinder is rotated and is the rheology sensor. SANS data may be acquired along an axis either tangential or normal to the circumference of the cylinder.

The cone/plate measurement system consist of a rotating cone (very nearly flat) and a fixed silicon wafer. This arrangement is used on the reflectometer where the neutron beam enters via the fixed silicon wafer and is reflected onto the detector. This requires the rheometer to be tilted by several degrees.Temperature of the sample is controlled via a Peltier hotplate under the silicon wafer.

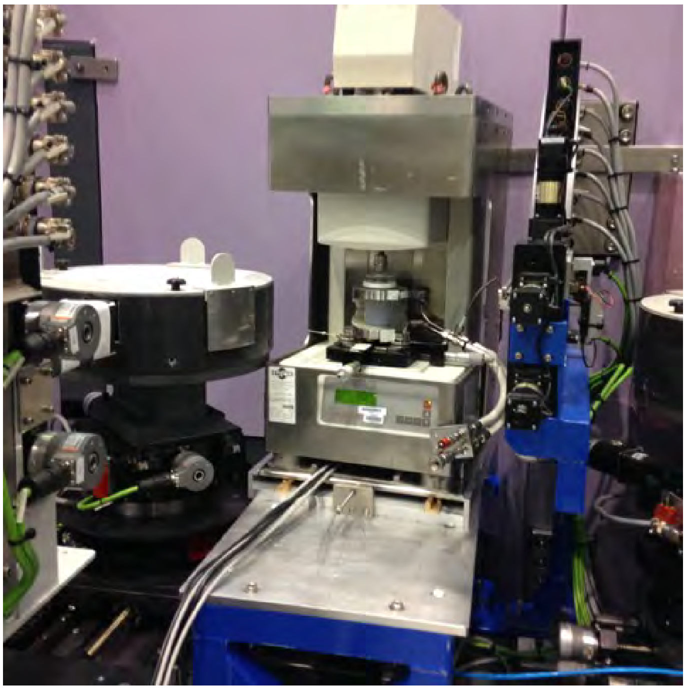


Figure 5.1: Photo of RH-1 mounted on Kookaburra.

5.1.2 Rapid viscosity analyser RV-1

Ancillary code	RV-1
Compatible instruments	Quokka, Bilby, Dingo
Project folder	BIP-0117, BIP-0251
User instruction	PO-UI-108: Rapid Visco Analyser User Instruction
SICS control	None uses Perten software VNC to laptop
Equipment Number	220192

This viscometer is a modified PERTEN Instruments RVA . These instruments were developed to characterise starch rheological behaviour in the food industry. To allow SANS measurements the standard paddle arrangement has been modified. Samples other than starch may be used in the RVA check with sample environment to ensure your sample is compatible. Samples

This viscometer is a modified PERTEN Instruments RVA . These instruments were developed to characterise starch rheological behaviour in the food industry. To allow SANS measurements the standard paddle arrangement has been modified. Samples other than starch may be used in the RVA check with sample environment to ensure your sample is compatible. Samples are contained within a block allowing accurate temperature control up to 140°C.

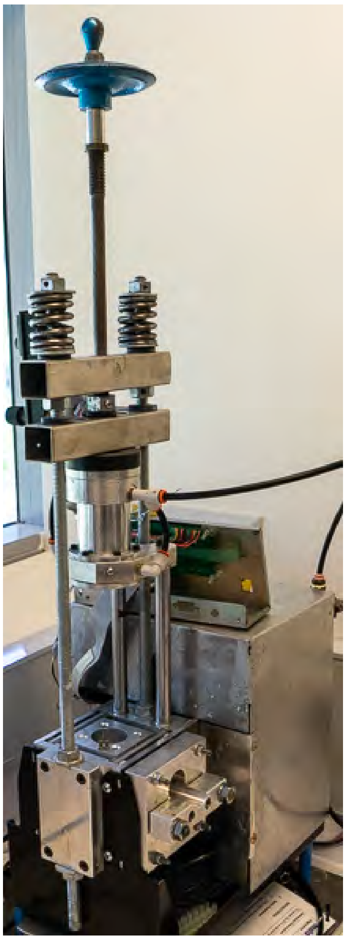


Figure 5.2: Photo of RV-1.

5.2 Solvent delivery and mixing

5.2.1 Programmable syringe pump SP-1

Ancillary code	SP-1
Compatible instruments	All Instruments
SICS control	Dose and time.
Equipment Number	220193

Holds one syringe with a volume of up to 50 cm^3, and has infusion rates from 0.00073 mL/hr (1 cm^3 syringe) to 1750 mL/hr (50 cm^3 syringe).
Standard syringe volumes available at ACNS are 1, 3, 5, 10, 20 and 50ml. All are compatible with the syringe pump. Be sure to change the syringe volume setting so the programmed dosages are dispensed.

Figure 5.3: Photo of SP-1.

5.2.2 Quaternary HPLC pump QP-1 & QP-2

Ancillary code	QP-1
Compatible instruments	All instruments
SICS control	Each channel, flow and time.
Equipment Number	220194, 220195

Three quaternary HPLC pumps are available. Two are integrated into SICS and normally used with SL-1on Platypusand Spatzthe third is available for general use. Quaternary pumps provide four solvent channels that can be mixed to give controlled concentrations and flow rates of from four stock solutions. A suitable flow cell is also required. Discuss with sample enviroment before your experiment.

5.2.3 Stop-flow mixing cell SF-1

Ancillary code	SF-1
Compatible instruments	Bilby, Quokka
Project folder	BIP-0036, BIP-0307
User instruction	PO-I-040: Stopped Flow Cell Operating Instruction
Manufacturer website	BioLogic
SICS control	None uses Biologic software VNC to laptop produces TTL pulse to trigger Histogram server on Quokka
Equipment Number	171049

A BioLogic SFM-300 stopped flow mixing cell is available to study small angle scattering from samples as a function of time after mixing. Up to three liquids may be mixed. Maximum sample delivery volume of 10 mL without refilling. 2ml required to flush and fill the sample cuvette.

A trigger signal is provided to synchronise neutron data collection with the start of mixing. This allows Kinetic measurements to be carried out.

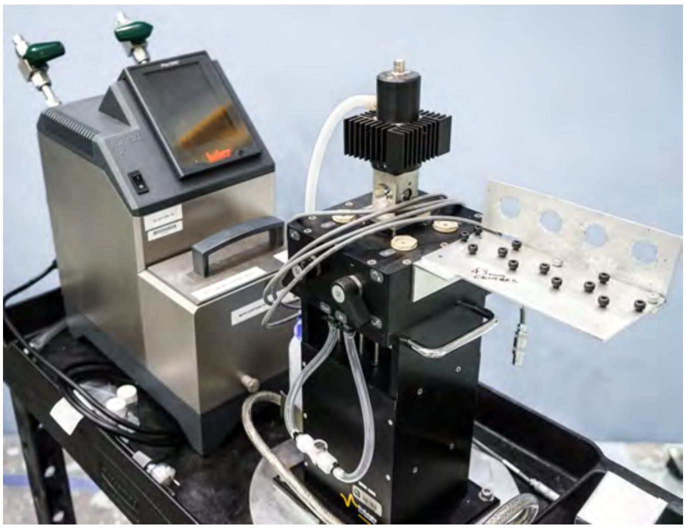


Figure 5.4: Photo of SF-1.

5.2.4 Peristaltic dosing pumps PP-1, PP-2

Ancillary code	PP-1, PP-2
Compatible instruments	All instruments
SICS control	Use PS-1to control flow rate.
Equipment Number	167729, 220196

A ColeParmer 7550-30 (PP1) and a Gilson Minipuls 3 (PP2 selection of 2 or 4 channel head) peristaltic pumps are available. one low flow 4 channel head and the other is high flow dual channel head. The pumps can be switch on and off remotely. Useful for use with SANS flow cell

Figure 5.5: PP-1 and PP-2

5.3 Monolayer sample preparation

5.3.1 Langmuir film balance LF-1 & LF-2

Ancillary code	LF-1 & LF-2
Compatible instruments	Platypus
Project folder	BIP-0049, BIP-0250, BIP-0310
SICS control	None uses Nima software VNC to laptop
Equipment Number	168753

We can provide in situ LB troughs for use on the reflectometers and LB trough with dipping function (Langmuir Schaefer trough) for offline multi-layer self assembled sample preparation. NIMA and Kibron troughs

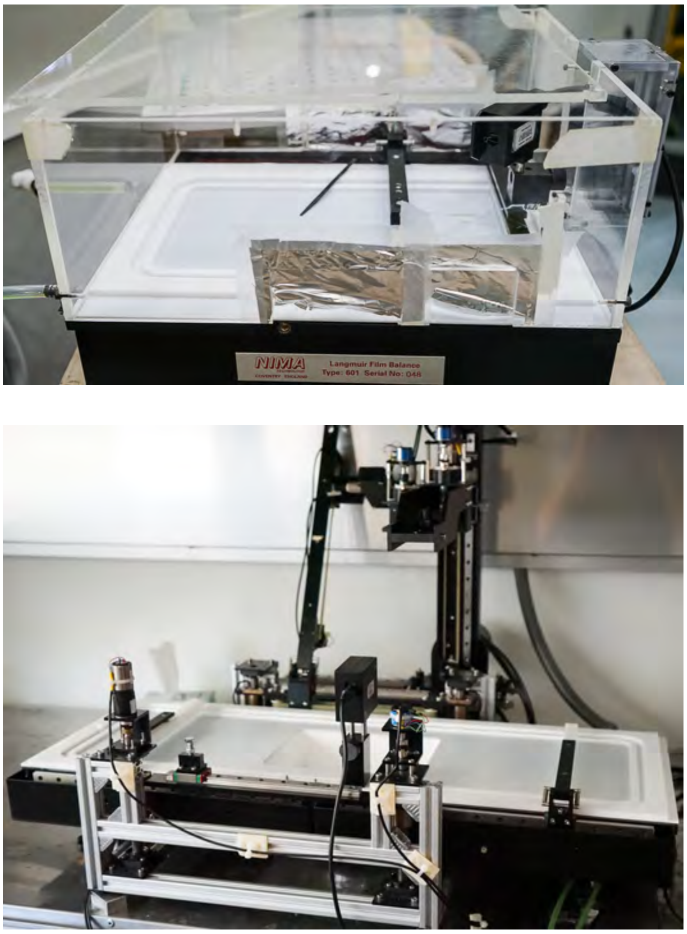


Figure 5.6: Photos of LF-1 and LF-2.

5.4 Thermal analysis equipment

5.4.1 Differential scanning calorimeter DSC-1

Ancillary code	DSC-1
Compatible instruments	Quokka
Project folder	BIP-0087, BIP-0296
SICS control	VNC to control laptop, TTL Trigger output monitored by SICS
Equipment Number	220197

The *in situ* DSC is based on a Mettler DSC-1. Modified pans are required so the neutron beam can pass through the DSC oven and the sample. Sample are small 7mm x 7mm x 1mm max dimensions to fit the modified pan. The DSC allows Tm, Tg, enthalpy of transition and phase changes to be accurately recorder while neutron data is collected. A trigger at the start of a DSC run synchronises the neutron collection with the DSC data. The temperature range for our differential scanning calorimeter is -50°C to 350°C.

Figure 5.7: Photo of DSC-1.

5.5 Optical spectroscopy and illumination equipment

5.5.1 *In situ* ATR FTIR spectrometer IR-1

Ancillary code	IR-1
Compatible instruments	Spatz
Project folder	BIP-0342
SICS control	VNC laptop OPUS software, SICS TTL trigger for scan using PS-1
Equipment Number	220003

IR-1 is a modified Bruker Vertex FT-IR used in conjunction with Silicon ATR neutron reflectivity solid liquid cells on Spatz. the source and beamsplitter are mounted in the unit above the sample while the detector is mounted below. the sample coated on the silicon ATR crystal is mounted vertically between the source and detector upon a linear stage to allow the signal from the to be optimised

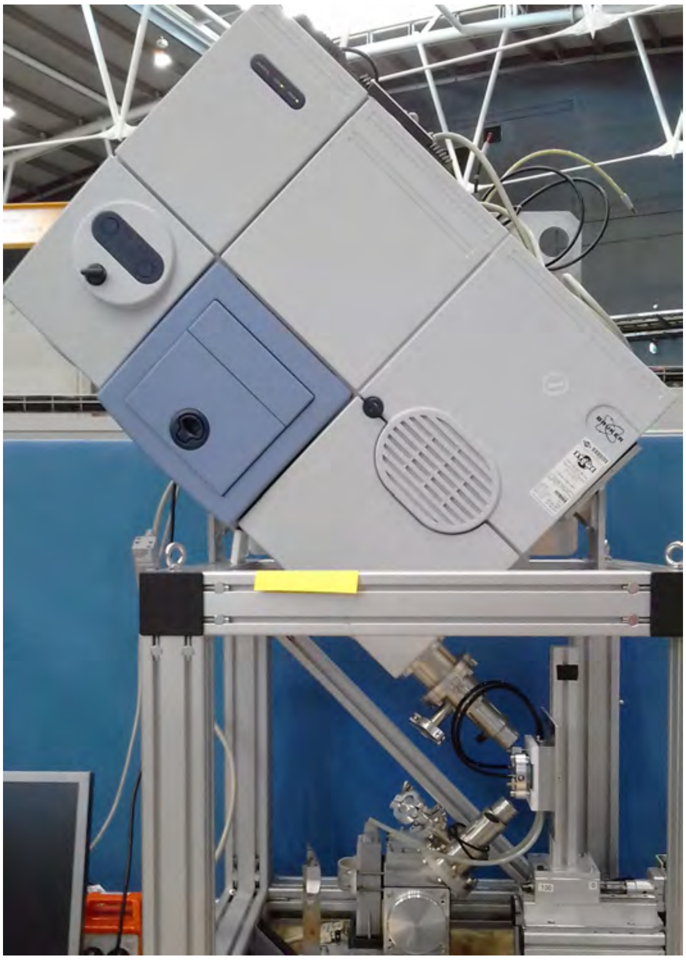


Figure 5.8: IR-1 bench testing.

5.5.2 Fibre optic spectrometers OF-1 & OF-2

Ancillary code	OF-1
Compatible instruments	all instruments
SICS control	VNC laptop Oceanview software SICS trigger light source using PS-1
Equipment Number	220198, 220199

A number of spectrometers are available in sample environment for *in situ* measurements. These compliment the spectrometers available in the laboratories at ACNS. For *in situ* measurements there is available a NIR and UV/Vis fibre optic spectrometers with associated fibres light source and vacuum feedthroughs that allow a range of applications on the neutron instruments. If you are interested in optical measurements please discuss your interests with Sample Environment staff prior to submitting your proposal. The graph below summaries the wavelength range available for our spectrometers and light sources. Please also look at the optical sample probes available in the next chapter.

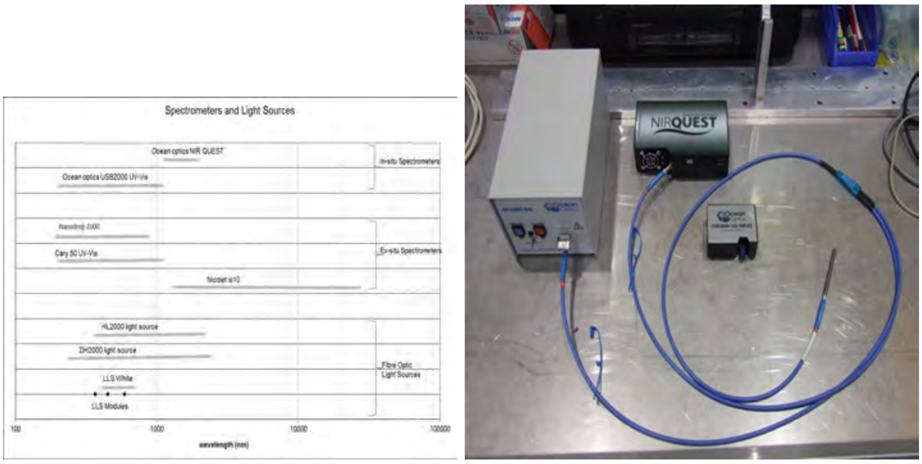


Figure 5.9: Summary of available wavelengths and picture of fibre optic spectrometers.

Chapter 6

Sample changers and manipulators

We offer a variety of sample environments for sample mounting, whose capabilities cover most different types of neutron instruments. Sample positioners for texture measurements are listed in §6.1, while multiple position sample changers for soft-matter experiments and robotic sample changers are described in §6.2 (note that some setups such as SC-5 & SC-10 are capable of either functionality). Note that some sample changers can provide temperature regulation of samples and are provided with a temperature bath.

All cryostats and furnaces are supplied with a standard set of sample probes, and its the temperature range of these that are given in the information for them. However their functionality can be extended with the use of the specialty probes in §6.3. Also, the range of sample cells available for use with our sample environments is listed in §6.4. While cells are not necessarily bookable items in their own right, it is best to discuss their use with your instrument scientist before your experiment.

6.1 Sample positioners

6.1.1 Eulerian cradle EC-1

Ancillary code	EC-1
Compatible instruments	Echidna, Quokka, Sika, Taipan, Wombat, Kowari
Complementary ancillaries	CF-5: bottom-loading cryostat ambient temperature mount
Project folder	BIP-0267
Manufacturer website	Huber
SICS control	Omega, chi, phi angles
Equipment Number	220205

This Huber 512.5 Eulerian cradle can be used in conjunction with the closed cycle refrigerator CF-5 to reach temperatures of 10 K to 300 K or with a adjustable ambient temperature mount. Can be used for single crystal and texture measurements. A laser alignment tool is available to ensure the sample is aligned to cradle's centre of rotation.

Figure 6.1: Photo of EC-1.

6.1.2 Eulerian cradle EC-3

Ancillary code	EC-3
Compatible instruments	Kowari
Project folder	BIP-0105, BIP-0183
SICS control	Omega, chi, phi angles
Equipment Number	220206

This small Eulerian cradle can be used for samples on Kowari.

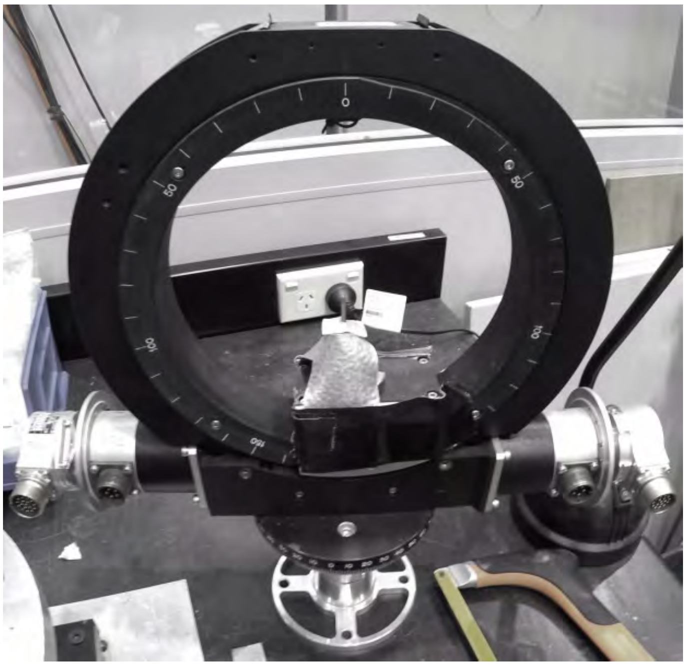


Figure 6.2: Photo of EC-3.

6.2 Sample changers

Two types of sample changers are listed in this section. We provide static sample changers that rely on the movement of the instrument sample stage to move different samples into the beam. These are simple linear translations of a collection of samples. These are SC-1, SC-2, SC-3, SC-7, SC-8, SC-9.The second type use a programmable robot to select a sample and present it to the beam and the instrument sample stage remains in a fixed position.

6.2.1 Ten-position sample changer SC-1

Ancillary code	SC-1
Compatible instruments	Bilby, Quokka
Temperature range	30°C to 300°C or 0°C to 100°C
Compatible sample cells	Demountable cell
Project folder	BIP-0031, BIP-0255
User instruction	PO-UI-099 SANS 300C 10 position sample changer User Instruction
SICS control	Single heater, individual sample temperature sensors available Silicone oil bath and single sensor in low temp mode.
Equipment Number	220207

The ten-position sample changer for Quokka and Bilby can hold up to ten SANS Demountable cell or Hellma cells. The sample changer can be configured to deliver two different temperature ranges. The default configuration uses temperature controlled fluid to control the sample temperature. The fluid circulates in the top and bottom plates. In the second configuration high power Watlow Firerod cartridge heaters are used to heat the body of the sample changer and cooling water circulates in the top and bottom plates. Weak thermal link is introduced between the plates and the body in this configuration.this gives a range of temperatures between 30°C and 300°C.
temperatures below 15° required a dry nitrogen purge to prevent condensation forming on the windows of the sample cells.

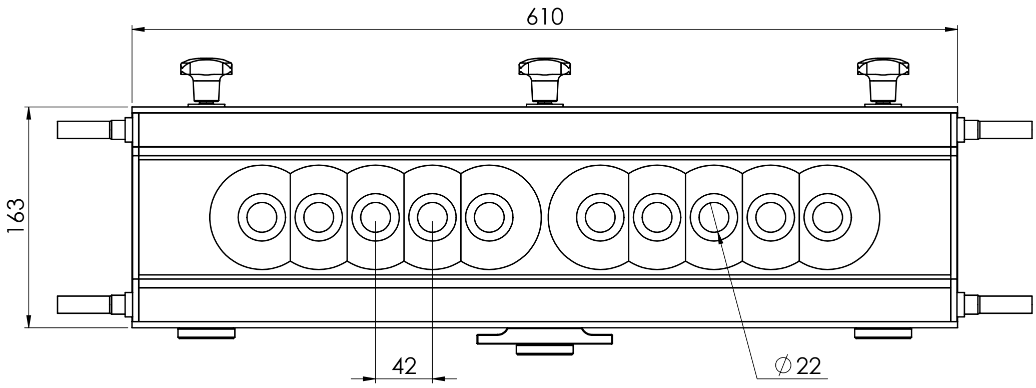


Figure 6.3: Schematic of SC-1.

6.2.2 Twenty-position sample changer SC-2

Ancillary code	SC-2
Compatible instruments	Bilby, Quokka
Temperature range	-15°C to 120°C
Compatible sample cells	Demountable cell
Project folder	BIP-0213, BIP-0255
SICS control	Silicone oil bath and single sensor
Equipment Number	168999

The twenty-position for Quokka and Bilby can hold up to twenty SANS Demountable cell or Hellma cells. Its uses temperature controlled fluid circulated through the body of the changer to control the temperature of the sample. A temperature sensor is mounted in one of the cell holders and this should be placed in the centre position (10 or 11) or close to the most temperature sensitive samples if the changer is not completely full. The sample changer operates at temperatures from -10°C to 80°C. An optical breadboard can be provided that mounts on the top of the changer. This can be used for mounting non standard sample holders or equipment such as power supplies or peristaltic pumps when flow-cells are being used in the changer.
A nitrogen shroud it provided for experiments below 15° to prevent condensation forming on the windows of the sample cells. This shroud, with no nitrogen flow, is also useful for high temperatures to prevent excessive heat loss

Figure 6.4: Photo of SC-2.

6.2.3 Air/Liquid cell sample changer SC-3

Ancillary code	SC-3
Compatible instruments	Platypus
Temperature range	5°C to 50°C
Compatible sample cells	Sealed sample cells
SICS control	5 sensors and heaters available Lakeshore compatible
Equipment Number	168662
Project folder	BIP-0112

Based on a design from the ISIS neutron scattering facility. Up to five air / liquid troughs mounted and their temperature controlled via membrane heaters on the base of the Teflon trough holders. Windows at either end of the troughs allow neutron reflectivity to be carried out on self assembled layer on the surface of the solvent used to fill the troughs. Discuss with your instrument scientist whether this sample or a LF-1 & LF-2trough would be the best selection for your experiment. The temperature range available is currently 25°C to 50°C. The trough is used for studies of liquid-air interfaces.

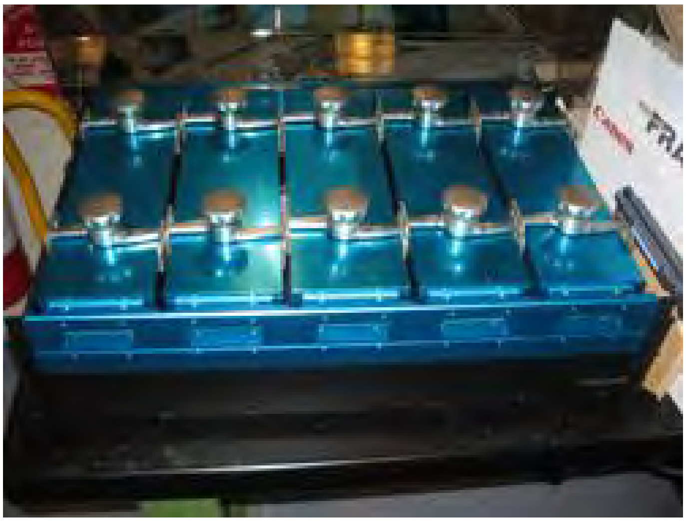


Figure 6.5: Photo of SC-3.

6.2.4 SCARA robotic sample changer SC-4

Ancillary code	SC-4
Compatible instruments	Echidna, Wombat
Compatible sample cells	Vanadium can
Project folder	BIP-0016
Manufacturer website	Epson
SICS control	Pick and place of samples
Equipment Number	168750

The Epson E2S553 SCARA robot “Robbie” is available to allow repeated automatic sample changes. It has a reach of 550 mm, and is able to use up to two trays of fifty Vanadium cans for testing individually in the neutron beam. This robot is also able to rotate and translate samples in the beam. To meet safety requirements the robot is enclosed and interlocked to the instrument enclosure door. An emergency stop button is located on the left hand side of the trolley. Ensure you know can identify the emergency stop button location prior to using the robot.

Note: Robbie is most frequently used on Echidna. A vacuum chamber is available for samples to be mounted in to reduce air scattering.

Figure 6.6: Photo of SC-4.

6.2.5 Six-axis robotic sample changer SC-5 & SC-10

Ancillary code	SC-5
Compatible instruments	Kowari, Wombat, Echidna
Compatible sample cells	Vanadium can
Project folder	BIP-0206, BIP-0314
User instruction	PO-UI-107: 6 Axis Robotic Sample Changer User Instruction
Manufacturer website	Epson
SICS control	Pick and place or texture (omega,chi,phi)
Equipment Number	174268, 220208

An Epson C3 robot “Rosie”(SC-5) is a six-axis robot and Epson C4 robot "RALF"(SC-10) are for sample pick and place as a replacement for SC-4 and texture measurements on Kowariand Wombat. SC-5 is configured for use on Echidnawhen higher monochromator angles are used and SC-4 physically can not fit between the sample table and the neutron guide. This robot can select from two trays of fifty Vanadium cans, and has a maximum load capacity of 3 kg. To meet safety requirements SC-5 is operated in low power mode and is interlocked to the enclosure door. The emergency stop button is on the instrument touchscreen and a mobile emergency stop is available in the enclosure. Note the position of the emergency stop before starting operation of the robot.

Figure 6.7: Photo of SC-5.

6.2.6 Five position sample changer SC-6

Ancillary code	SC-6
Compatible instruments	Kookaburra,
Compatible sample cells	Demountable cell
SICS control	none ambient only
Equipment Number	220209

Non rotating ambient temperature holder for Kookaburra. This holder is useful for experiments requiring the use of the Kookaburraflow cell and other non-routine experiments. The changer can hold either the large round or rectangular Kookaburra cells. Only book SC-6 if the experiment can not be carried out in SC-9

6.2.7 Five position rotating tumbler SC-7

Ancillary code	SC-7
Compatible instruments	Bilby, Quokka
Complementary ancillaries	SC-2: 20 position sample changer
Compatible sample cells	Hellma cell
SICS control	rotation speed via PS-1
Equipment Number	220210

The rotating sample tumbler for Quokka and Bilby can hold up to five round Banjo-style Hellma cells of differing path lengths. The cells are gently rotated to help keep particulates in suspension. No temperature control is available for this sample changer. For /QUO this sample changer mounts on top of SC-2and so it should be booked as well. Both sample changers can be used at the same time.

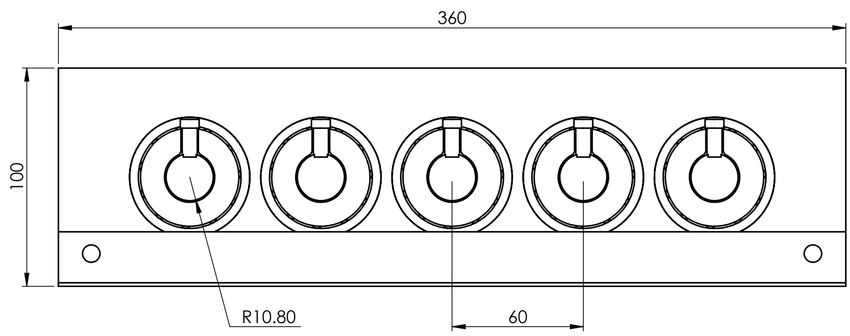


Figure 6.8: Schematic of SC-7.

Figure 6.9: Photo of SC-7.

6.2.8 Twelve position Peltier sample changer SC-8

Ancillary code	SC-8
Compatible instruments	Bilby, Quokka
Temperature range	-20°C to 90°C
Compatible sample cells	Hellma cell Demountable cell
Project folder	BIP-0363
SICS control	Watlow controller temperature control for each sample (peltier) Silicone oil bath base temperature control
Equipment Number	220211

The twelve-position sample changer operates at temperatures from -20°C to 90°C. It provides temperature control using Peltier technology to actively cool and heat each sample position independently. Thermal isolation allows for a temperature difference of up to 40°C between adjacent cells. Through a spring-loaded mechanism, sample changes are readily achieved with both Demountable cells and Hellma cells. A shroud and nitrogen purge is required for temperatures below 15° C to prevent condensation on the windows of the sample holders.

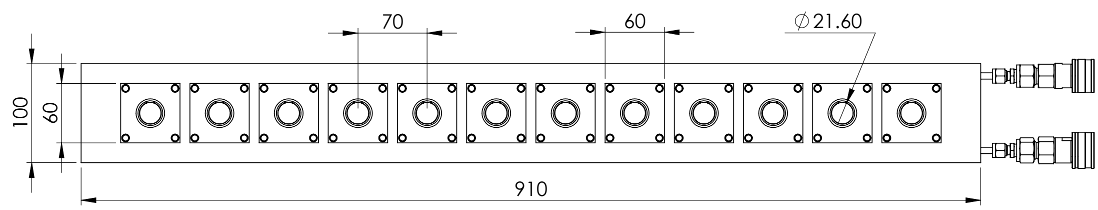


Figure 6.10: Schematic of SC-8.

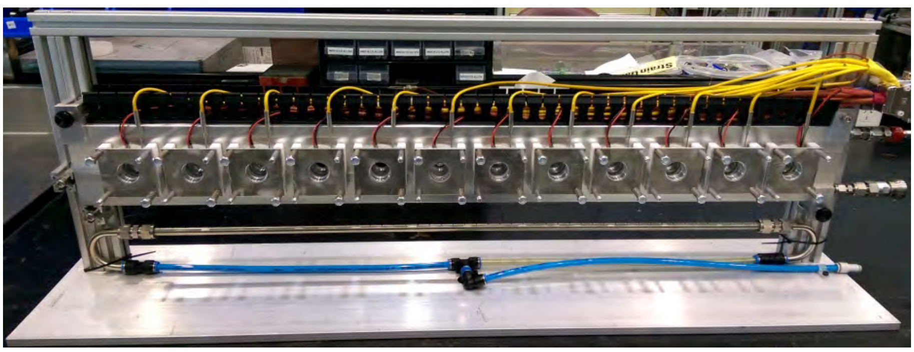


Figure 6.11: Photo of SC-8.

6.2.9 Six position rotating sample changer SC-9

Ancillary code	SC-9
Compatible instruments	Kookaburra
Temperature range	20°C to 80°C
Compatible sample cells	Hellma cell Demountable cell
Project folder	BIP-0365
SICS control	Watlow controller temperature control for each sample heater Silicone oil bath base temperature control
Equipment Number	220212

The Kookaburra six-position rotating tumbler operates at temperatures from ambient to 80°C. A motor-driven gear train allows for samples to be rotated at fixed speed. Temperature control is via temperature controlled fluid circulating through the main plate of the sample changer. membrane heaters are provided near the samples so small temperature variation can be controlled . Temperature sensor and membrane heaters are connected via electrical slip-rings to allow the sensor and heater to rotate with the sample while still being connected to the control circuit. Dedicated cell are used with this changer to allow the full 50mm neutron beam to be used. Through a screw-in mechanism, sample changes are readily achieved.

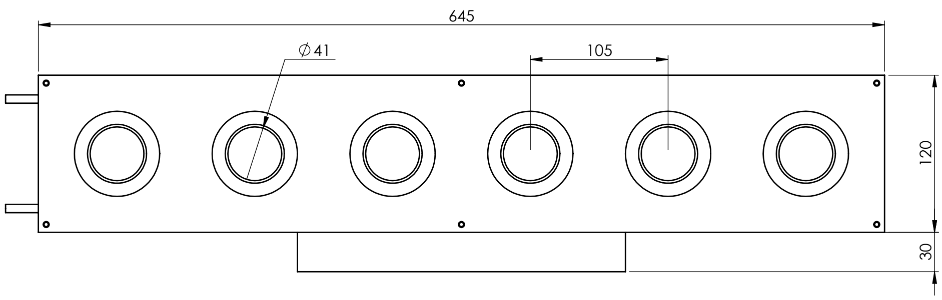


Figure 6.12: Schematic of SC-9.

Figure 6.13: Photo of SC-9.

6.3 Sample probes

The furnaces and cryofurnaces at ACNS are provided with a standard probes when they are booked. Hence these probes are not separately bookable but are supplied by default. However we also have a range of special purpose probes that are described below. Please consult with the Sample Environment group before booking any of these specialised sticks.
Probes listed as standard below are not bookable as they are supplied by default when the cryostat is booked.

6.3.1 CF-7, CF-8 Cryofurnace standard probe

Ancillary code	None
Temperature range	4 K to ∼400 K
Thermometry	DT-670 silicon diode
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

The standard for CF-7 & CF-8use /DT s silicon diode sensors, which allow temperatures from 4 K to ∼400 K to be measured. These sensor are nonlinear and have excellent resolution at low temperatures. Samples or sample cans must be mounted on a male M6 thread, which is often a Vanadium can. These probes can be fitted with a copper bracket to provide temperature control at both the top and bottom of the sample.

Note: These probes are supplied by default and therefore do not have a ancillary code.

Figure 6.14: Photo of Standard CF7, CF8 probe.

6.3.2 CF-7, CF-8 Cryofurnace full-range probe TL-1 & Tl-3

Ancillary code	TL-1 & TL-3
Temperature range	4 K to $\sim$800 K
Thermometry	Rhodium iron sensor
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

The full-range sample positioning probes for top loading cryofurnaces are essentially the same as the standard probes, except that the DT-670 silicon diode temperature sensors (maximum temperature 400 K) are replaced with Rhodium iron sensors, which allow temperatures from 4 K to $\sim$800 K to be accessed on a single sample probe. Samples or sample cans must be mounted on a male M6 thread, which is often a Vanadium can. These probes can be fitted with a copper bracket to provide temperature control at both the top and bottom of the sample.

Note: These probes should only be used for experiments requiring temperatures above 400 K, as the Rhodium iron sensor sensors are fragile and less accurate than DT-670 silicon diodes.

Figure 6.15: Photo of TL-1.

6.3.3 CF-7, CF-8 Cryofurnace gas delivery probe TL-5 & TL-9

Ancillary code	TL-5 & TL-9
Temperature range	4 K to 300 K
Thermometry	DT-670 silicon diode
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace GD-1: gas delivery system VD-1: vapour delivery system
Project folder	BIP-0270

These gas delivery probes are usually used in conjunction with the Hiden Isochema IMI-HTP gas sorption/desorption system GD-1 to allow for gas delivery experiments in the top loading cryofurnaces CF-7 & CF-8. Samples are typically contained in Vanadium can cans sealed with indium wire. These probes are assembled from two separate parts to allow sample mounting in the helium glove box. A removable aluminium radiation shield with temperature control for the bottom of the sample cell can be fitted.

Figure 6.16: Photo of TL-5.

6.3.4 CF-7, CF-8 Cryofurnace optical spectroscopy probe

Ancillary code	none
Temperature range	4 K to ∼400 K
Thermometry	DT-670 silicon diode
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

The optical spectroscopy probe incorporates a fibre optic reflection sensor in the central tube of the sample stick. The reflection sensor comprises six excitation fibres surrounding a sensing optical fibre. The stick can used in conjunction with the Ocean Optics spectrometers and light sources. *In situ* optical spectroscopy can be carried out during a neutron experiment. NIR and UV/Vis fibres, vacuum feedthroughs and spectrometers are available Please discuss your requirement with sample environment well before your experiment.

Note: This probe does not have a booking code and needs to be requested from sample environment prior to your experiment.

Figure 6.17: Optical probe with NIR spectrometer on Wombat

6.3.5 CF-11, CF-12 Circulating cryostat standard probe

Ancillary code	TL-1 & TL-3
Temperature range	4 K to ∼800 K
Thermometry	Rhodium iron sensor
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

Need to insert other details of probes for other CF' loading cryofurnaces and magnets. including carbon fibre options.

6.3.6 CF-11, CF-12 Circulating cryostat full-range probe

Ancillary code	TL-1 & TL-3
Temperature range	4 K to $\sim$800 K
Thermometry	Rhodium iron sensor
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

Need to insert other details of probes for other CF' loading cryofurnaces and magnets. including carbon fibre options.

6.3.7 CF-11, CF-12 Circulating cryostat gas delivery probe

Ancillary code	TL-1 & TL-3
Temperature range	4 K to ∼800 K
Thermometry	DT-670 silicon diode
Sample containers	Vanadium can: VCR flange
Complementary ancillaries	GD-1: Gas dosing & VD-1: Vapour delivery

Insulated and heated capillary allows deliver of gas or vapour to sample *in situ*.

Figure 6.18: Version 1 gas delivery stick

6.3.8 CF-11, CF-12 Circulating cryostat light probe

Ancillary code	
Temperature range	4 K to ∼800 K
Thermometry	Rhodium iron sensor
Sample containers	Annular aluminium/quartz can
Complementary ancillaries	CF-11 & CF-12:

The Light stick allows UV-Visible spectrum light to conveyed via fibre optics to the sample space. The light sources available at ACNS are listed under OF-1. In other respects the sample probe has the same parameters as the standard CF-11, CF-12 sample stick..

Figure 6.19: Fibre connection to sample probe and disassembled light stick cell.

6.3.9 CF-13, CF-14 Circulating cryostat standard probe

Ancillary code	TL-1 & TL-3
Temperature range	4 K to $\sim$800 K
Thermometry	Rhodium iron sensor
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

Need to insert other details of probes for other CF' loading cryofurnaces and magnets. including carbon fibre options.

6.3.10 CF-13,CF-14 Circulating cryostat full-range probe

Ancillary code	TL-1 & TL-3
Temperature range	4 K to ∼800 K
Thermometry	Rhodium iron sensor
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

Need to insert other details of probes for other CF' loading cryofurnaces and magnets. including carbon fibre options.

6.3.11 Cryostat carbon-fibre probe

Ancillary code	TL-1 & TL-3
Temperature range	4 K to ∼450 K
Thermometry	DT-670 silicon diode
Sample containers	Vanadium can
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace

Carbon fibre probes are available in place of standard probes for CF11, CF12, CF13 and CF14, The carbon fibre shaft improves the cool-down time over a standard thin walled stainless shaft.

6.3.12 F-1 Furnace gas delivery probe HTS-1

Ancillary code	HTS-1
Temperature range	300 K to 1900 K
Thermometry	C-type thermocouple K-type thermocouple
Complementary ancillaries	F-1: high-temperature vacuum furnace
Project folder	BIP-0266

These probes are used to allow gas delivery experiments to be performed for samples in the high-temperature vacuum furnace F-1. A range of outer tube materials to hold the sample (alumina, quartz, stainless steel, platinum) and thermocouples (C-type, K-type) can be used, so please discuss your experimental requirements with the Sample Environment group.

Figure 6.20: Photo of HTS-1.

6.4 Sample cells

6.4.1 Vanadium sample can

Maximum temperature	800 K
Maximum pressure	30 bar at 100°C
Complementary ancillaries	CF-1 & CF-3: bottom-loading cryofurnace CF-2: bottom-loading cryofurnace CF-5: bottom-loading cryostat CF-6: bottom-loading cryostat CF-7 & CF-8: top-loading cryofurnace CF-10: top-loading cryostat CF-11 & CF-12: top-loading cryofurnace OC-1: orange cryostat F-1: high temperature vacuum furnace F-5: low temperature vacuum furnace TL-1 & TL-3: top loading cyrofurnace full-range probe TL-5 & TL-9: top loading cyrofurnace gas delivery probe SC-4: pick-and-place robot SC-5 & SC-10: six-axis robot
Manufacturer website	MTI Albany

We provide vanadium sample cans of two different sizes. The larger cans have a diameter of 3/8", are 2.5" long and have a volume of 4 mL, while the smaller cans have a diameter of 1/4" and a volume of 1.5 mL.

The can can either be sealed with a lid with an M6 male thread for mounting on cryostats and furnaces; or onto a flange with a Swagelok VCR gasket, lead or copper gaskets for gas delivery using TL-5 & TL-9; we also have some flange-less cans, and some lids for use with the sample changing robots SC-4 and SC-5 & SC-10.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

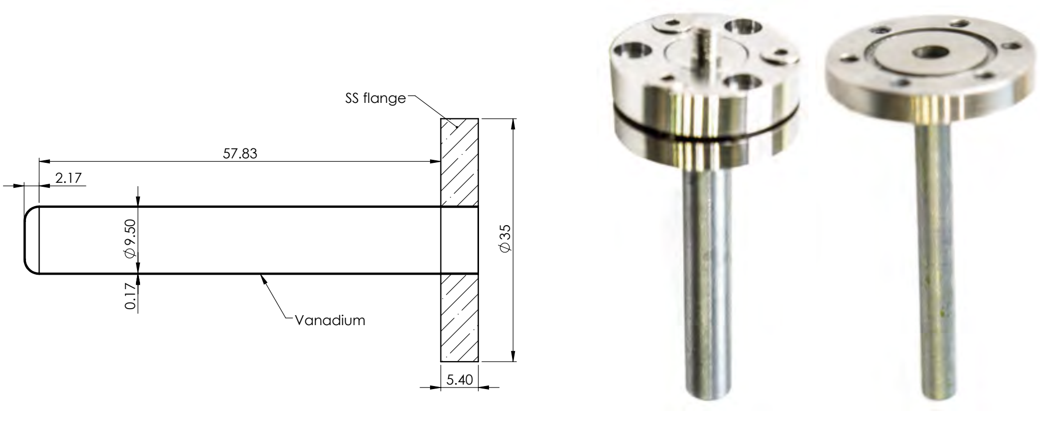


Figure 6.21: Schematic of a flanged 3/8" vanadium can (left), and photos of a 3/8" (middle) and 1/4" (right) flanged vanadium can.

6.4.2 Aluminium annular sample can

Maximum temperature	600 K
Maximum pressure	6 bar at 300 K
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace CF-10: top-loading cryostat CF-11 & CF-12: top-loading cryofurnace AVM-1: asymmetrical vertical magnet OC-1: orange cryostat

Our aluminium annular cans are assembled from an outer cup, and inner cup and a lid, and are sealed with PTFE or copper gaskets. Different path lengths for the sample of 0.1 mm, 0.2 mm, 0.5 mm, 1 mm and 2 mm are possible by using the corresponding inner cup, which have a wall thickness of 0.25 mm. Cans with a narrow flange diameter are available for use on CF-10 and AVM-1. Gas delivery cans are also available for use with GD-1 and VD-1 on CF-11 & CF-12.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

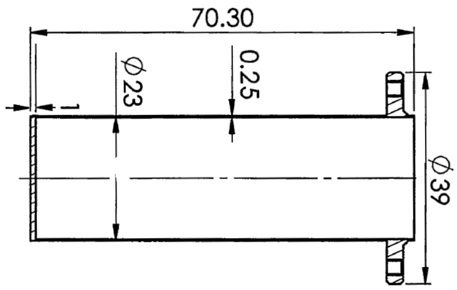


Figure 6.22: Schematic of the large-flanged outer cup of an annular aluminium can.

Figure 6.23: Photo of (from left to right) an aluminium annular can outer cup, inner cup, lid, and an assembled a gas delivery can.

6.4.3 Aluminium flat sample can

Maximum temperature	800 K
Maximum pressure	6 bar at 300 K
Complementary ancillaries	CF-7 & CF-8: top-loading cryofurnace CF-11 & CF-12: top-loading cryofurnace

Aluminium sample cans in a flat planar geometry are available with a range of path lengths (0.5 mm, 1 mm, 2 mm). There are also slight variations in the size of the sample space (29 × 50 mm or 24 × 47 mm). These cans are sealed with indium. Please contact the Sample Environment group if you have specific requirements.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

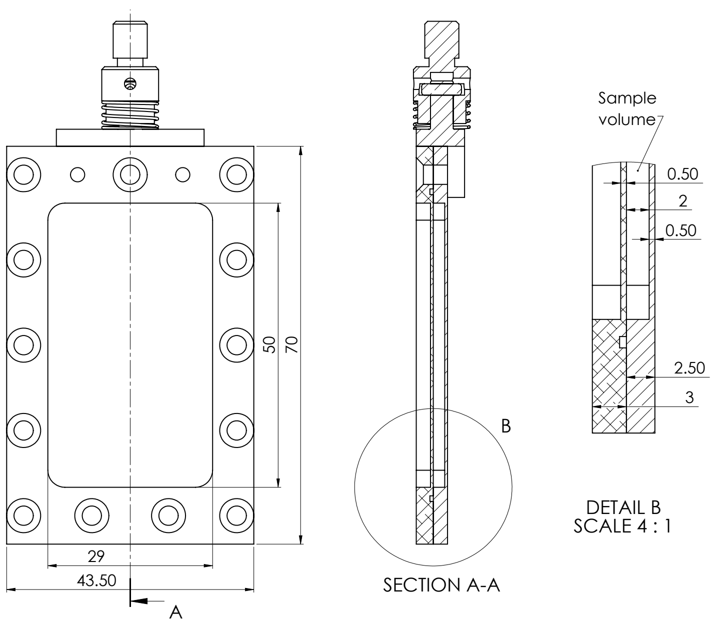


Figure 6.24: Schematic of a flat aluminium cell with a 2 mm path length.

Figure 6.25: Photo of some flat aluminium can parts.

6.4.4 Stainless steel can for deuterated samples

Maximum temperature	800 K
Maximum pressure	44 bar at 200°C
Project folder	BIP-0215

These stainless steel cells are used for deuterated samples. There are two different designs which are 90 mm long, one with a diameter of 15 mm (wall thickness 0.7 mm, volume 11.5 mL) and one of diameter 10.2 mm (wall thickness 0.5 mm, volume 7.1 mL). They are sealed with Swagelok VCR fittings.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

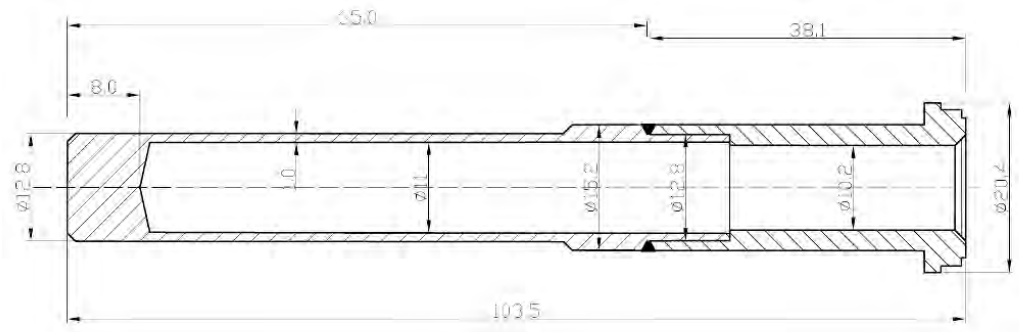


Figure 6.26: Schematic of the stainless steel cans for deuterated samples.

Figure 6.27: Photo of some stainless steel cans for deuterated samples.

6.4.5 Aluminium can for clathrate experiments

Maximum temperature	300 K
Maximum pressure	100 bar at 25°C
Project folder	BIP-0129

These aluminium methane pressure cells have a volume of 4.2 mL, and are used for gas clathrate experiments. They are 10 mm in diameter and 54 mm long. They are sealed with a cone fitting.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

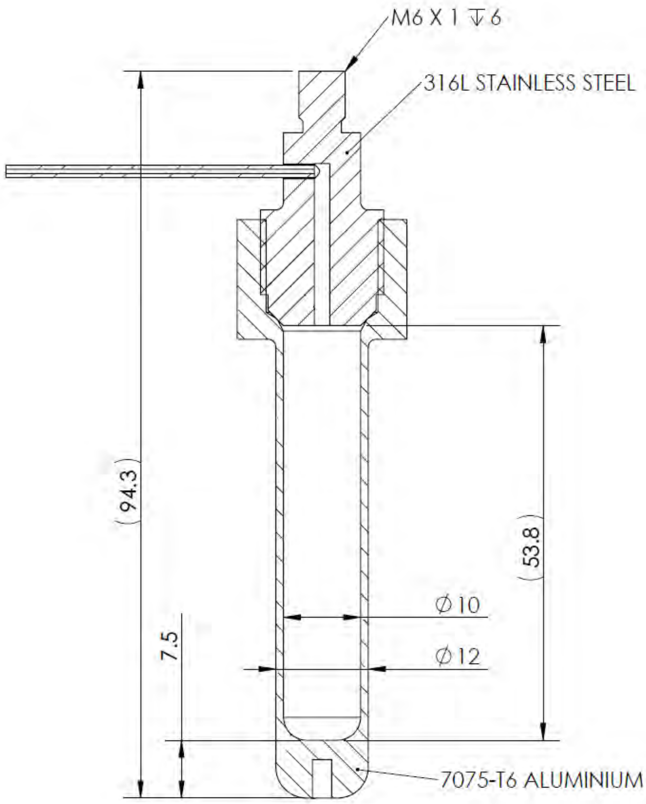


Figure 6.28: Schematic of an aluminium can for clathrate samples.

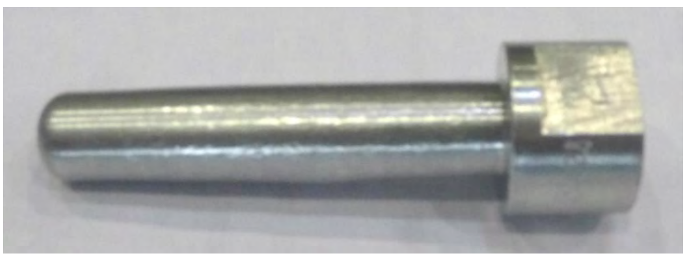


Figure 6.29: Photo of an aluminium can for clathrate samples.

6.4.6 100 bar Stainless steel furnace can

Maximum temperature	800 K
Maximum pressure	100 bar at 300°C (derated at higher temperatures)
Complementary ancillaries	F-5: low temperature vacuum furnace CF-11 & CF-12: top-loading cryofurnace
Project folder	BIP-0138, BIP-0170

These stainless steel cans for gas pressure experiments at elevated temperatures are available in two sizes. The larger cans have a ϕ10 mm bore, a volume of 8.8 mL and a neutron window of 0.89 mm wall thickness. The smaller cans are essentially the same, except that have a ϕ7 mm bore, a volume of 2.5 mL and a 0.51 mm wall thickness.

These cans can either be plumbed into a gas line with Swagelok VCR fittings for *in situ* gas delivery in the low temperature furnace F-5, or attached to a lid with an M6 threaded stub to be mounted without *in situ* gas loading on CF-11 & CF-12. They were designed for use with hydrogen and deuterium, and other gases may possibly be used. The pressure rating of these cans is temperature dependent, and while they are rated to 100 bar at 300°C they can be used at higher temperatures with a corresponding reduction in maximum pressure. Please discuss your requirements with the Sample Environment group prior to use.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

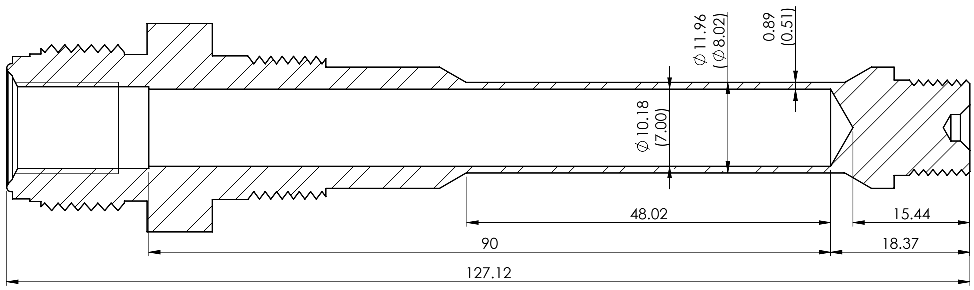


Figure 6.30: Schematic of the 10 mm stainless steel cans. Note that dimensions which are different for the 7 mm cans are shown in parentheses.

Figure 6.31: Photo of 7 mm (top) and 10 mm (bottom) stainless steel cans for high pressure furnace experiments.

6.4.7 Bottom-loading cryostat exchange gas can

Maximum temperature	300 K
Thermometry	DT-670 silicon diode
Maximum pressure	1 bar at 300 K
Complementary ancillaries	CF-1 & CF-3: bottom-loading cryofurnace

These aluminium cans allow large samples to be loaded into a sealed cell containing helium exchange gas, which are mounted on bottom-loading cryofurnaces and can be used at temperatures below ambient. They are suitable for holding single crystal samples.

The can contains a DT-670 silicon diode mounted on the inside of the lid, while temperature control is achieved on the end of the cold stage of the cryostat.

Note: These cans are not considered consumables, and must be cleaned and returned after use.

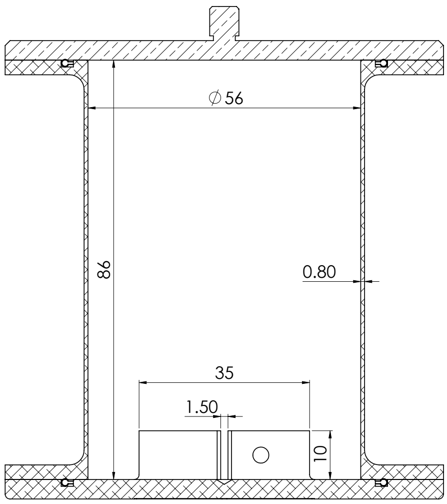


Figure 6.32: Schematic of exchange gas can for bottom-loading cryofurnaces.

Figure 6.33: Photo of exchange gas can for bottom-loading cryofurnaces.

6.4.8 Dilution sample can

Complementary ancillaries DL-1 : Dilution insert.

These copper can are formed from standards swagelok parts and specially machined parts. These cans are always filled from the bottom (ie do not open from the threaded end once sealed,). Always use an appropriate thermal transfer medium with your sample such as d-propanol to ensure the sample is in thermal contact with the can. The tube should never be subjected to torsional forces always use two spanner to open the can. *Note:* These cans are not considered consumables, and must be cleaned and returned after use.

6.4.9 Selecting a cell for SANS instruments

We have a variety of cells for use on Quokka, Bilby and Kookaburra. They vary chiefly in the path length and/or the volume of the cell. The choice of cell will be affected by:

- The physical properties of the sample, and how difficult it is to present a homogeneous sample of uniform thickness to the neutron beam.
- The physical conditions under which the measurement may be made, for example some cells are more suitable for high temperature measurements.
- The required sample thickness. While thicker samples will minimise data collection time (more scattering), the value of this approach may be offset by the onset of multiple scattering effects.
- The amount of sample you have and/or dilution of solutions which may be necessary to obtain scattering due to individual particles shape (form factor). More dilute samples may be used with longer path-lengths.
- The ease of removing the sample from the cell (cleaning). Cells are quite expensive and we do not consider them to be consumables.

Below are some typical equilibration times for calculations of beam-time to achieve a temperature stability of ± 0.5°C.

Temperature change	*Equilibration time*
25 °C to 5 °C	60 minutes
5 °C to 25 °C	50 minutes
25 °C to 50 °C	75 minutes
50 °C to 25 °C	80 minutes
25 °C to 80 °C	85 minutes
80 °C to 25 °C	90 minutes
25 °C to -10 °C	85 minutes (± 1 °C)
-10 °C to 25 °C	90 minutes

Some generalisations which may help you plan beam time are:

- Smaller steps take less time to come to equilibrium
- More accurate temperature control takes longer
- Heating or cooling a sample takes longer at temperatures further from ambient

6.4.10 Hellma cell

Complementary ancillaries	SC-2: twenty position sample changer SC-7: five-position rotating sample changer SC-8: twelve-position Peltier sample changer SC-9: six-position rotating sample changer
Manufacturer website	Hellma Analytics

The standard cells suitable for most samples encountered on QUOKKA and BILBY are Hellma type 120 "banjo" cells of a variety of sample path lengths. These cells are suitable for low-viscosity liquids and suspensions. They are mounted in the 20-position sample changers in standard aluminium blocks.

Sample path length	*Cell volume*
1 mm	0.28 mL
2 mm	0.56 mL
5 mm	1.4 mL

Note: These cells are not considered consumables, and must be cleaned and returned after use.

Figure 6.34: Photo of Hellma cell.

6.4.11 Demountable cell large

Complementary ancillaries	SC-1: ten-position sample changer SC-2: twenty-position sample changer
Project folder	BIP-0041

Standard demountable cell blocks are suitable for Solids, (sheets and powder)and high viscosity liquids, suspensions and pastes. These cells consist of a gap for a sample between two quartz windows, and are sealed with o-rings. Cells are available in a variety of path lengths, where PTFE spacers are used for gaps below 1 mm and also allow small samples to be confined to the beam centre.
Modified demountable cells are available fitted with inlet and outlet tubing to allow flow cell experiments to be carried out. Talk to sample environment about a suitable peristaltic pump for your experiment.

Sample path length	*Cell volume*
0.1 mm	0.06 mL
0.2 mm	0.12 mL
0.5 mm	0.3 mL
1 mm	0.6 mL
2 mm	1.2 mL
3 mm	1.8 mL
5 mm	2.9 mL

Note: These cells are not considered consumables, and must be cleaned and returned after use.

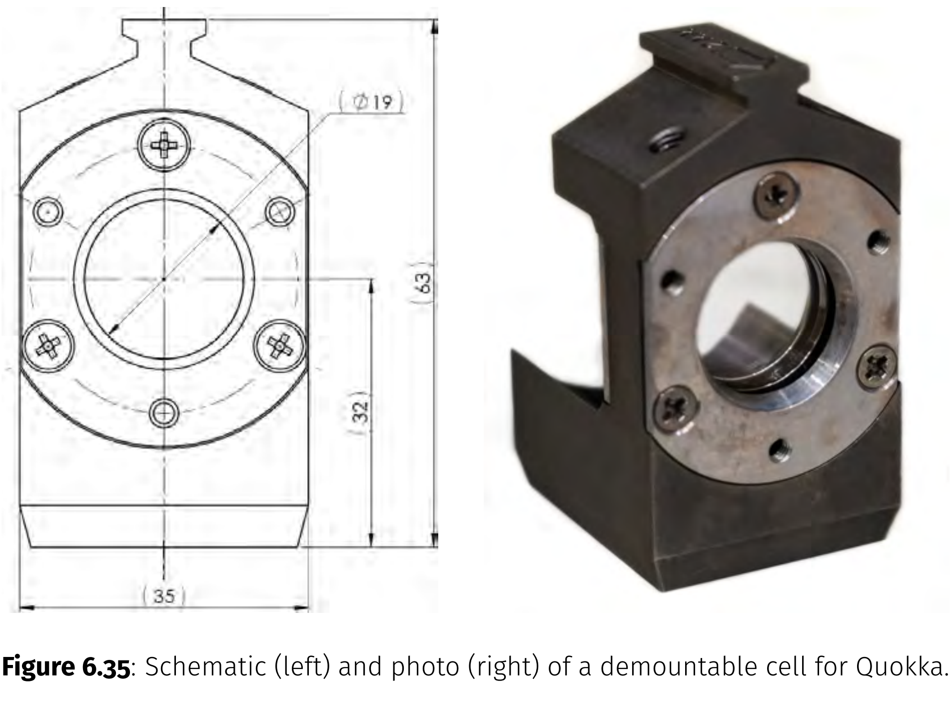


Figure 6.35: Schematic (left) and photo (right) of a demountable cell for Quokka.

6.4.12 Demountable cell Kookaburra

Complementary ancillaries	SC-6: five position ambient sample changer SC-9: six-position rotating sample changer
Project folder	BIP-0041

Kookaburra sample cells use 50mm quartz windows to match the neutron beam dimensions. KOOKABURRA has a relatively low neutron flux due to its exceptionally high collimation hence as much of the beam as possible must be utilised so collection times do not become unreasonable long. Hence more sample is required for a given path length. Occasionally this means it is necessary to use an adaptor so the slim demountable cells can used in place of the circular Kookaburracells. The housing may be rectangular, for use with SC-6or round, for SC-8.

Note: These cells are not considered consumables, and must be cleaned and returned after use.

6.4.13 Solid-liquid cell SL-1 & SL-2

Ancillary code	SL-1
Compatible instruments	Platypus
Project folder	BIP-0289

A 100 mm or 50mm diameter and 40 x 80mm rectangular silicon wafer cells are available for use with liquid reflectometry experiments. Electrochemical cells are also available both with and without provision for reference junctions. If wishing to do electrochemical experiments please specify in your proposal whether you with to use 2, 3 electrode system. When booking the solid liquid cells a quaternary HPLC pump and remote switch valve is also provided. These provide sample/solvent flow, concentration control and cell selection. Up to 3 sample cells may be mounted at once on Platypus.

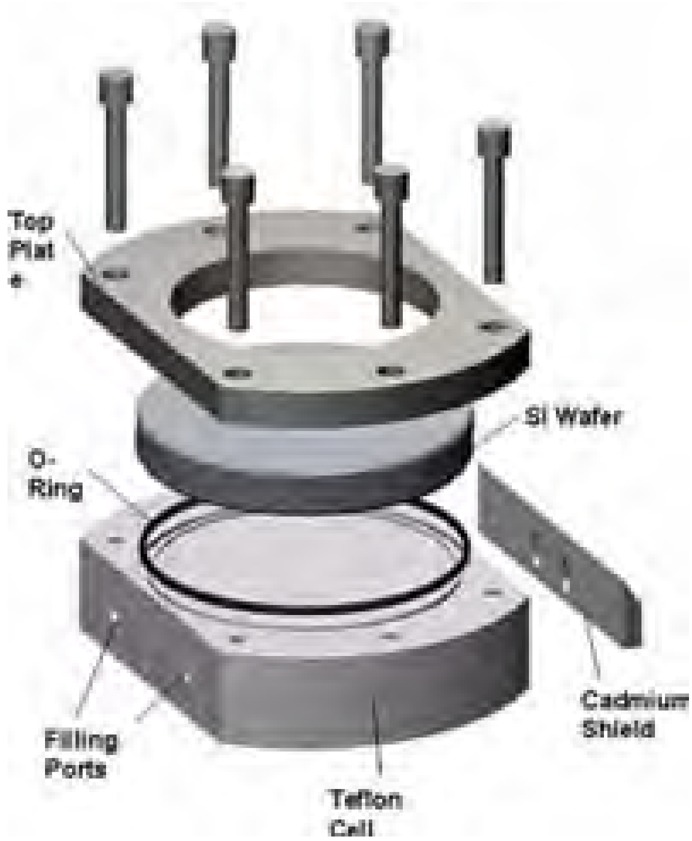


Figure 6.36: Photo of SL-1.

6.4.14 Vapour chamber DEC-1

Ancillary code	DEC-1
Compatible instruments	Platypus, Quokka, Bilby, Kookaburra
SICS control	Lakeshore 336/340 single heater and temperature sensor And/or Silicone oil or Glycol bath
Equipment Number	220213

Commercial cubic chamber with an ISO CF100 flanges on each face. Two faces are fitted with 100mm quart windows, One incorporates a 50 pin electrical lead-through and two 1/4 inch liquid leadthroughs and one flange is fitted with a vacuum fitting. The remaining flanges are blank but could be modified to provided other leadthroughs into the chamber. The sample stage is an aluminium block that incorporates a heater cartridge of the aluminium tubing for temperature control. A PT1000 sensor is provided to monitor the sample temperature. Two blocks are available to suit either reflectometry or scattering experiments. This chamber can be evacuated to below 10^{-9} mbar making it suitable for use with the Static version of VD-1. The chamber can be used whenever a sample requires a controlled atmosphere or vacuum on the scattering instruments.

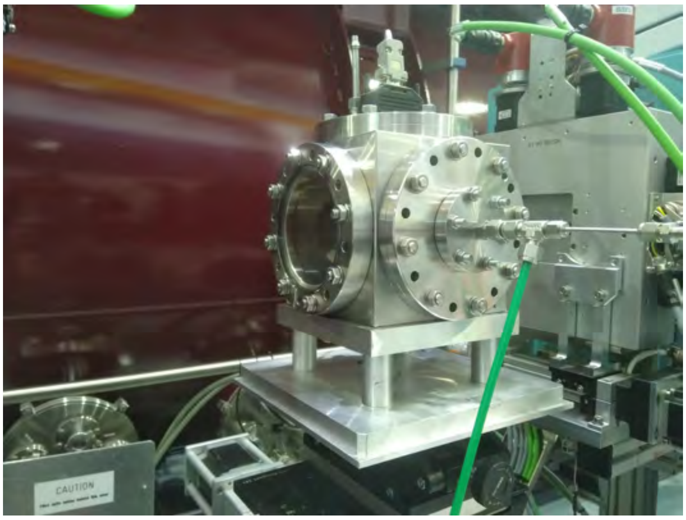


Figure 6.37: DEC-1 mounted on Spatz in flow mode.

Chapter 7

Neutron Instruments

This section provides a brief description of the neutron beam instruments including a list of compatible sample environment equipment. Items in bold type are routinely used on the listed instrument. Please consider these items of sample environment first. Selecting other items may require equipment changes at the start of your beam time, the extra time for this should be included in your proposal. If the listed equipment does not suit your needs please contact sample environment. There may be a solution using existing equipment that we haven't considered when compiling this list. Sample environment equipment at ACNS is not assigned to particular instruments. This allows some novel solutions to be tried out. Included is some information about the sample stage and enclosure of the instrument. If more information is required concerning the neutron instrument please contact your Instrument Scientist.

7.1 Diffraction Instruments

7.1.1 Echidna: High-resolution powder diffractometer

Instrument Type	Diffractometer
Tilt stage	2 x +/-20 °
XY Stage	2 x +/- 20 mm
Z-Stage	none
Rotation	+/-180 °
Maximum load with tilt stage	150 kg
Maximum load rotation only	500 kg
Stage to beam distance	255mm
Beam Line	TG1

Echidna is a high resolution neutron powder diffraction instrument. It compliments Wombat by providing higher resolution while sacrificing acquisition speed. The SE area is confined by the horseshoe shaped detector and the neutron guide. The sample stage and detector move on air pads on a polished granite floor. The sample stage and detector move as one unit on an arc centered on the monochromator axis.
Either robotic sample changer can be used. However SC4 is routinely installed. SC5 is mounted on a narrow trolley designed to be used when the position of the detector prevents SC4 fitting between the sample stage and neutron guide.
If tilt and translation is not required these stages can be removed to give a larger base to beam distance. This is required for CF-10, CF-11, CF-12 and AVM-1. The services panel for Echidna is on the far wall of the enclosure and hence ancillary equipment may be position behind the detector and generally longer leads and hoses are required for Echidna.

Top-loading cryofurnaces CF-7 & CF-8
High temperature vacuum furnace F-1
Robotic sample changer SC-4
Bottom-loading cryofurnace CF-1 & CF-3
Bottom-loading cryostat CF-5
Top-loading cryostat CF-10
Top-loading cryofurnace CF-11 & CF-12
Orange cryostat OC-1
Low temperature vacuum furnace F-5
Environmental chamber F-6
Dilution refrigerator insert DL-1
^{3}He one-shot insert OS-1
12 T vertical magnet AVM-1

10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Eulerian cradle EC-1
Paris-Edinburgh cell P-3

Full-range probe TL-1 & TL-3
Gas delivery probe TL-5 & TL-9
F-1 gas delivery probe HTS-1

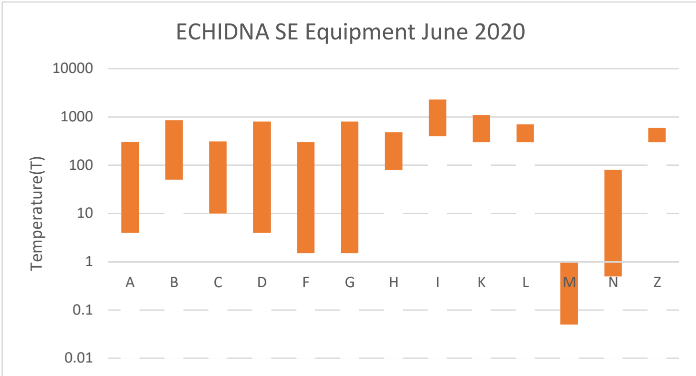


Figure 7.1: Temperature range for equipment compatible with Echidna

7.1.2 Wombat: High-intensity powder diffractometer

Instrument Type	Diffractometer
Tilt stage	2 x +/-20 °
XY Stage	2 x +/- 20 mm
Z-Stage	none
Rotation	+/-180 °
Maximum load with tilt stage	150 kg
Maximum load rotation only	500 kg
Stage to beam distance	255mm
Beam Line	TG1

Wombat is a high flux neutron powder diffraction instrument and incorporates an oscillating collimator between the sample and detector. It compliments Echidna by providing higher speed acquisition sacrificing resolution. The SE area is confined by the horseshoe shaped detector and the neutron guide. The sample stage and detector move on air pads on a polished granite floor. The sample stage and detector move as one unit on an arc centred on the monochromator axis.
If tilt and translation is not required these stages can be removed to give a larger base to beam distance. This is required for CF-10, CF-11, CF-12 and AVM-1. A safety interlock is provided for interlocking hazardous sample environment equipment to the enclosure door. A local exhaust is provided for exhaust of gas and vapours from the enclosure.

Top-loading cryofurnaces CF-7 & CF-8
High temperature vacuum furnace F-1
Paris-Edinburgh cell P-3
Bottom-loading cryofurnaces CF-1 & CF-3
Bottom-loading cryostat CF-5
Top-loading cryostat CF-10
Top-loading cryofurnaces CF-11 & CF-12
Orange cryostat OC-1
Low temperature vacuum furnace F-5
Environmental chamber F-6
Dilution refrigerator insert DL-1
^{3}He refrigerator insert OS-1
12 T vertical magnet AVM-1
1 T horizontal magnet HM-3

10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Eulerian cradle EC-1
6 axis Robot SC-5 & SC-10
Rheometer RH-1

Full-range probe TL-1 & TL-3
Gas delivery probe TL-5 & TL-9
F-1 gas delivery probe HTS-1

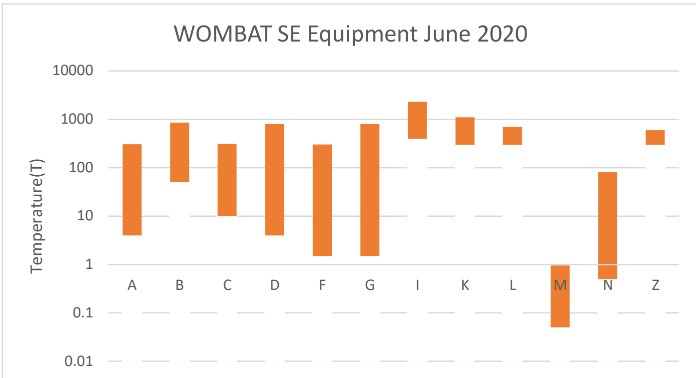


Figure 7.2: Temperature range for equipment compatible with Wombat

7.1.3 Koala: Laue diffractometer

Instrument Type	Single Crystal Diffractometer
Tilt stage	none
XY Stage	2
Z-Stage	fine sample height adjustment
Rotation	+/- 180 °
Maximum load	50 kg TBC
Stage to beam distance	not applicable
Beam Line	TG3

Koala is a Quasi-Laue diffractometer. A single crystal sample is exposed to the neutron

beam and the diffracted beams expose points on a sensitive film on the inside of the cylinder surrounding the sample. After exposure the cylinder (referred to as the drum) is rotated while the image is collected in a spiral and the film is erased. The spiral data is then reconstructed into the original image. Do not attempt to enter the the enclosure if the beam is off and the drum is rotating as this may halt the collection process. Samples for Koala are generally of the order of a few millimetres. These small sample are mounted on an aluminium pin and the height is set using a calibrated video camera mounted on the side of the instrument.
Sample environment is mounted from the top of the shielding on the neutron guide a ladder is provided. This access is not for user access. Always ensure this is locked into the guide before climbing. The sensitive film is sensitive to both neutron and light so be sure there are no light path from the sample environment into the drum. This can happen if any of the fixing screws are left out as the holes in the mounting plate go through to the interior of the drum. The sensitive film is also easily damaged and a guard is provided to protect it whenever the drum is opened for sample and equipment changes.

Bottom-loading cryofurnace CF-2 **Cryostream** Cobra

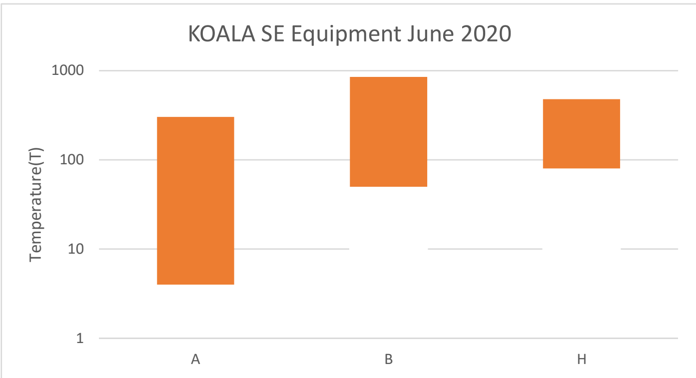


Figure 7.3: Temperature range for equipment compatible with Koala

7.1.4 Joey: Neutron laue camera for single-crystal alignment

Instrument Type	Crystal alignment Laue Camera
Tilt stage	2
XY Stage	none
Z-Stage	none
Rotation	+/- 180 °
Maximum load	10 kg
Stage to beam distance	Not applicable
Beam Line	TG3

Joey is a Laue camera used for aligning single crystal samples and determining the quality of samples. It can be helpful to use Joey to align your samples prior to mounting in a cryostat for experiments on the neutron diffraction instruments. It is very useful for co-aligning a number of single crystals to form a mosaic large enough for a neutron measurement. Generally no sample equipment is required for experiments on Joey. Experiments using Joey have to be coordinated with Koala. To allow sufficient neutron flux for Joey to operate Koala needs to be in a particular configuration. Contact your Instrument scientist for more information on Joey.

Ambient temperature mount

7.1.5 Kowari: Strain scanner

Instrument Type	Strain Diffractometer
Tilt stage	none
XY Stage	2 x +/-250mm
Z-Stage	700mm
Rotation	+/- 185 °
Maximum load	1000 kg
Stage to beam distance	0 to 700mm
Beam Line	TG3

Kowari is a strain diffractometer. A small detector is mounted on the perimeter of the sample stage and can be rotated on an arc centred on the sample table. Both the detector and table are mounted on air pads and can rotate as a unit about the central axis of the monochromator on the beam line. To measure strain a Bragg peak is selected and the variation in the diffracted angle for an unstrained sample is used to determine the strain in the sample. Kowari samples can be very large and heavy hence manual handling assessments may need to be made. The instrument enclosure is large and mostly unobstructed for mounting sample environment equipment. An interlock is provided for the safe use of SC5.

Ambient temperature mount	10 kV electric field system EF-1
Load frame P-2	Potentiostat/galvanostat PG-1
Bottom-loading cryostat CF-5	Battery tester BT-1
Environmental chamber F-6	
Bottom-loading cryostat CF-5	Eulerian cradle EC-3
	6 axis Robot SC-5 & SC-10

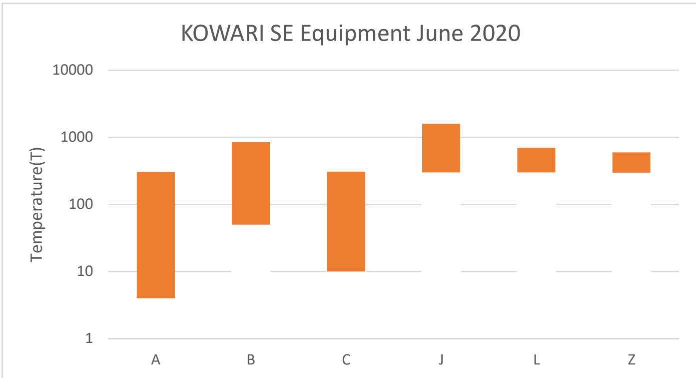


Figure 7.4: Temperature range for equipment compatible with Kowari

7.2 Spectrometry Instruments

7.2.1 Sika: Cold triple-axis spectrometer

Instrument Type	Triple axis spectrometer
Tilt stage	2
XY Stage	2
Z-Stage	1
Rotation	+/- 180 °
Maximum load	150 kg TBC
Stage to beam distance	adjustable around 255mm
Beam Line	CB1

Sika is a triple axis spectrometer and differs from Taipan in that is mounted on a cold neutron beam. The sample and detector stages are mounted on air pads that glide over the polished granite floor. The detector moves on an arc centred on the sample stage and the sample stage moves on an arc centred on the monochromator. Be aware of the range of movement of the parts of spectrometer during an experiment to be sure no equipment will hinder the instrument. Experiments will often require large range of rotation and tilt and sufficient slack lead is required to avoid damage to the sample environment equipment. A practice run on the range of movement of the sample environment equipment is recommended prior to the start of the experiment. Mounting of AVM-1 CF-10, CF-11 or CF-12 requires the removal of the tilt stages. Samples often remain active when measured on Sika always check the sample environment equipment for activity before removal from the sample stage. This instrument is sited in the reactor beam hall hence experiments invovling flammable gases have to be cleared with Reactor Operations before the experiment takes place.

Top-loading cryofurnaces CF-1 & CF-3	10 kV electric field system EF-1
Orange cryostat OC-1	Potentiostat/galvanostat PG-1
High temperature vacuum furnace F-1	Battery tester BT-1
Bottom-loading cryofurnaces CF-7 & CF-8	Gas delivery system GD-1
Top-loading cryostat CF-10	Gas spectrometer GS-1
Top-loading cryofurnaces CF-11 & CF-12	Vapour delivery system VD-1
Paris-Edinburgh cell P-3	Eulerian cradle EC-1
Low temperature vacuum furnace F-5	6 axis Robot SC-5 & SC-10
Environmental chamber F-6	
Dilution refrigerator insert DL-1	
^{3}He refrigerator insert OS-1	Full-range probe TL-1 & TL-3
12 T vertical magnet AVM-1	Gas delivery probe TL-5 & TL-9
	F-1 gas delivery probe HTS-1

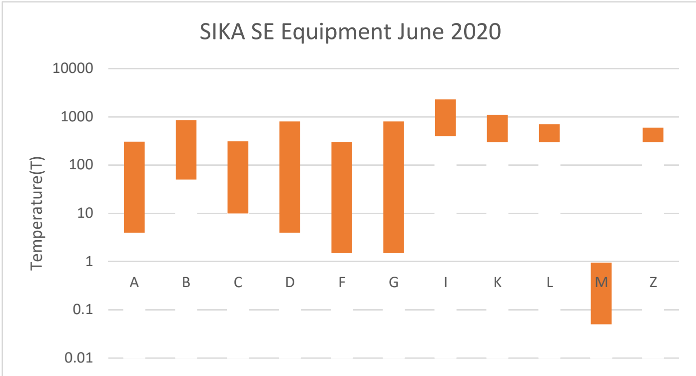


Figure 7.5: Temperature range for equipment compatible with Sika

7.2.2 Taipan: Thermal triple-axis spectrometer

Instrument Type	Triple Axis Spectrometer
Tilt stage	2 x +/-20 °
XY Stage	2 x +/- 20 mm
Z-Stage	none
Rotation	+/-180 °
Maximum load with tilt stage	150 kg
Maximum load rotation only	500 kg
Stage to beam distance	255mm
Beam Line	TB1

Taipan is a triple axis spectrometer with sample stage and detector mounted on separate movable stages. These stages are mounted on air pads that glide over the polished granite floor. The detector moves on an arc centred on the sample stage and the sample stage moves on an arc centred on the monochromator. Be aware of the range of movement of the parts of spectrometer during an experiment to be sure no equipment will hinder the instrument. Experiments will often require large range of rotation and tilt and sufficient slack lead is required to avoid damage to the sample environment equipment. A practice run on the range of movement of the sample environment equipment is recommended prior to the start of the experiment. Mounting of AVM-1 CF-10, CF-11 or CF-12 requires the removal of the tilt stages. Samples often remain active when measured on Taipan always check the sample environment equipment for activity before removal from the sample stage.This instrument is sited in the reactor beam hall hence experiments involving flammable gases have to be cleared with Reactor Operations before the experiment takes place.

Top-loading cryofurnaces CF-1 & CF-3
Orange cryostat OC-1
Paris-Edinburgh cell P-3
Bottom-loading cryofurnaces CF-7 & CF-8
Bottom-loading cryostat CF-5
Top-loading cryostat CF-10
Top-loading cryofurnaces CF-11 & CF-12
High temperature vacuum furnace F-1
Low temperature vacuum furnace F-5
Environmental chamber F-6
Dilution refrigerator insert DL-1
^{3}He refrigerator insert OS-1
12 T vertical magnet AVM-1

10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Eulerian cradle EC-1
6 axis Robot SC-5 & SC-10

Full-range probe TL-1 & TL-3
Gas delivery probe TL-5 & TL-9
F-1 gas delivery probe HTS-1

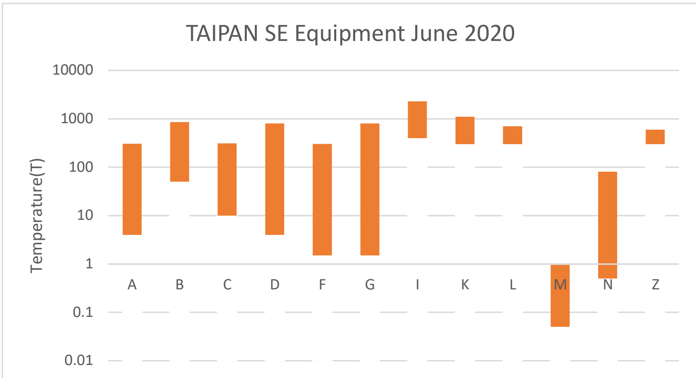


Figure 7.6: Temperature range for equipment compatible with Taipan

7.2.3 Taipan: Be Filter

Instrument Type	Be Filter Spectrometer
Tilt stage	0
XY Stage	0
Z-Stage	none
Rotation	0
Maximum load	100 kg TBC
Stage to beam distance	255mm
Beam Line	TB1

Taipan: Be Filteris a band-pass filter spectrometer. It is mounted on the Taipan monochromator and hence requires the removal of the Taipan sample stage and detector. The sample environment area for Taipan: Be Filteris severely constrained by the shield required for its operations. This is reflected in the how few pieces of sample environment is available for use. Only sample rotation is available with CF-11 or CF-12 the sample stage of Taipan: Be Filterhas no translation or tilt stages. Powder or liquid samples are the primary type of samples expected for use on Taipan: Be Filter. The cryostats are mounted from above while the PE cell has to be swung in through the side door of the instrument. Taipan: Be Filtermay move during an experiment so its is important to be sure sample environment leads are long enough for the full range required for the experiment. This instrument is sited in the reactor beam hall hence experiments involving flammable gases have to be cleared with Reactor Operations before the experiment takes place. Sample rotation is available with CF-11 or CF-12.

Note: Prior to mounting the PE Cell; the frame has to be rotated on its stand. Positions for Wombat and Taipan: Be Filterare marked.

Top-loading cryofurnaces CF-7 & CF-8
Top-loading cryofurnaces CF-11 & CF-12
Paris-Edinburgh cell P-3

10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Full-range probe TL-1 & TL-3
Gas delivery probe TL-5 & TL-9

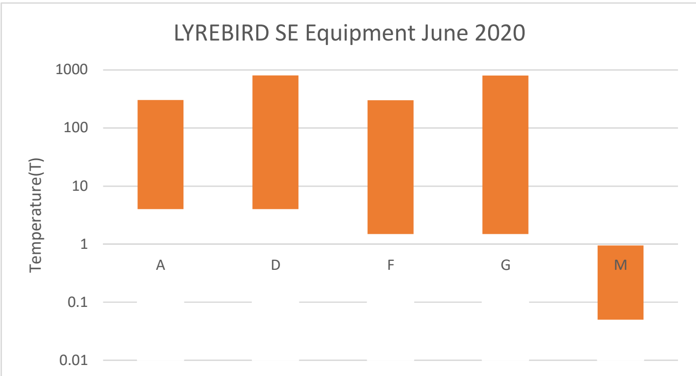


Figure 7.7: Temperature range for equipment compatible with Taipan: Be Filter

7.2.4 Emu: High-resolution backscattering spectrometer

Instrument Type	Back-Scatter Spectrometer
Tilt stage	none
XY Stage	none
Z-Stage	none
Rotation	none
Maximum load	500 kg TBC
Stage to beam distance	not applicable
Beam Line	CG3

Emu is a back-scatter spectrometer consisting of a large vacuum vessel containing a chopper and the detector array. A Doppler wavelength selector is mounted outside the detector vessel on a large granite block. The vessel incorporates a well for mounting sample environment equipment. This allows sample environment changes with out venting the detector vessel. Access to the sample area is via a two staircases. Space can be cramped on the upper level. Particular attention should be made to ensure trip hazards in this area are removed.

Top-loading cryofurnaces CF-11 & CF-12
High temperature vacuum furnace F-1
Bottom-loading cryofurnaces CF-1 & CF-3
Top-loading cryostat CF-10
Dilution refrigerator insert DL-1

10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Gas delivery probe TL-5 & TL-9

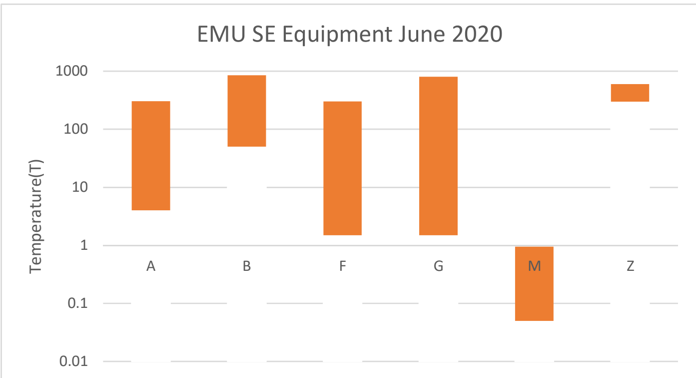


Figure 7.8: Temperature range for equipment compatible with Emu

7.2.5 Pelican: Time-of-flight spectrometer

Instrument Type	TOF Spectrometer
Tilt stage	none
XY Stage	none
Z-Stage	none
Rotation	none
Maximum load	500 kg TBC
Stage to beam distance	not applicable
Beam Line	CG1

Pelican is a time of flight spectrometer. It consists of a large vacuum vessel that contains the detector array. Sample environment is lowered into the vacuum chamber and forms part of the outer wall of the vacuum vessel. Hence changes of sample environment requires venting of the vessel and subsequent pump down, this can take several hours. Also the sample environment has to be vacuum tight. Check sealing surface and o-rings before assembly. The sample area is accessed via a short vertical ladder, take care when using this and ensure the gate at the top of the ladder remains closed. Space for sample environment equipment is limited on top of the vacuum vessel. Some equipment is mounted on the mezzanine that surround the detector vessel. As the detector can be moved there is a need to ensure that sample environment leads are long enough for the full range of movement expected during an experiment.

Top-loading cryofurnaces CF-11 & CF-12
High temperature vacuum furnace F-1
Bottom-loading cryofurnaces CF-1 & CF-3
Top-loading cryostat CF-10
Dilution refrigerator insert DL-1

10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Gas delivery probe TL-5 & TL-9

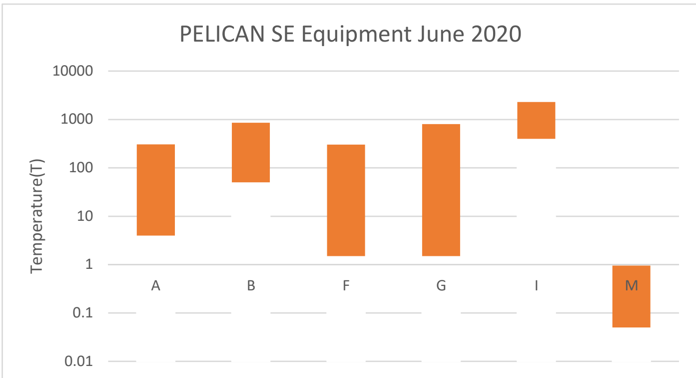


Figure 7.9: Temperature range for equipment compatible with Pelican

7.3 Scattering Instruments

7.3.1 Bilby: Small-angle neutron scattering

Instrument Type	Small angle scattering
Tilt stage	2 x +/-10 °
X Stage	+/- 500mm from beam centre across beam
Y Stage	1500mm along the beam
Z-Stage	500mm
Rotation	+/- 175 °
Maximum load	650 kg
Stage to beam distance	0 - 500mm
Beam Line	CG2A

Bilby is a small angle neutron scattering instrument that can be operated with Fermi chopper to select wavelength or in time of flight mode. A rotation stage is provided on Bilby the angular range of rotation is limited by the type of sample environment mounted and should be checked before driving this axis. A two set of stairs are used to reach the small sample area. The area for sample environment is constrained by the Quokka wall and Spatz neutron guide. A tunnel is provide under the neutron guide this is not to be used to gain access to the sample area. It is for egress if someone become incapacitated in the Alignment of the sample environment is assisted by the use a laser cross-hair that can be mounted in place of a guard aperture. Due to the constraints of the sample area please discuss use of HM-2 with your instrument scientist before arrival.

Twelve-position sample changer SC-8
Bottom-loading cryostat CF-6
Bottom-loading cryostat CF-1 & CF-3
5 T horizontal magnet HM-1
10 T horizontal magnet HM-2
1 T horizontal magnet HM-3
^{3}He refrigerator insert OS-1
Ten-position sample changer SC-1
Five-position rotating tumbler SC-7
^{3}He refrigerator insert OS-1
10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
SANS high pressure cell SPC-1
Battery tester BT-1
Gas delivery system GD-1
Gas spectrometer GS-1
Vapour delivery system VD-1
Rheometer RH-1
Full-range probe TL-1 & TL-3

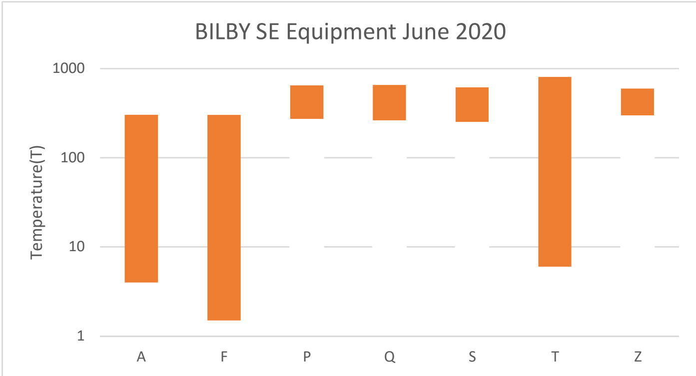


Figure 7.10: Temperature range for equipment compatible with Bilby

7.3.2 Quokka: Small-angle neutron scattering

Instrument Type	SANS with velocity selector for wavelength selection
Tilt stage	2 x +/-15 °
X Stage	+/- 500mm from beam centre across beam
Y Stage	+/- 250mm from beam centre along beam
Z-Stage	500mm
Rotation	+/- 180 ° reduced if X stage fitted
Maximum load	600 kg
Stage to beam distance	0 - 500mm
Beam Line	CG1

Quokka is a small angle neutron scattering instrument with velocity selector for wavelength selection. A super-mirror is installed for polarised neutron analysis. A rotation stage is provided on Quokka the angular range of rotation is limited by the type of sample environment mounted and should be checked before driving this axis. A set of stairs are used to reach the sample position. These lock into the linear rails the sample stage moves on. Before climbing these stairs ensure they are fixed in place. A second ladder is provided for use when tall cryostats are mounted on the sample stage ensure all wheels are locked before ascending this ladder. Both of these stairs must be climbed facing the treads. Alignment of the sample environment is assisted by the use a laser cross-hair that can be mounted in place of a guard aperture. An interlock is provided for hazardous sample environment equipment so it can only be energised once the enclosure door is closed.A local exhaust is provided to exhaust gases and vapours from the enclosure.

Twenty-position sample changer SC-2	10 kV electric field system EF-1
Bottom-loading cryostat CF-6	Potentiostat/galvanostat PG-1
Bottom-loading cryostat CF-1 & CF-3	SANS high pressure cell SPC-1
5 T horizontal magnet HM-1	Battery tester BT-1
10 T horizontal magnet HM-2	Gas delivery system GD-1
1 T horizontal magnet HM-3	Gas spectrometer GS-1
^{3}He refrigerator insert OS-1	Vapour delivery system VD-1
Ten-position sample changer SC-1	Rheometer RH-1
Twenty-position sample changer SC-2	Stop-flow mixing cell SF-1
Five-position rotating tumbler SC-7	Differential scanning calorimeter DSC-1
^{3}He refrigerator insert OS-1	Rapid viscosity analyser RV-1

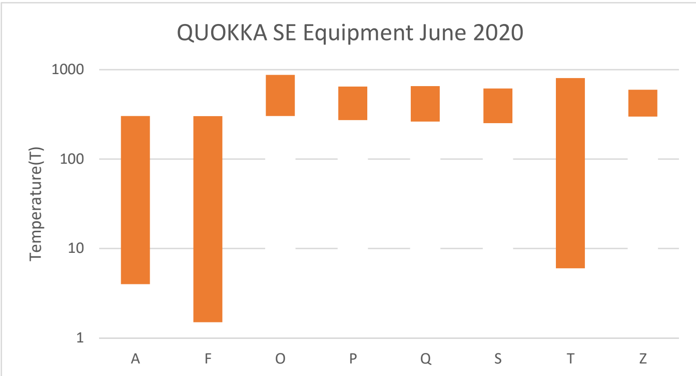


Figure 7.11: Temperature range for equipment compatible with Quokka

7.3.3 Kookaburra: Ultra-small-angle neutron scattering

Instrument Type	Ultra-small angle scattering
Tilt stage	none
XY Stage	none
Z-Stage	1
Rotation	none
Maximum load	50 kg on integral sample changer
Maximum load	500 kg on Heavy Table when installed
Stage to beam distance	not applicable
Beam Line	CG3

Kookaburra is an ultra-small angle scattering instrument using channel cut silicon crystal arranged to use Bragg refection to collimate the beam. The crystal are mounted on tilt and translation stage and these are mounted on a large granite base. Do not touch the crystal mounts of the base to avoid having to realign the beam. Sample environment other than SC9 requires the assembly of a support structure over the granite table. This means the crystal are moved further apart and background is increased in this configuration. An upgrade is underway on this instrument that will affect the mounting on sample environment equipment.

Six-position rotating sample changer SC-9	10 kV electric field system EF-1
Bottom-loading cryostat CF-1 & CF-3	Potentiostat/galvanostat PG-1
5 T horizontal magnet HM-1	Battery tester BT-1
Rheometer RH-1	Gas spectrometer GS-1
Stop-flow mixing cell SF-1	Vapour delivery system VD-1

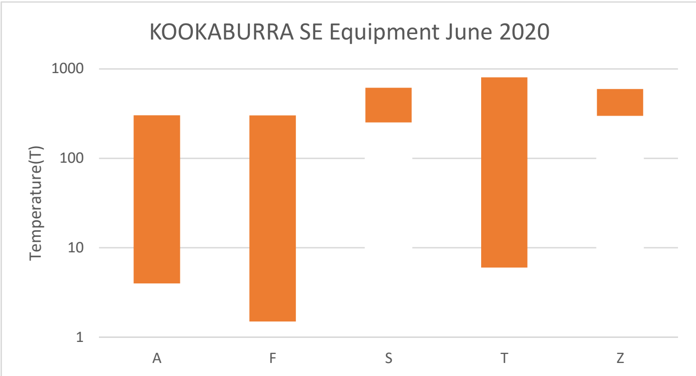


Figure 7.12: Temperature range for equipment compatible with Kookaburra

7.4 Reflectometry Instruments

7.4.1 Platypus: Horizontal neutron reflectometer

Instrument Type	Horizontal Reflectometer
Tilt stage	2 x +/- 20 °
X Stage	750mm
Z-Stage	300mm
Rotation	none
Maximum load	250 kg
sample plate to goniometer centre	240mm
Stage to beam distance	0-300 mm
Beam Line	CG3

Platypus is a neutron reflectometer. It includes a super-mirror for polarised neutron analysis. The sample normally a horizontal plate is placed on the sample stage and manual z stage. So the sample remains stationary when tilted the distance from the sample to the tilt stage has to be adjusted manually so the sample corresponds with the centre of rotation the tilt stage. The sample can then be lifted to beam height using the remotely controlled large z stage on which all the other stages are mounted. A line laser is provide to indicate the approximate height of the beam. The sample area is restricted around the sample stage and special care should be taken when passing under the guide to reach the far side. When the field coils for polarised analysis are on the guide you will need to ensure you don't bump them if you are moving around them. A local exhaust is provided just outside the sample area for exhaust of gases and vapours from the sample enviroment equipment.

Solid-liquid cell and HPLC Pump SL-1
Bottom-loading cryostat CF-6
1 T horizontal magnet HM-3
Syringe pump SP-1
Langmuir film balance LF-1 & LF-2
Air/Liquid cells SC-3
10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Rheometer RH-1
Gas spectrometer GS-1
Vapour delivery system VD-1

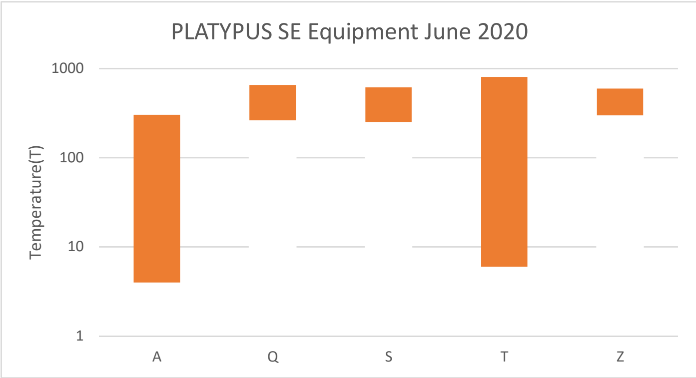


Figure 7.13: Temperature range for equipment compatible with Platypus

7.4.2 Spatz: Vertical neutron reflectometer

Instrument Type	Vertical reflectometer
Tilt stage	2 TBC
XY Stage	2 TBC
Z-Stage	1 under development
Rotation	+/- 180 TBC
Maximum load	250 kg TBC
Stage to beam distance	255mm
Beam Line	CG2B

Spatz is a vertical reflectometer. The sample normally a vertical plate is placed on the sample stage and z stage. So the sample remains stationary when tilted the sample is moved to the centre rotation stage A line laser is provided to indicate the approximate height of the beam.

Spatz is our newest instrument and more sample environment equipment is currently being developed including *in situ* FTIR

Solid-liquid cell and HPLC Pump SL-1
In situ FTIR IR-1
Syringe pump SP-1
Langmuir film balance LF-1 & LF-2
10 kV electric field system EF-1
Potentiostat/galvanostat PG-1
Battery tester BT-1
Vapour delivery system VD-1
Gas spectrometer GS-1

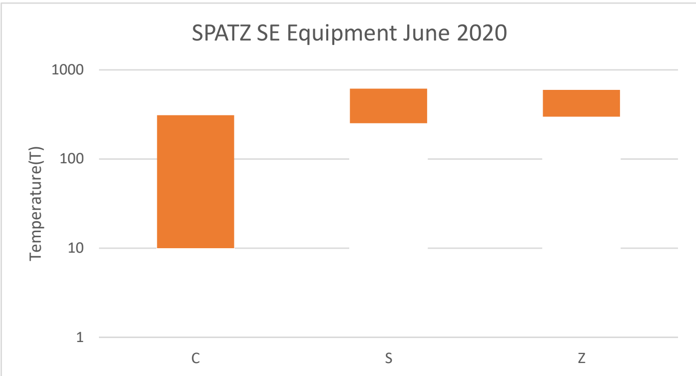


Figure 7.14: Temperature range for equipment compatible with Spatz

7.5 Imaging Instruments

7.5.1 Dingo: Neutron radiography/imaging/tomography

Instrument Type	Radiography/Tomography
Tilt stage	none
XY Stage	2
Z-Stage	1
Rotation	+/- 180 °
Maximum load	10 kg
Stage to beam distance	not applicable
Beam Line	TB2

Dingo is a neutron imaging instrument capable of radiography and tomography with other techniques under development.The photos generated by neutrons interacting with the scintillation screen are reflected through 90 and captured by a camera, various camera types are available. The sample is mounted on a the translation/rotation stage between the neutron flight tube and a scintillation screen. The sample area is within a concrete bunker. The ceiling height restricts the sample environment that can be used . Samples measured on Dingo are invariably active at the end of an experiment. Special procedure must be followed to safely handle and store these samples. As the sample area is enclosed the use of some gases has to be risk assessed prior to the experiment. This instrument is sited in the reactor beam hall hence experiments invovling flammable gases have to be cleared with Reactor Operations before the experiment takes place.

Ambient temperature mount	10 kV electric field system EF-1
Bottom-loading cryostat CF-5(cylindrical tail)	Potentiostat/galvanostat PG-1
High temperature vacuum furnace F-1	Battery tester BT-1
	Vapour delivery system VD-1
	Gas spectrometer GS-1

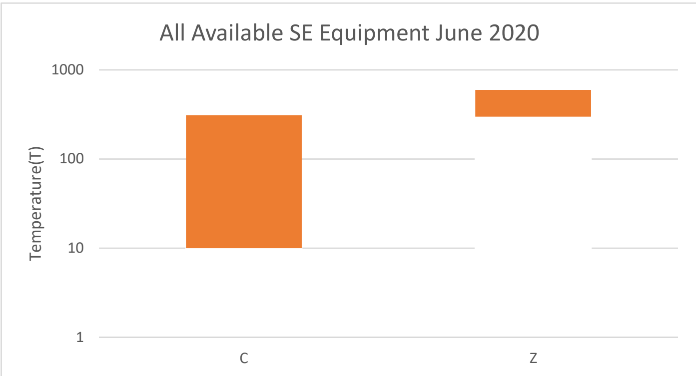


Figure 7.15: Temperature range for equipment compatible with Dingo

Chapter 8

Laboratory Equipment

Contact the Lab Manager via email (acns_laboratories@ansto.gov.au)

The ACNS guide hall laboratories are provided for external researchers to prepare of samples for examination on the guide hall and reactor beam hall instruments. This means these labs operate differently to those that are available in universities and other research facilities. Safety is of greatest concern because of the high flux of researchers arriving and leaving we insist that all work in the labs is discussed with the lab manager before arrival. This is to ensure that workers using incompatible chemicals or techniques are safely separated during their stay. Furthermore while working the labs please keep a high awareness of the other researchers in the lab. Talk to other user in the lab and ensure each other's safety by being aware of what is going on around your work space.

To ensure your visit to the ACNS Labs is a smooth as possible please ensure equipment is booked for use in your proposal or after discussions with the lab manager prior to your scheduled experiment time. Please ensure you have experience with the equipment you have booked prior to your visit. At least read the manual where applicable, make and model of our equipment is given below. Time is generally limited once you arrive and there may not be time for training before your samples are needed on the neutron instrument. While we attempt to ensure the accuracy and calibration of equipment in the labs we cannot be sure the last person to use a piece of equipment has left it in the best condition. Hence it is advised to use good laboratory practice and check accuracy of equipment before use. For instance dispense set volumes of Milli-Q water from the pipette you intend to use onto an analytical balance to check its operation.

The ACNS labs have a limited supply of general chemicals always provided the lab manger, prior to arrival, all chemicals you intend to use. Do not assume a chemical is available because you have access in your own lab. The ACNS labs are for sample preparation, dilution, mixing and some optical measurements there is no provision for synthesis in these labs

8.1 Balances

- Sartorius TE313S
- Wedderburn Precisia XB6200D
- Kern ABT-120-SDM
- Sartorius BP210S
- Wedderburn Precisia XT620M
- Wedderburn Precisia XB4200C
- Sartorius Cubis
- Vibra AF-E
- Vibra AF-CE
- Ohaus Explorer Pro
- Ohaus Navigator
- Anton PAAR density meter DMA5000

8.2 Milli-Q water purification units

- Academic
- Gradient A10
- Symplicity

8.3 pH Meters

- Metrohm pH meter 827pH
- Metrohm pH meter

8.4 Benchtop water baths

- Techne water bath
- Major science stirring water bath SWB series

8.5 Hotplates and stirrers

- Cole Parmer Vela hotplate
- IKA colour squid stirrer
- SEM hotplate stirrer
- Flatspin stirrer
- Stuart hotplate stirrer CB162
- Plus extras

8.6 Ovens and furnaces

- Labec
- Thermoline
- MTI tube furnace GSL 1600x
- Labec muffle furnace HT 04/17
- Vacuum oven
- Thermoscientific Sanyo CO2 incubator

8.7 Ultrasonic equipment

- Grant XUBA3
- Unisonics
- Symplicity
- Ultrasonics Eco EcoCT5
- Ultrasonic sonic vibracell and probe

8.8 Vortex and centrifuge equipment

- Eppendorf centrifuge 5810R
- Scientific industries Vortex Genie 2
- Benchmark mini centrifuge
- Eppendorf minispin

8.9 Gloveboxes

- Innovative Technology (helium) glovebox
- MBraun (argon) glove box MB 200B
- Plaslabs (nitrogen) dry box
- LABCONCO glovebox (Positive pressure)

8.10 Benchtop spectrometers

- Thermoscientific Nanodrop 2000 spectrophotometer
- Thermoscientific FTIR Nicolet IS/O
- UV-VIS Varian Cary 50 BIO

8.11 Specialised sample preparation

- Specac hydraulic press
- Jetlight UV Ozone cleaner 144AX
- Spin coater WS-400BZ-6NPP/lite

8.12 Laboratory glassware washers

- Miele Professional G7883
- Miele Professional G7804

Chapter 9

General Notes

These are notes for use of ancillary equipment not covered in main chapters. Some quick tips and things to watch when working with the sample environment equipment. If anything looks, smells, sounds or feels (hot or cold) different while you are using it tell your local contact and/or sample environment. Your observation of a change in behaviour may indicate some deterioration that may be rectified before damage and the loss of beam-time occurs. If you think something is broken please label and set aside. Don't leave it in the hope that eventually we can find it before someone else needs it.

9.1 Vacuum equipment

9.1.1 Vacuum lines

The majority of vacuum lines used at ACNS are flexible corrugated stainless steel lines with standard KF fittings. Please avoid compressing (standing on a line or catching in a moving part of an instrument) or kinking the vacuum lines. If a line is severely deformed do not try to straightening as this may result in the wall cracking. Notify SE if you think a line has been damaged before using it.

Before joining a vacuum line to a piece of sample environment equipment, (ie in preparation for a sample change) carry out the following steps.

- check the hose for kinks and damage.
- check the interior is clean
- check the sealing flange for deformation or radial scratches
- check the o-ring and retainer are clean.
- bend the o-ring and check it is not perished.
- if the o-ring is dry; apply vacuum grease and wipe off excess.

If any of the above occur inform sample environment. If sample environment are not available label the damaged hose before collecting a replacement.

9.1.2 Vacuum gauges and displays

There are two types of vacuum gauge you will see as a user. They both use the same type of digital display but have different methods of operation and measurement ranges.

Firstly, the small rectangular gauges used during sample changes and for general use are composite piezo/Pirani gauges. The piezo part of the gauge measures the deformation of a diaphragm and operates above approximately 7 mBar and the Pirani part of the gauge measures the thermal conductivity of the residual gas and operates from 7 mBar to below 10-3 mBar. You may notice a discontinuity of readings as the gauge changes from one method to the other. so around 7 mBar the pressure reading may appear to rise before it continues to decrease this is normal. In some cases the ultimate vacuum achievable by our vacuum pumps is beyond the measurement range for these gauges. If the pressure drop below the minimum 'UR' is displayed on the digital display. Standing for under-range.

Secondly the large cylindrical gauges used on the turbomolecular pumps (discussed below) are combined cold cathode and Pirani vacuum gauges. The cold cathode gauges operate by ionising gas molecules and measuring the current generated. A permanent magnet is used to force the ionised molecules to travel a spiral path to the anode. This increase in path length increases the sensitivity of the gauge at low pressures. These gauges measure to below 10-9 mBar. *Please note; Helium is not easily ionised and these gauges may read a lower vacuum pressure when pumping on residual helium gas. Nor should the reading be relied upon for pressures above 10 mBar*

9.1.3 Vacuum pumps

Use of turbos, Ballast on dry pumps, tricks when pumping damp chambers

Sample environment use several types of vacuum pump. Please discuss any vacuum requirements with us so the appropriate pump can be supplied. In order of ultimate vacuum the pumps we use are dual diaphragm (also used in as backing pumps for turbo molecular pumps), scroll pumps, multi-stage roots rotor and turbomolecular pumps.

Diaphragm pumps are used for rough vacuum high volume applications. these pumps are robust and can pumps vapours but have an ultimate vacuum of around 1 mBar. The pumps operate using a reciprocating diaphragm and two one way vales to produce vacuum. They are used to back turbomolecular pumps, rough vacuum for drying, re-circulation in the vapour system VD-1and for evacuating demountable sample cells prior to filling with solvent. These pumps are very robust. Note any deterioration in ultimate vacuum or excessive noise from the pump this could indicate a failure to the diaphragm or bearings in the pump.Notify sample environment and tag out for service.

Scroll pumps are predominately used in the helium re-circulation systems of some of our top loading cryostats and on our glove boxes. You will probably not need to operate these pumps. The method of operation is to move one meshed spiral in a circular motion against a fixed spiral. The moving spiral is sealed by a Teflon insert referred to as a tip seal. This produces a powder in the vacuum system and the seals hence need replacing at regular intervals.

Multi stage roots rotor pumps are oil free medium vacuum pumps with ultimate vacuums depending on size in the range from 10^{-2} mBar to 10^{-3} mBar. These are used for sample changes and roughing down large vacuum chambers. These pumps have difficulty pumping vapours and in extreme cases can seize up and cease to operate. If there is any chance of water or condensable vapour existing in the chamber to be evacuated the ballast should be cracked open when initially pumping down. the ballast introduces a small amount of air into the pump to assist in moving the condensate to the exhaust. This also decreases the ultimate vacuum. When doing a sample change where the probe may be moist it is recommended to open the ballast valve 1/4 of its total travel. (1/16th of a complete turn). To turn the pump on use the large black switch on the rear of the pump. Do not remove the silver connector on the back of the pump. This is the remote control connector and if it is removed the pump will not turn on using the local switch.

Turbo molecular pumps spin at remarkable speed (15000 Hz) to impart momentum to molecules of gas and attempt to deflect them toward the exhaust. By this means ultimate vacuum of below 10^{-7} mBar can be achieved. They do not operate like a fan or turbine though they do look like one. In fact the pumps used at ACNS incorporate a turbo drag section as well but we wont go into this. Because they only operate effectively at very high speed the bulk of the gas must be removed before the turbo pump begins to spin so the viscous drag is low enough that the motor can spin the pump. The roughing of the vacuum chamber is carried out by an integral dual diaphragm pump. The start of the turbo is initiated automatically after a set time period after the start of the diaphragm pump. The current to the turbo motor is then ramped slowly up maximum until the speed operating speed is reached. Because of the high speeds the bearing are either levitation or ceramic bearing so ideally the pumps should not be moved while at operating speed. Similarly turbo pumps can not handle rapid increases in pressure (inadvertent opening of valves) as the rapid deceleration can irreparably damage the pump. The ramp to maximum and time to operating speed is set in the control unit. If the time to lower the pressure in the vacuum chamber exceeded that programmed the pump will shut down. Hence a turbo should be monitored after switch on until the vacuum is better than 10^{-1} mBar. If this pressure is achieved the pump will probably continue to operate. If the vessel takes longer to evacuate due out-gassing or trapped moisture (virtual leaks) there are several things to try.

- If the vessel known to be damp purge with dry nitrogen and evacuate with a multi-stage roots rotor pump several times.
- For large vessels rough pump with an appropriately sized roots rotor pump to its ultimate vacuum arrange valves and vacuum gauge so the vessel pressure can be read with the pump valve closed. This will give an impression of the amount of out-gassing occurring. Pump until a pressure below 10^{-1} mBar can be maintained with pump valve closed. Then swap to the turbomolecular pump.
- For dry vessels allow the vessel to vent to about 100 mBar and retry this will work if the turbo is almost reaching 10^{-1} mBar prior to timing out and may be attempted several times.

9.2 Pressure equipment

9.2.1 Pressure regulators

If you have not used pressure regulators before your visit to ACNS there are a few points to note.

Pressure regulators maintain a constant pressure (not flow) on their outlet as long as the inlet pressure is higher. This is achieved by connecting a valve to a flexible diaphragm within the regulator. On one side of the diaphragm is the outlet gas chamber and on the other a spring connected to the adjustment knob. Gas stops flowing to the outlet when the adjustable spring pressure equals the outlet pressure. For this reason **to lower the outlet pressure the knob must be wound anti-clockwise**. That is in the opposite direction to turning off a tap.

Points to note;

- Always check the regulator is wound out before opening a gas bottle.
- open a gas bottle tap slowly. Open completely then close 1/4 turn
- adjust the regulator knob slowly.
- when finished with a regulator wind the knob all the way out to stop flow and then in 1/4 turn.

Never leave a tap or regulator hard against a open stop. In an emergency people can forget which way to turn taps and knobs and if they are against an open stop it can be assumed a tap is off when it is in fact opened hard all the way. The only time a tap or knob should be against a stop is it is closed.

9.2.2 High pressure helium lines

The pressure lines connected to the helium cold heads on the dry and re-condensing cryostats require special handling. These lines consist of a corrugated stainless tubing covered with stainless steel braid. The pressures in these lines is very high and this makes the lines themselves quite stiff however if they are kinked they can be easily damaged. These line have a minimum safe bend radius of 1 metre. do not attempt to bend them in a tighter radius as a failure of a line will delay your experiment. If the sample enviroment will move during the experiment always do a run from the extremes of the travel to ensure the lines are not kinked by the movement of the neutron instrument. This is especially important if the cryostat is going to be rotated. Avoid complete rotations and always rotate the cryostat back to the starting point after 180° rather than continuing past the 360° mark. Do not run you hands along the helium pressure lines as the stainless steel wires in the braid can break leaving stiff wires sticking out of the line that can easily puncture the skin.

9.3 Cryogens

Prior to using any cryogens in the guide hall all user must complete a safety handling course conducted by one of the sample environment team. Liquid nitrogen and liquid helium are provided for the running of the wet cryostats provided by the sample environment team. Training is provided on arrival for safe transfer of cryogens into the particular piece of sample environment you are using. Various cryogen level meters are provided to monitor cryogen levels in cryostats and storage Dewars. Be sure you are aware of the operation of the particular meter you are using. If liquid nitrogen is required for sample preparation in the laboratory please discuss your needs with the lab manager in the first instance. Small dewars can be provided by sample enviroment for use within the fume hoods of the laboratory spaces.

When using helium transfer lines please notify sample environment if excessive condensation occurs on the line as this may indicate that the annular space around the tubing of the transfer line requires evacuation. Prior to doing a helium transfer ensure o-rings are in good order on the cryostat fill port and on the storage Dewar.

9.4 New Equipment Template

Ancillary codes	....
Compatible instruments	....
Temperature ranges	....
Time to cool coils	....
Time to base temperature	....
Thermometry	....
Maximum temperature	....
Maximum magnetic field	....
Maximum voltage	....
Maximum current	....
Maximum applied load	....
Maximum pressure	....
Compatible sample probe	....
Compatible sample cells	....
Mount required for sample	....
Sample containers	....
Geometries provided	....
Complementary ancillaries	....
Project folder	....
User instruction	....
SICS control	....
Equipment Number	....