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The LOQ Instrument Handbook Volume 1

S M King and R K Heenan

June 1996

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The LOQ Instrument Handbook

Volume I

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Introduction

Dear LOQ User,

Welcome to the *LOQ Instrument Handbook*!

We hope that you will find this handbook useful. Our aim has been to provide you with as much information about the capabilities and method of operation of the LOQ diffractometer and its associated equipment as we think you need to know. We hope that this information will help you to write good proposals and to conduct productive experiments.

This is not really a User Manual in the normal sense. We believe that a book of words is no substitute for hands-on instruction, and in any event the sheer diversity of sample environment equipment routinely employed on LOQ would have made a step-by-step guide unworkable! That said, there will be times when something goes wrong and you cannot contact us. Then, just maybe, the information in this file, together with a little common sense, will get you going again. But, if in doubt, please seek advice!! Is one of the CRISP or SURF instrument scientists nearby? Can the Main Control Room help?

Be aware that there are other sources of information that you can turn to. One very good one (since you are bound to interact with a computer at some point during your experiment!) is the *PUNCH Manual* in the black ring binder. This contains information about the ISIS computers and their operating systems, the ICP, the CAMAC system and GENIE. A *PUNCH Mini-Guide* is available from the ISIS Computer Support Group. In addition there are several on-line help libraries (which may be accessed by typing HELP or INFO when logged into an ISIS VAX computer) covering a multitude of topics.

Finally, it goes without saying that in such a leading-edge environment, change is inevitable. Whilst we will do our best to ensure that the information in this report is updated at reasonably frequent intervals, please tell us if you find any errors or glaring omissions! Equally, we welcome your constructive criticisms and suggestions about what this file should contain since the best people to tell us are yourselves!

It is our intention to eventually produce a companion volume to this report. Volume II will deal with the details of time-of-flight SANS at pulsed neutron sources and the consequences for data reduction and analysis, based on our experiences.

Steve King & Richard Heenan

For convenience this volume is divided into five sections:

The LOQ Instrument Scientists

Dr Richard Heenan
Main interests: *Microemulsions, micelles, polymer-surfactant interactions & model fitting!*

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Telephone: (office) +44 (0)1235-446744

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Telephone: (office) +44 (0)1235-446437

Fax: (24 hours) +(44) 01235-445720, (office hours only) +44 (0)1235-445103

The LOQ instrument may be dialled direct on +44 (0)1235-446859

The Rutherford Appleton Laboratory switchboard is +44 (0)1235-821900

World Wide Web

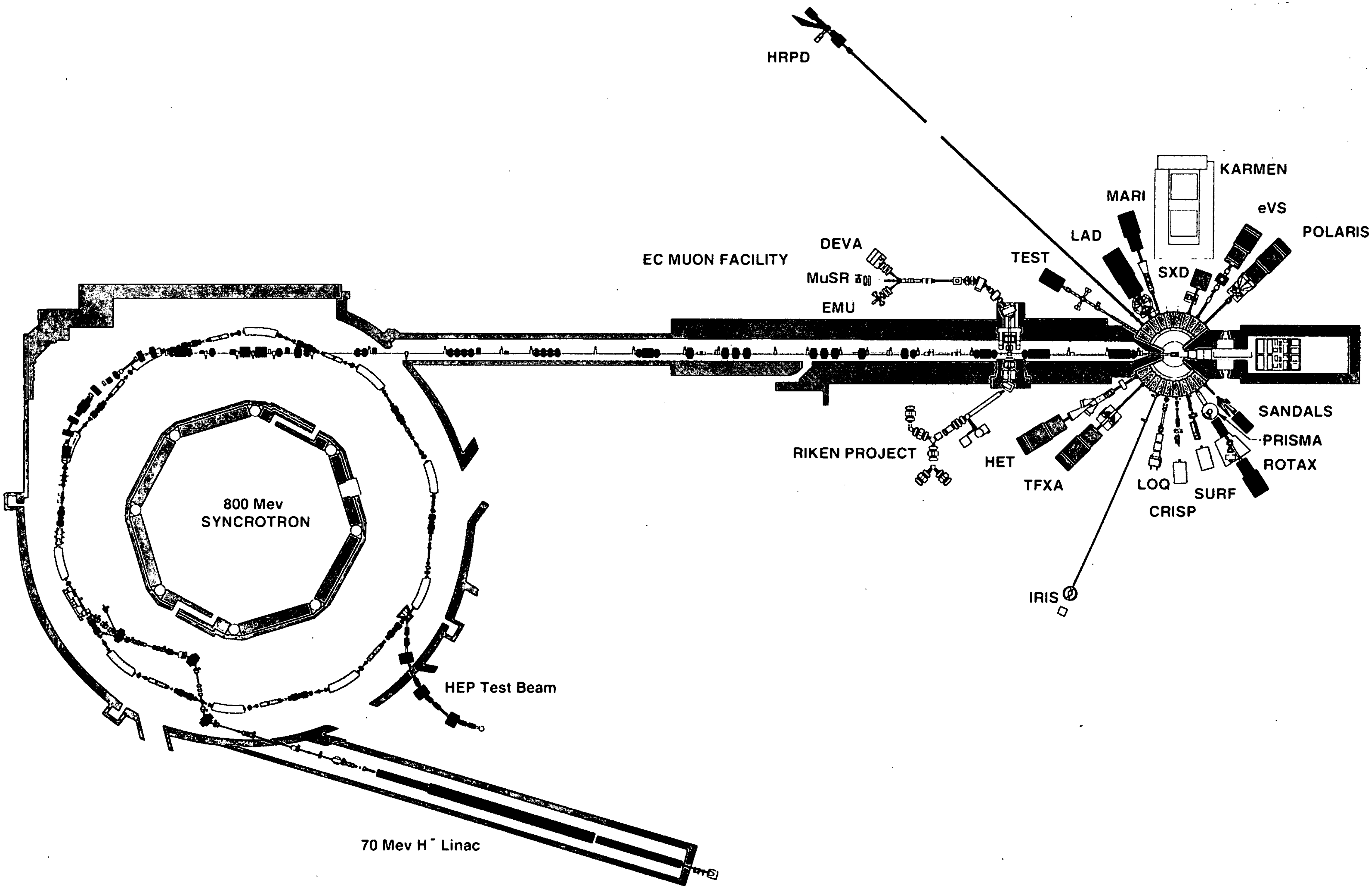
The URL of the LOQ Home Page is: **http://ndnt01.nd.rl.ac.uk/instr/loq/loq.htm**

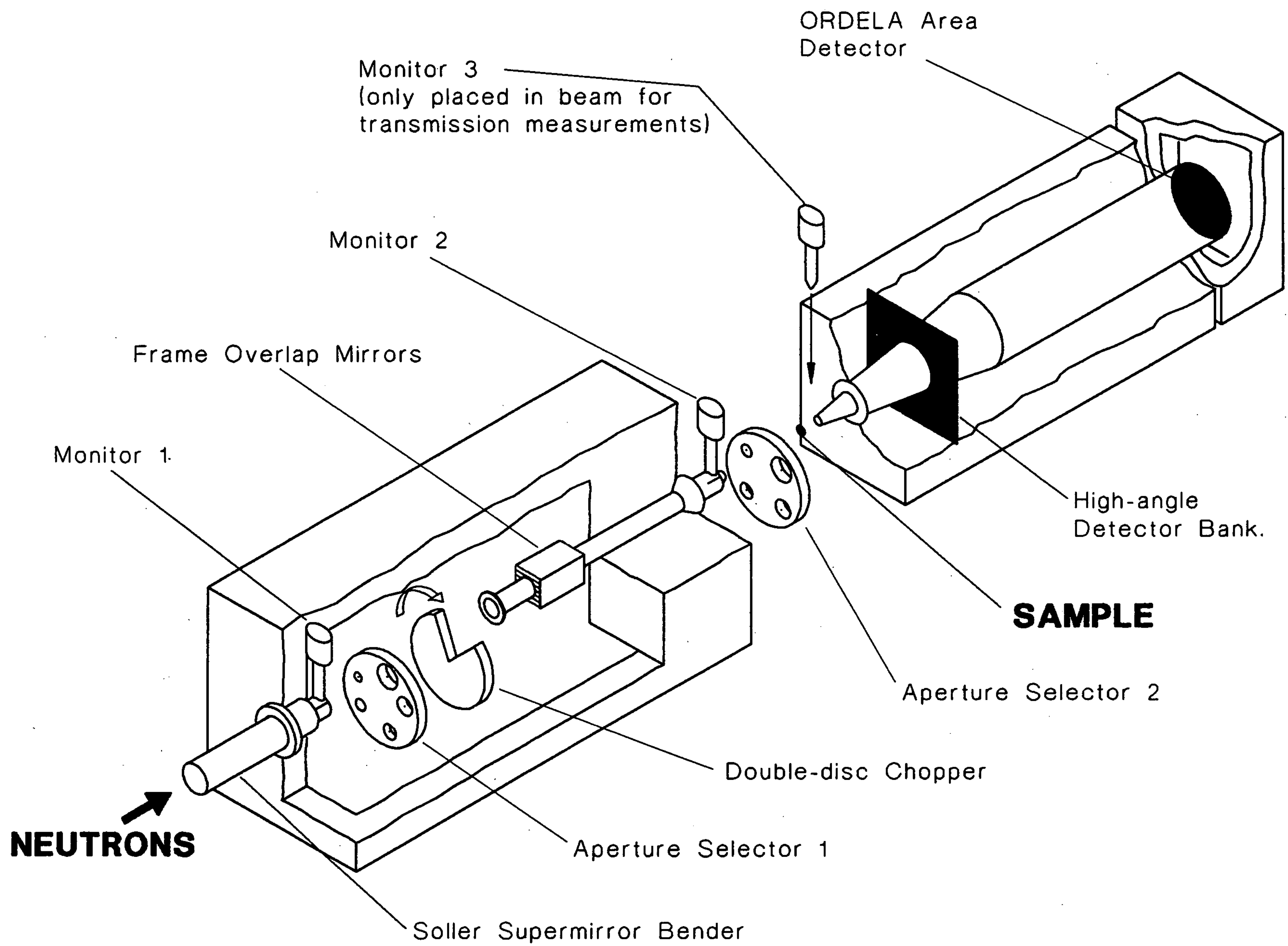
Out-of-Hours Support

ISIS staff do not operate a 24 hour callout system. Most are happy for you to contact them at home except during the hours 23:00 to 07:00. The only exceptions to this rule are when (a) there is a failure that affects all beamlines, or (b) there is a safety problem. In either case the ISIS Main Control Room should be notified first on extension 6789. Please do not abuse this system.

If you believe that the only cure for a problem is for a member of staff to come in at night, please do not wait until 23:00 before contacting them!

A list of home telephone numbers for various ISIS staff may be found in the LOQ cabin.


HRPD
KARMEN
MARI
eVS
LAD
POLARIS
DEVA
TEST
EC MUON FACILITY
SXD
MuSR
EMU
SANDALS
PRISMA
RIKEN PROJECT
HET
ROTAX
TFXA
LOQ
SURF
CRISP
800 Mev
SYNCROTRON
IRIS
HEP Test Beam
70 Mev H⁻ Linac



THE LOQ DIFFRACTOMETER

LOQ - Small Angle Scattering Instrument

LOQ uses small-angle diffraction to investigate the shape and size of large molecules, small particles or porous materials with dimensions in the range 1 - 100 nm. Length scales of up to 400 nm can be probed in highly anisotropic systems. This instrument is therefore of interest to those involved in the study of colloids, polymers, biomolecules, alloys, composites and porous systems. For more information about LOQ, visit our World Wide Web home page at **http://ndnt01.nd.rl.ac.uk/instr/loq/loq.htm**

Specification

Incident wavelengths	2.0 - 10.0 Å at 25 Hz
Momentum transfer, Q	0.008 - 0.24 $Å^{-1}$ (0.008 - 1.4 $Å^{-1}$ from mid-1996)
Dynamic range in Q	30 (175 from mid-1996)

Instrument

ISIS beam line	N5, viewing the 25 K liquid hydrogen (lower) moderator.
Primary flight path	Soller supermirror bender (24 mrad, to remove neutrons with wavelengths less than 2 Å), upstream scintillator monitor, aperture dial N^o 1, variable-opening (2 - 126°) disc chopper, frame overlap mirror (removes neutrons with wavelengths greater than 12 Å), 3 m evacuated flight tube, sample position scintillator beam monitor, aperture dial N^o 2, final collimation tube.
Sample position	Around 11.1 m from moderator. Approximate size is 0.4 m (parallel to beam) by 1.5 m. No height restriction. Beam is approximately 0.63 m above base plate. Crane access possible (SWL 1000 Kg). Sample transmission scintillator monitor on motorised rack. Provided with water, helium and electrical services. Secondary, top-loading, in-vacuum sample position with limited access and services around 12.5 m from moderator (giving approximate Q range 0.01 - 0.34 $Å^{-1}$).
Beam size at sample	Defined by aperture N^o 2 and final collimation. Between 2 - 20 mm diameter. Typically 8 mm diameter.
Neutron flux at sample	Dependent on collimation, ISIS accelerator performance and target type. Typical time-averaged flux is $2x10^5$ $cm^{-2}s^{-1}$ (ISIS at 50 Hz, 200 μA 800 MeV proton beam, tantalum target).
Secondary flight path	Evacuated tank to main detector.
Detectors	^3He-CF_4 filled ORDELA "area" detector 15.15 m from moderator. Active area is 64 cm × 64 cm. Detector mapping under software control. External, annular, high-angle, scintillator detector bank 11.6 m from moderator (from mid-1996)

Computer System

Data Acquisition	Standard ISIS DAE (DAE I).
Hardware	DEC VAXstation 3200 with colour monitor. 1.6Gb fixed disk (allows online storage of data for about 1 ISIS cycle). EtherNet connection to ISIS HUB computer (twin VAX 4000 Model 500's with WORM optical storage unit). Additional Tektronix-type terminal. An RS232 connection to an ISIS terminal server is also available to users with laptop computers.
Software	VAX VMS 6.1 operating system, *GENIE©*, *COLETTE©* (instrument-specific data reduction in one or two dimensions either online or from file), *FISH©* (sophisticated model-fitting), *VULOQ©* (data visualisation, colour and b/w, 1D, 2D and 3D), *SANDRA©* (data manipulation utility).

Sample Environment

Control	Standard ISIS CAMAC where used. PC control is also available. User-supplied monitoring equipment can be interfaced provided that sufficient notice is given.
Standard	In air. Alignment laser to position samples. Computer-controlled sample changer connected to computer-controlled water/oil bath (-20 to +95°C) taking any two of; two 10-position racks for 10 mm wide quartz cells (maximum path-length 10 mm) or two 4-position racks for 50 mm × 50 mm cadmium masks with various diameter apertures or one 8-position rack for 20 mm wide quartz cells. A nitrogen gas purge, electrical heaters and Pt-100 or K-type thermocouples are also available.
Non-standard (Must be requested on proposal)	"Orange" helium cryostat, closed-cycle refrigerator, RAL furnace, *Newport* electromagnet (for field across neutron beam), *Goudsmit* electromagnet (for field across or along neutron beam), Couette-type & Poiseuille-type shear flow cells (please contact SMK), user-supplied apparatus.

Notes for proposers:

1. Where necessary, suitable quartz sample cells are available for loan. Please discuss with the instrument scientists.
2. For low temperature work down to 10 K we recommend the use of a closed-cycle refrigerator rather than an orange cryostat. This is because a CCR has fewer aluminium alloy thermal shields to scatter neutrons.
3. The *Newport* electromagnet provides approximately 1.0 T over 15 mm decreasing to about 0.5 T over 50 mm. The *Goudsmit* electromagnet provides at least 1.5T over 50 mm. 1.9T has been achieved with smaller pole gaps.
4. The variable-opening chopper is under computer-control. This permits, for example, automatic quasi-monochromatic running for inelastic effect measurements. Please note, however, that in this mode the time resolution is poor and count rates are low.

LOQ
QUICK REFERENCE CHART

ICP COMMANDS
These commands affect data collection and the configuration of the instrument. For this reason they can only be issued from a terminal or window known as the *supervisor* process. The supervisor process is normally the window with the *dashboard* display on the workstation in the LOQ cabin.

The following commands are common to all ISIS instruments:

BEGIN
- starts a run

END
- stops a run and saves the data to a (•RAW) file

UPDATE
- copies a snapshot of the run in progress to the *CRPT* (Run 0)

PAUSE
- suspends data collection

RESUME
- restarts data collection after a PAUSE command

STORE
- saves the data in the CRPT to an intermediate (•SAV) data file without stopping a run in progress

ABORT
- stops a run abruptly (Warning: data is lost !)

CHANGE THICK x.x
- changes the pathlength of the sample stored in the "raw" data file to x.x mm

CHANGE TITLE " " " title " " "
- changes the title of the run shown on a dashboard display and stored in the "raw" data file for that run

LOAD setup.dat
(Note: seek advice before use !)
- loads the *ICP* with the configuration file called setup.dat LOQ has several such files but the three most important are:

NORMAL.DAT
- for normal 25Hz operation

FIFTYHZ_SHORT.DAT
- for 50Hz operation (Note: Reduced Q range !)

QUIET.DAT
- for operation on the *DAE* internal clock

CHANGE / NOTAB
- used to modify the *ICP* configuration after a LOAD command

The following commands are specific to LOQ:

DOSANS
- configures the instrument for normal (SANS) scattering measurements

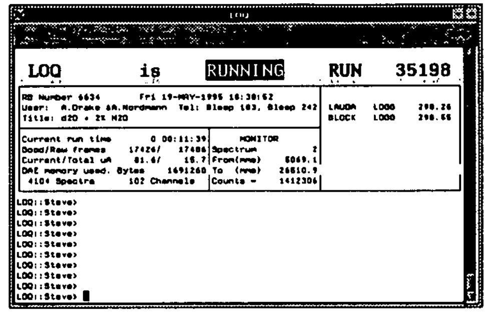
DOTRANS
- configures the instrument for (TRANS) transmission measurements

DOBIGSANS
- configures the instrument for high resolution SANS measurements (Note: seek advice before use !)

@ PILOT
- starts the sample changer command file PILOT.COM
- execute in the dashboard window!

DASHBOARD
This display tells you what LOQ is doing. You can create/remove a dashboard display by typing:

STAT ON or **STAT OFF**



CAMAC COMMANDS
These commands control and monitor sample environment equipment or other beamline equipment. They can be issued from any terminal or window.

CSET CHANGER z or **xxx.xx**
- moves the sample changer to the position called z or to xxx.x mm
- if using the "old" translator substitute the word SAMPLE for CHANGER

CSHOW CHANGER also **/ ENQ**
- shows the current position (and status with / ENQ) of the sample changer translator

CSET TEMP / LOG or **/ LOG=n**
- start computer logging of the device called TEMP at intervals of n secs
- n defaults to 60 seconds
- valid devices other than TEMP are; TEMP1, LAUDA, BLOCK and HAAKE

CSET TEMP / NOLOG
- stop computer logging of TEMP

CSET TEMP / RANGE=n xxx.x
- set the device called TEMP to a value of xxx.x ± (n / 2)
- valid devices other than TEMP are; TEMP1, LAUDA and HAAKE
- the / RANGE qualifier has no effect with the devices LAUDA or HAAKE
- the LAUDA bath only works in Kelvin, the HAAKE bath only works in Celcius

The following commands control the chopper and should not be used without authorisation:

STOPCHOP
- stops the chopper

STARTCHOP
- starts the chopper

PARKCHOP
- parks the chopper in open position

@ ANGLE n
- sets the opening of the chopper to n°
- n must be even & between 2 and 126

@ PHASE xxxx
- sets the chopper phase to xxxx µs

@ SPEED n
- sets the chopper speed to n Hz
- n is normally 25 or 50

SOFTWARE

COLETTE
- LOQ-specific data reduction & display program

GENIE
- standard ISIS data display program

VULOQ
- colour 2D & 3D data visualisation

FISH
- sophisicated model fitting program

SANDRA
- data manipulation utility

ADD (on LOQ) / **ADDLOQ** (on ISISE)
- raw data file addition utility

PILOT FILES
These are computer command files that *BEGIN* runs, wait for a specified time period or number of neutrons and then *END* runs for you. They are normally used with the sample changer. The files are called PILOT.COM and each time the file is edited the computer system creates a new version. You can edit a pilot file whilst one is in progress.

To edit a pilot file, type:
EDIT PILOT.COM

This is a screen editor. You can use the arrow keys or the mouse to move around. Please note that unlike PC or MAC editors the mouse has no other function in this editor!

Move down to the section of the file contained between the rows of "==" signs. This is the section you must edit. Every line must start with a $ sign, but if you want to include a remark, place a ! sign after the $ like this; "$! remark".

For every sample on the sample changer that you wish to run enter:
$MOVE z n "title" x.x "delay_type"

where z is the sample changer position, n is the run length (an integer!), x.x is the pathlength of the sample in mm, and the optional delay_type specifies what n relates to; "neutrons", "minutes" (1<mins<60) or the default (if omitted), microamp-hours (proton current x hrs).

Insert any labels (these can be used for iteration) as follows:
$label_name:

Add any flow control as follows:
$GOTO label_name

Please note that if you do not require any iteration, the last $MOVE line of your file should be followed by:
$GOTO END

Exit the pilot file:
[Control] - Z or
[PF1] - 7 EXIT [Enter]

EDITOR FUNCTION KEYS
Use the numeric keypad only!

[PF2]	help
[PF4]	delete line
[PF1] - [PF4]	replace line
[.]	select
[6]	cut
[PF1] - [6]	paste

GLOSSARY OF ISIS TERMINOLOGY

CAMAC
- the electronics controlling or monitoring your sample environment

CRPT
- Current Run Parameter Table

DAE
- Data Acquisition Electronics

DASHBOARD
- the instrument status display

DIRECT BEAM
- a transmission run on an empty sample position
- used to calculate sample transmissions
- also see *EMPTY BEAM*

EMPTY BEAM
- a scattering run on an empty sample position
- provides the instrument background scattering

ICP
- Instrument Control Program

PILOT FILE
- command file primarily used to control the sample changer
- usually the file PILOT.COM

SANS RUN
- a scattering run

SUPERVISOR
- process that controls the instrument

TRANS RUN
- a transmission run

MISCELLANEOUS INFORMATION

TELEPHONE / FAX NUMBERS
In an emergency at RAL dial 2222

RAL Switchboard
+44 (0)1235-821900

ISIS Fax Machine (24hr access)
+44 (0)1235-445720
This fax machine is located in the reading room on the top floor of R3

University Liason Secretariat (ULS)
+44 (0)1235-445592
Internal Extension 5592
Email ULS @ ISISE.RL.AC.UK
The ULS have their own fax machine:
+44 (0)1235-445103

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Internal Extension 6744
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Steve King's Office
+44 (0)1235-446437
Internal Extension 6437
Email SMK @ ISISE.RL.AC.UK

ISIS Main Control Room
+44 (0)1235-446789
Internal Extension 6789

LOQ Instrument Cabin
+44 (0)1235-446859
Internal Extension 6859

CRISP Instrument Cabin
+44 (0)1235-445898
Internal Extension 5898

SURF Instrument Cabin
+44 (0)1235-446894
Internal Extension 6894

Chemistry Laboratory (by LOQ)
+44 (0)1235-445192
Internal Extension 5192

Data Assessment Centre (DAC)
+44 (0)1235-446355
Internal Extension 6355

ISIS Computer Support
0585-286687
Internal Extension 3029
Email SUPPORT @ ISISE.RL.AC.UK

RESTURANT MEALTIMES

Monday - Friday

Breakfast	07:30 - 08:30
Lunchtime	11:45 - 13:45
Evening Dinner	17:15 - 19:15

Weekends

Breakfast	08:00 - 09:00
Lunchtime	12:00 - 13:00
Evening Dinner	18:00 - 19:00

R1 COFFEE LOUNGE
Monday - Friday
09:30 - 11:15 & 11:45 - 15:30

Coherent Scattering Lengths - I

(1 fm = 10^{-15} m = 10^{-13} cm)

Z	Nucleus	M	b (fm)
1	H	1.0079	-3.739_
1	D	2.016	6.671_
2	He	4.0026	3.26__
3	Li	6.941	-1.9___
4	Be	9.01218	7.79__
5	B	10.81	5.3___
6	C	12.011	6.646_
7	N	14.0067	9.36__
8	O	15.9994	5.803_
9	F	18.99840	5.654_
10	Ne	20.179	4.566_
11	Na	22.98977	3.58__
12	Mg	24.305	5.375_
13	Al	26.98154	3.449_
14	Si	28.0855	4.1534
15	P	30.97376	5.13__
16	S	32.06	2.847_
17	Cl	35.453	9.577_
18	Ar	39.948	1.909_
19	K	39.0983	3.67__
20	Ca	40.08	4.76__
21	Sc	44.9559	12.29__
22	Ti	47.9	-3.438_
23	V	50.9415	-0.3824
24	Cr	51.996	3.635_
25	Mn	54.938	-3.73__
26	Fe	55.847	9.54__
27	Co	58.9332	2.78__
28	Ni	58.7	10.3___
29	Cu	63.546	7.718_
30	Zn	65.38	5.68__
31	Ga	69.72	7.288_
32	Ge	72.59	8.185_
33	As	74.9216	6.58__
34	Se	78.96	7.97__
35	Br	79.904	6.795_
36	Kr	83.8	7.81__
37	Rb	85.4678	7.09__
38	Sr	87.62	7.02__
39	Y	88.9059	7.75__
40	Zr	91.22	7.16__
41	Nb	92.9064	7.054_
42	Mo	95.94	6.715_
43	Tc	97	6.8___
44	Ru	101.07	7.21__
45	Rh	102.9055	5.88__
46	Pd	106.4	5.91__
47	Ag	107.868	5.922_

Coherent Scattering Lengths - II

Z	Nucleus	M	b (fm)
48	Cd	112.41	5.1___
49	In	114.82	4.065_
50	Sn	118.69	6.225_
51	Sb	121.75	5.57__
52	Te	127.6	5.8___
53	I	126.9045	5.28__
54	Xe	131.3	4.92__
55	Cs	132.9054	5.42__
56	Ba	137.33	5.07__
57	La	138.9055	8.24__
58	Ce	140.12	4.84__
59	Pr	140.907	4.45__
60	Nd	144.24	7.69__
61	Pm	145	12.6___
62	Sm	150.4	0.8___
63	Eu	151.96	7.22__
64	Gd	157.25	6.5___
65	Tb	158.9254	7.38__
66	Dy	162.5	16.9___
67	Ho	164.9304	8.01__
68	Er	167.26	8.16__
69	Tm	168.9342	7.07__
70	Yb	173.04	12.43__
71	Lu	174.967	7.21__
72	Hf	178.49	7.77__
73	Ta	180.9479	6.91__
74	W	183.85	4.86__
75	Re	186.2	9.2___
76	Os	190.2	10.7___
77	Ir	192.22	10.6___
78	Pt	195.09	9.6___
79	Au	196.9665	7.63___
80	Hg	200.59	12.692__
81	Tl	204.37	8.776__
82	Pb	207.2	9.405__
83	Bi	208.9804	8.532__
84	Po	209	0.____
85	At	210	0.____
86	Rn	222	0.____
87	Fr	223	0.____
88	Ra	226.0254	10.____
89	Ac	227.028	0.____
90	Th	232.0381	10.52__
91	Pa	231.0359	9.1___
92	U	238.029	8.417_
93	Np	237.0482	10.55__
94	Pu	244	0.____
95	Am	243	8.3___
96	Cm	247	0.____

Scattering Length Densities

The intensity of small-angle neutron scattering from component 1 relative to component 2 is directly proportional to the square of the difference in scattering length densities between the two components (or what is often called "the contrast")

$$contrast = (\Delta\delta)^2 = (\delta_1 - \delta_2)^2$$

where the scattering length density of one molecule of component z may be readily calculated from the simple expression

$$\delta_z = \sum_{i=1}^{N} b_i \times \frac{D\,N_A}{M}$$

Here b_i is the scattering length (see the previous table) of atom i, N is the total number of atoms in the molecule, and D is the bulk density (in g cm^{-3}) and M is the molar mass (in g mol^{-1}) of component z. N_A is Avogadro's number. When the component in question is a polymer it is only necessary to calculate δ for one repeat unit. δ has dimensions of $(length)^{-2}$ and is normally expressed as 10^{10} cm^{-2} or 10^{-6} $Å^{-2}$. It can also be negative (particularly if it contains a lot of hydrogen).

It should be noted that the scattering length density is remarkably sensitive to the value of the density used in its calculation and so a reliable knowledge of the density is a pre-requisite for a successful experiment.

Some illustrative scattering length densities of common materials are given in the tables below.

Substrates

	Density (g cm^{-3})	δ (10^{10} cm^{-2})
Silicon	2.33	+2.07
TiO_2	4.1	+2.57
SiO_2	2.0	+3.15
Quartz	2.2	+3.47

Solvents

	(protonated form)		*(perdeuterated form)*	
	Density (g cm^{-3})	δ (10^{10} cm^{-2})	Density (g cm^{-3})	δ (10^{10} cm^{-2})
Water	0.997	-0.56	1.104	+6.38
Octane	0.703	-0.53	0.815	+6.43
Cyclohexane	0.779	-0.28	0.89	+6.70
Toluene	0.865	+0.94	0.943	+5.67
Chloroform	1.492	+2.38	1.5	+3.16
Carbon Tetrachloride	1.594	+2.81	1.594	+2.81

Polymers

	(protonated form)		*(perdeuterated form)*	
	Density (g cm^{-3})	δ (10^{10} cm^{-2})	Density (g cm^{-3})	δ (10^{10} cm^{-2})
Poly(butadiene)	0.89	+0.41	0.89	+6.60
Poly(ethylene)	0.92	-0.33	1.095	+8.24
Poly(dimethylsiloxane)	0.98	+0.06	1.06	+5.04
Poly(ethylene oxide)	1.127	+0.64	1.23	+7.06
Poly(methyl methacrylate)	1.22	+1.10	1.22	+7.22
Poly(styrene)	1.05	+1.41	1.12	+6.47

Pilot Files

What are Pilot Files?

Pilot files are what we call computer command files that BEGIN runs, wait for a specified time period or number of incident neutrons, and then END runs automatically. They are a particularly efficient method of controlling the sample changer (see Pages 3-9 and 3-11), although it is possible to write variants for other tasks (such as temperature scanning a single sample, for example). This description assumes that you are using the sample changer.

To write a pilot file involves writing a short computer program in a language called DCL (DEC Command Language) - the language of the DEC VMS operating system running on the LOQ FEM (see Section 2.23). Although DCL is a fairly straightforward language to learn, to save time we have provided a template command file called PILOT.COM - the ".COM" extension signifying that this is a command file - for you to edit. Consequently you will need to know a little about the VAX editor. (If you are conversant with VAX/VMS computer systems then you can skip the next section).

The VAX Editor

The VAX editor you will use is a screen editor. To move around the screen, use the "arrow" cursor keys, the "page up" and "page down" keys or the mouse (click the left mouse button to move the cursor to the pointer position). Please note that unlike a PC or MAC computer system, the mouse has no other function in this editor.

To create a new line, press:

{Return}

To QUIT the file (losing any changes), type:

{PF1}, 7, QUIT, {Return}

To EXIT the file (saving any changes), type:

{PF1}, 7, EXIT, {Return} or {Ctrl}-Z
This creates a new version of the file; ie, it creates PILOT.COM;n+1

Other functions are (use the numeric keypad):

{PF2}	Help
.	Select
6	Cut the selected region
{PF1}, 6	Paste the cut selection
{PF4}	Delete line
{PF1}, {PF4}	Replace deleted line

Editing a Pilot File

To begin editing the template, type:

EDIT PILOT.COM
or **EDIT/EDT PILOT.COM**

Just specifying PILOT.COM is equivalent to specifying PILOT.COM;n where n is the most recent version number.

The cursor will be at the top of the file. You will notice that every line starts with a "$" sign, but that in some cases the dollar sign is followed by a "!" mark. The "$" tells the computer to interpret the line, but the "!" specifies that the remainder of the line is a remark or comment. There are a lot of comments, but you will also observe that there are several lines of DCL code. Take care not to delete or to insert characters in these lines as you will seriously affect the operation of the pilot file.

Move the cursor down through the file until you reach the section contained between the two rows of equals signs:

```
$!=================================================================
.
$!                    This is the region that you must edit
.
$!=================================================================
```

For every run that you wish to conduct - whether it be multiple runs on a few samples or single runs on several samples - enter:

$MOVE position length "title" x.x "delay_type"

The MOVE command is followed by 5 parameters. These parameters must be separated from one another, and from the MOVE command, by a space, and must be specified in the order shown. The "delay_type" parameter is optional however (see below).

The parameters are:

position
- the sample changer position. This may be given as the code displayed on the sample rack (eg, "A", "B", "C", etc) or as an absolute distance in mm.

length
- the run length (as an integer value!). This may be given as incident neutrons, minutes (though this is only programmed for 1<minutes<60, i.e. 59 minutes is the maximum run time in this mode, and note also that if you use this option the run will progress even if the beam is down!) or, the default (see below), is microamp-hours. Microamp-hours are the product of the ISIS proton current and time (in hours). Thus, if the accelerator is operating at 200 μA, and LOQ is at 25 Hz, there will be 100 microamp-hours to the hour.

"title"
- the title for the run. This will be displayed both on the LOQ dashboard status display and written into the raw data file.

x.x
- the neutron pathlength through the sample in mm. This will normally be the same as the sample thickness.

"delay_type"
- the units for the *length* parameter. If the "delay_type" parameter is omitted then the computer will assume that the *length* parameter relates to microamp-hours. For *length* to be in incident neutrons enter "NEUTRONS", and for *length* to be in time enter "MINUTES".

For example:

$!==
$! Move to Position A and wait for 95 microamp-hours (approximately 1 hour)
$MOVE A 95 "Sample 1" 2.0
$! Move to Position SIX (note that this is not the same as a position of 6.0 mm)
$! and wait for 7 million (70 x 100000) neutrons (approximately 1 hour)
$MOVE SIX 70 "Sample 2" 1.0 "NEUTRONS"
$! Move to a position of 123.45 mm and wait for 59 minutes
$MOVE 123.45 59 "Sample 3" 1.27 "MINUTES"
$!==

If the sequence of runs you have entered is only to be executed <u>once</u>, then insert the following line after the last MOVE command:

$GOTO END

If, on the other hand, you want the pilot file to cycle - useful for overnight running - then after the last MOVE command enter:

$GOTO LOOP

and immediately above the <u>first</u> line you want repeated enter the corresponding label (note that the colon ":" is essential) like so:

$LOOP:

So, now consider a more useful example:

$!==
$DOTRANS
$MOVE A 10 "Sample 1 Trans" 2.0
$MOVE B 10 "Sample 2 Trans" 2.0
$MOVE C 10 "Sample 3 Trans" 2.0
$DOSANS
$LOOP:
$MOVE A 95 "Sample 1 Sans" 2.0
$MOVE B 95 "Sample 2 Sans" 2.0
$MOVE C 95 "Sample 3 Sans" 2.0
$GOTO LOOP
$!==

Upon execution the first command encountered is DOTRANS. This instructs the LOQ FEM to reconfigure LOQ for transmission measurements. Transmission runs are then run on three samples, after which the DOSANS command reconfigures LOQ for scattering measurements.

The pilot file then conducts a sequence of scattering measurements on each of the three samples <u>and continues to do so until the pilot file is interrupted</u>. Notice that the transmission runs are much shorter than the scattering runs. This is normal. As a general rule of thumb, transmission measurements only need to run for 5 - 10 minutes; though the actual length of time will of course depend on the nature of the particular sample. Also note that the DOSANS command is placed above the label "LOOP:". This is not critical, but there is no point in making the computer set LOQ up for scattering measurements when it is already in the correct configuration - you would just be wasting time!

Starting a Pilot File

To start a pilot file, gain input focus in the *Supervisor* window - on LOQ this is almost always the terminal window with the "Dashboard" status display on the LOQ colour workstation monitor - and type:

LOQ> **@PILOT**

Interrupting and Restarting Pilot Files

When you start a pilot file, a copy is loaded into the computers memory. This means that you can re-edit a pilot file whilst it is running, without affecting the operation of the instrument.

To stop, or interrupt, the pilot file that is running, gain input focus in the *Supervisor* window and type:

{Ctrl}-Y

The computer will beep at you and tell you that you have succeeded! So far, so good. However, if a run was in progress when you interrupted the pilot file you will find that it will still be in progress even when the pilot file is not. This run can be terminated manually simply by typing END, but what if you want to re-start a slightly modified pilot file without stopping the run in progress? All it is necessary to do, is to ensure that the first <u>executable</u> MOVE line that the computer encounters in the revised pilot file is the one for the run in progress. You can even change the length, title, pathlength and delay type <u>but don't under any circumstances change the sample changer position code</u>! For example;

Suppose that the original pilot file...

```
$!=================================================================
$MOVE  A  95  "Sample 1"  2.0
$MOVE  B  95  "Sample 2"  2.0
$MOVE  C  95  "Sample 3"  2.0
$GOTO END
$!=================================================================
```

... is interrupted during execution of the run "Sample 2" to insert a new sample, "Sample 4", and to change some run lengths. The new pilot file would then be...

```
$!=================================================================
$!MOVE  A  95  "Sample 1"  2.0  (the completed run is commented out, or it could be deleted)
$MOVE  B  95  "Sample 2"  2.0
$MOVE  C  190  "Sample 3"  2.0
$MOVE  D  190  "Sample 4"  2.0
$GOTO END
$!=================================================================
```

Notes

Section 1

Technical Information

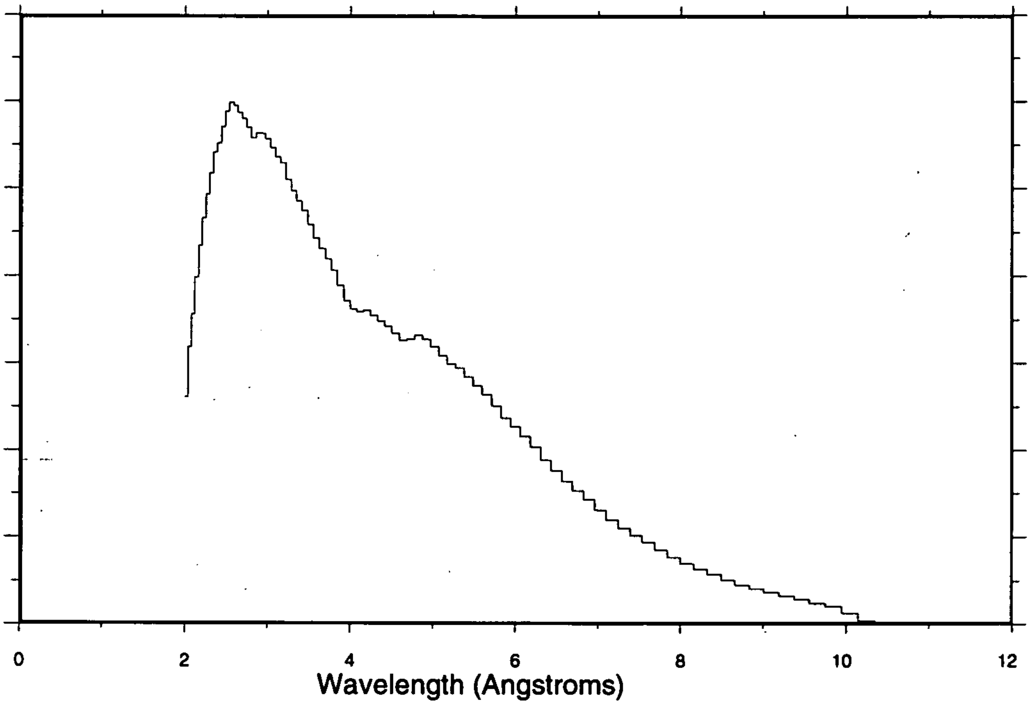
Contents

Wavelength Distribution

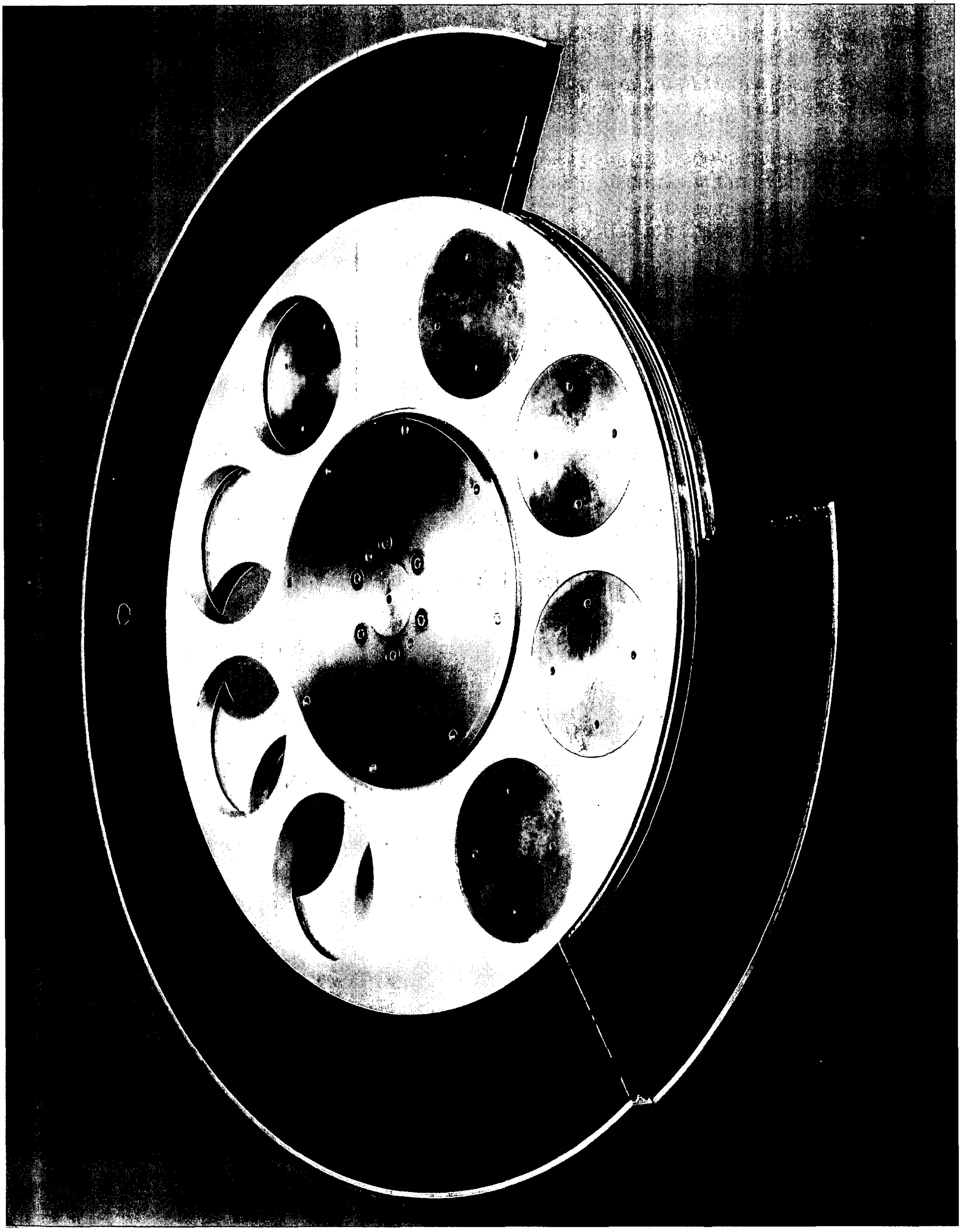
Shown below is the normal distribution of neutron wavelengths from the hydrogen (cold) moderator as seen at the sample position on LOQ. This range is determined by the sector opening and rotational frequency of the LOQ chopper, usually 126° and 25Hz, and is the broadest distribution possible when ISIS is operating normally - remember that ISIS operates at 50Hz - which still avoids *frame overlap* (a condition where the short wavelength neutrons from pulse n+1 overtake the long wavelength neutrons from pulse n!).

By changing the sector opening of the LOQ chopper and/or its rotational frequency any wavelength band from this distribution can be selected. This aspect can be quite useful for certain types of experiment or certain samples. For example, some metal alloys and ceramics Bragg scatter short wavelength neutrons so strongly that the SANS signal can be masked. By operating LOQ at 50Hz and only collecting data with the long wavelength half of the distribution the multiple Bragg edges can be avoided whilst data collection every pulse recovers some of the flux lost by excluding the short wavelength neutrons. Conversely, if the very low-Q scattering (below approximately 0.01$Å^{-1}$) is not needed, operating LOQ at 50Hz and only collecting data with the short wavelength neutrons almost doubles the flux. Finally, by closing down the sector opening of the chopper to a few degrees LOQ can operate quasi-monochromatically. In such instances the count rate is too low to conduct a reactor-instrument type of SANS experiment, but this mode of operation has been used successfully to study inelastic effects during a SANS event.

When ISIS operates at 25Hz or less (for example, during periods of *machine physics*) it is possible to stop the LOQ chopper and to park it open. In these instances the limits of this distribution are determined by the Soller supermirror bender (at short wavelengths) and the frame overlap mirrors (at long wavelengths). These limits are approximately 1.3Å and 13Å respectively. The hydrogen moderator actually produces significant numbers of neutrons with wavelengths in excess of 13Å but these cannot be utilised on LOQ.



The LOQ double-disc wavelength-limiting chopper (seen here out of its casing).
[Negative 90FC3221]



Count Rates

The *count rate* is simply the number of neutrons recorded by a detector in a given time period. From the user's perspective the most important count rate is that measured by a beam-sampling monitor detector (see Page 1-4) placed just before the sample. Note however that since this number depends on the efficiency of the monitor, the choice of collimation and varies with the proton beam current and target type (at ISIS) or power level (at a reactor) it is a parameter specific to a particular instrument at a particular facility and cannot reliably be used to compare different SANS instruments. Such comparisons must be done using the *flux*. This is an absolute number normally determined from the induced activity of an irradiated foil.

The count rate is of interest because it relates the length of time a sample is run for to the counting statistics (the size of the error bars).

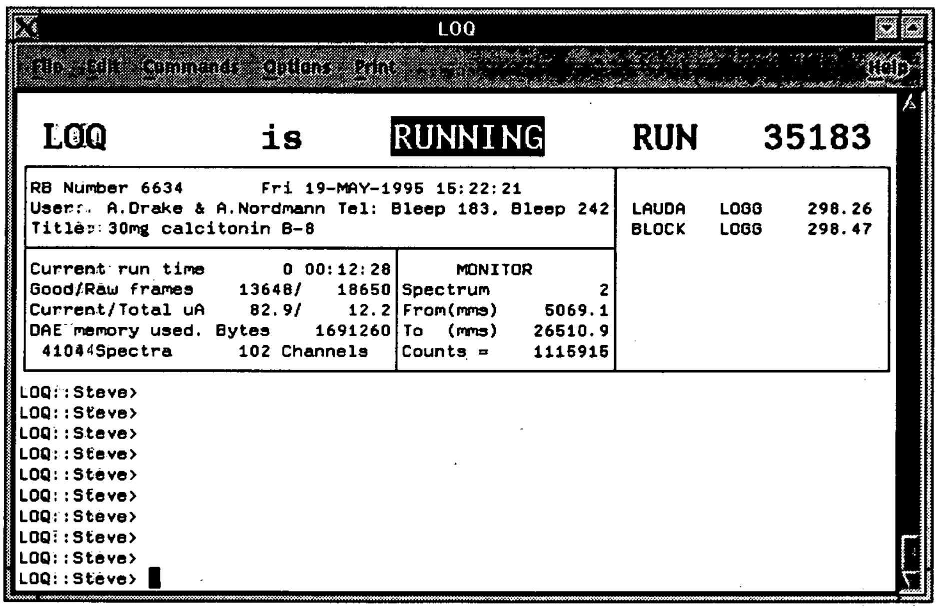
There several ways of specifying a samples run-time. One could use time (though that has drawbacks, for instance when the beam goes down!). The method most frequently employed at reactor-based SANS instruments is to use actual neutrons (as recorded by the incident beam monitor). This method can be used on LOQ, but at ISIS a more sensible practice is to use *microamp-hours*, that is, proton current multiplied by time (ie, the integrated proton charge).

On LOQ the number of microamp-hours and neutrons at Monitor 2 when ISIS is operating at 200 μA (the ISIS proton current is displayed on the red matrix sign in the Experimental Hall) is:

Tantalum Target	**100 μA Hours**	**7.96 ± 0.42 M counts Hour^{-1}**
Uranium Target	**100 μA Hours**	**13.74 ± 0.60 M counts Hour^{-1}**

These figures assume LOQ is operating at 25 Hz. At 50 Hz, the microamp-hour figures should be doubled, whilst the counts-per-hour figures should be multiplied by the relative count rates shown in the table overleaf.

The numbers of microamp-hours and neutrons that a sample has "seen" are displayed in the lower two boxes of the *Dashboard* display (see below) as *Total uA* and *Counts* respectively.



LOQ Configurations

LOQ is normally operated in one of the following three basic configurations. In decreasing order of usefulness, they are:

Instrument Configuration File (LOAD filename)	Chopper Frequency (Hz)	Chopper Phase (μs)	Approximate Wavelengths (Å)	Approximate Q-range ($Å^{-1}$)	Relative Count Rate
NORMAL.DAT	25	5020	2.2 - 10.0	0.008 - 0.24*	1.00
FIFTYHZ_SHORT.DAT	50	5020	2.6 - 6.7	0.012 - 0.22	1.45
FIFTYHZ_LONG.DAT	50	10800	6.3 - 10.0	0.008 - 0.07	0.17

Please note that the chopper phases in the above table are illustrative examples only. Actual phases may differ from the above values, particularly after chopper maintenance, by a few hundred μs.

All of the data in the above table assumes a chopper sector opening of 126 °.

Other configurations of LOQ may be possible, please consult the instrument scientists.

*Note: 0.008 - 1.4 $Å^{-1}$ from mid-1996

LOQ Detectors

Three sets of detector are used on LOQ; *monitors*, a *main detector* and a *high-angle bank*.

MONITORS

- Are scintillator glass detectors operating at ~1 kV (see page 2-11).
- There are 2 types.
- Low-efficiency (bead array) monitors. These are used for sampling the neutron wavelength distribution of the beam before the sample and therefore sit in the beam permanently. LOQ has two of these monitors; one (called Monitor 1 or just M1) is 6.356 m from the moderator, the other (M2) is 10.431 m from the moderator.
- High-efficiency (slab) monitors for sample transmission measurements. LOQ has one of these monitors (M3 or Tx Mon) situated at 11.497 m from the moderator. It is inserted after the sample as required.
- The detection principle is:

$${}^{1}_{0}n + {}^{6}_{3}Li \longrightarrow {}^{3}_{1}T + {}^{4}_{2}\alpha + 4.79MeV\ \gamma \xrightarrow[Ce^{III} \text{ or } ZnS]{} (\text{light}) \longrightarrow PMT$$

MAIN DETECTOR

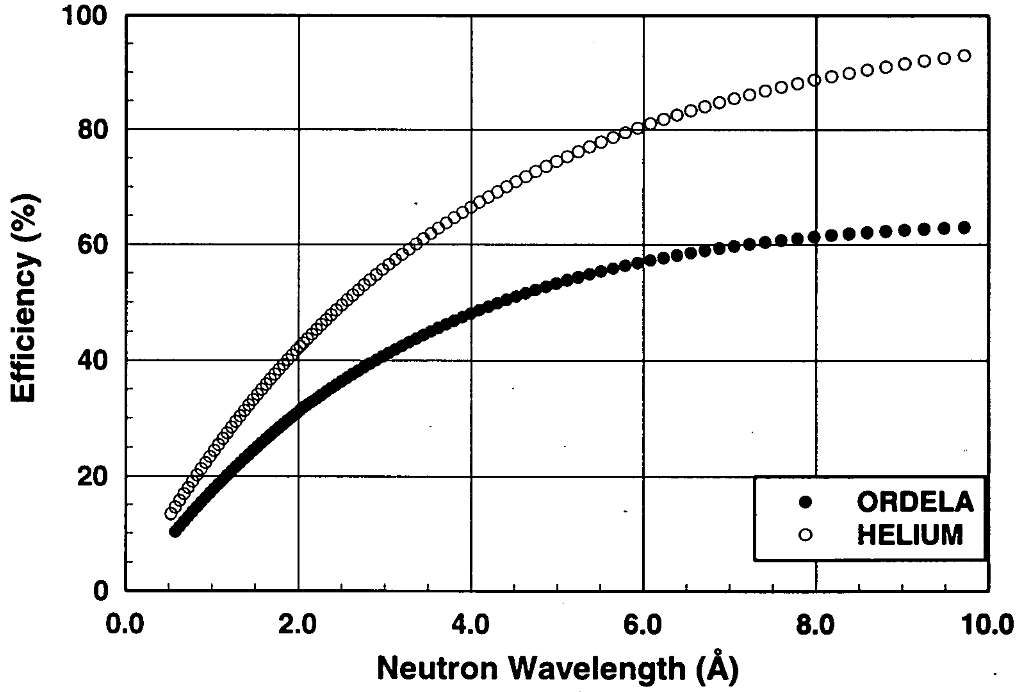
- Is a position-sensitve (or "area") detector 15.150 m from the moderator.
- Is a gas detector (65% ^{3}He and 35% CF_4 at ~1atm) operating at ~4 kV (see Page 2-11).
- Is 1.2 m in diameter with a central neutron-sensitive region 64 × 64 cm square.
- Has a spatial resolution of 1 cm^2.
- The detection principle is:

$${}^{1}_{0}n + {}^{3}_{2}He \longrightarrow {}^{3}_{1}T + {}^{1}_{1}p + 0.77MeV\ \gamma \longrightarrow (25000 \text{ free electrons}) \longrightarrow$$

$$\longrightarrow (250 \text{ electron-ion pairs per free electron at each cathode}) \longrightarrow$$

$$\longrightarrow (\text{current pulse}) \longrightarrow (\text{rise-time coded for position})$$

The graph below shows the detection efficiency of the ORDELA main detector (allowing for both the 5 mm thick fused silica entrance window to the secondary flight path and the 12 mm thick aluminium pressure window on the detector itself) compared to the detection efficiency of the gas fill alone.



HIGH-ANGLE DETECTOR BANK

- Is an annular area detector 11.6 m from the moderator.
- Is a 4-quadrant, ZnS-type, scintillator glass detector.
- Has 1.2 x 1.2 cm square pixels.
- Overlaps with the main detector Q-range between $0.15 \le Q \le 0.24$ $Å^{-1}$.

LOQ Distances

L_{m-s} = moderator-to-sample distance (metres)

L_{s-d} = sample-to-detector distance (metres)

Sample Environment	L_{m-s}	L_{s-d}
New LOQ Sample Changer (Hellma Cell Racks)	**11.048**	**4.102**
(when base table is in position for the Poiseuille Cell)	11.025	4.125
New LOQ Sample Changer (4-position racks)	**11.063**	**4.087**
Old LOQ Sample Changer (Hellma Cell Racks)	11.046	4.104
Old LOQ Sample Changer (4-position racks)	11.061	4.089
Couette Shear Cell	11.039	4.111
Poiseuille Flow Cell (base table moved upstream by one hole for back feet)	11.110	4.040
Durham Heated Rack	11.034	4.116
RAL Furnace in TF	10.96	4.19
Goudsmit Magnet	10.87	4.28
Calibration Hole	11.100	4.050

These are the lengths that the COLETTE data reduction program will use in order to calculate your *Q*-scale, so it is in your own interests to make sure that you use the right values!

Prior to the 3rd October 1995, $L_{m-s} + L_{s-d}$ = 15.473 m. L_{m-s} values did not change.

LOQ Aperture Selector Codes

The diameter quoted refers to the size of the hole used to collimate the incident neutron beam.

Aperture 2 Code	Diameter (mm)		Aperture 1 Code	Diameter (mm)
01 (TRANS)	01.0 (Cd)		01	40.0
02	11.0		02	No Hole
03	01.5 (Cd)		03	02.0
04	14.0		04	03.0
05	17.0		05	04.0
06	20.0		06	05.0
07	22.0		07	06.0
08	24.0		08	07.0
09 (SANS)	12.0 (Cd)		09	08.0
10	30.0		10	09.0
11	40.0		11	10.0
12	No Hole		12	11.0
13	02.0		13	12.0
14	03.0		14	14.0
15	04.0		15	17.0
16	00.5 (Cd)		16 (Normal)	20.0
17	06.0		17	22.0
18	07.0		18	24.0
19	08.0		19	27.0
20	09.0		20	30.0

The position of Aperture #1 is not normally changed during routine experiments, however Aperture #2 frequently swaps between two positions; one for scattering (SANS) measurements and one for transmission (TRANS) measurements.

The changeover of Aperture #2 is normally initiated from the LOQ computer by the *DOSANS* and *DOTRANS* commands, but may also be initiated manually by pushing the appropriate buttons on the Aperture Control Crate (see Section 2.18). The positions of Aperture #2 during "SANS" and "TRANS" measurements are determined by the aperture codes shown above. These codes should be entered on the Aperture Control Crate with the thumbwheel switches.

Calibration of LOQ Data

The LOQ data reduction program, COLETTE (see Section 4), ultimately generates data in the form **differential cross-section** *(dσ/dΩ)(Q)* - commonly but inaccurately referred to as *I(Q)* **- as a function of *Q*.**

The quantity *(dσ/dΩ)(Q)* has units of cm^{-1}, and the values you obtain from COLETTE are near enough on an absolute scale (typically they are within ± 5%). However, if you intend to derive quantitative results from the cross-section data (using Zimm plots, for example) then you should make the time to run one of the LOQ standard samples (and an appropriate background) in order to check the instrument calibration.

Suitable standards are problematical on a time-of-flight SANS diffractometer because of the possibility of wavelength-dependent effects. Following Wignall & Bates (J. Appl. Cryst., *1987*, 20, 28) we choose to use solid blends of hydrogenous and perdeuterated polystyrene homopolymers of the same molecular weight. The scattering from such systems is relatively simple, extends over a reasonable *Q*-range and follows the well-known Debye scattering law:

$$\frac{d\sigma}{d\Omega}(Q) \propto \frac{2[\exp(-u)+u-1]}{u^2} \quad \text{where } u = (Q \times Radius\ of\ Gyration)^2$$

For a partially-deuterated polystyrene blend it can be shown that:

$$\frac{d\sigma}{d\Omega}(Q=0) \approx 4.0331\times10^{-3}\ x\,(1-x)\,M_w$$

where *x* is the weight fraction of perdeuterated polystyrene in the blend and M_w is its weight-average molecular weight. Thus, to calibrate your data it is simply necessary to multiply it by a calibration constant, *K*, generated from your standard sample runs:

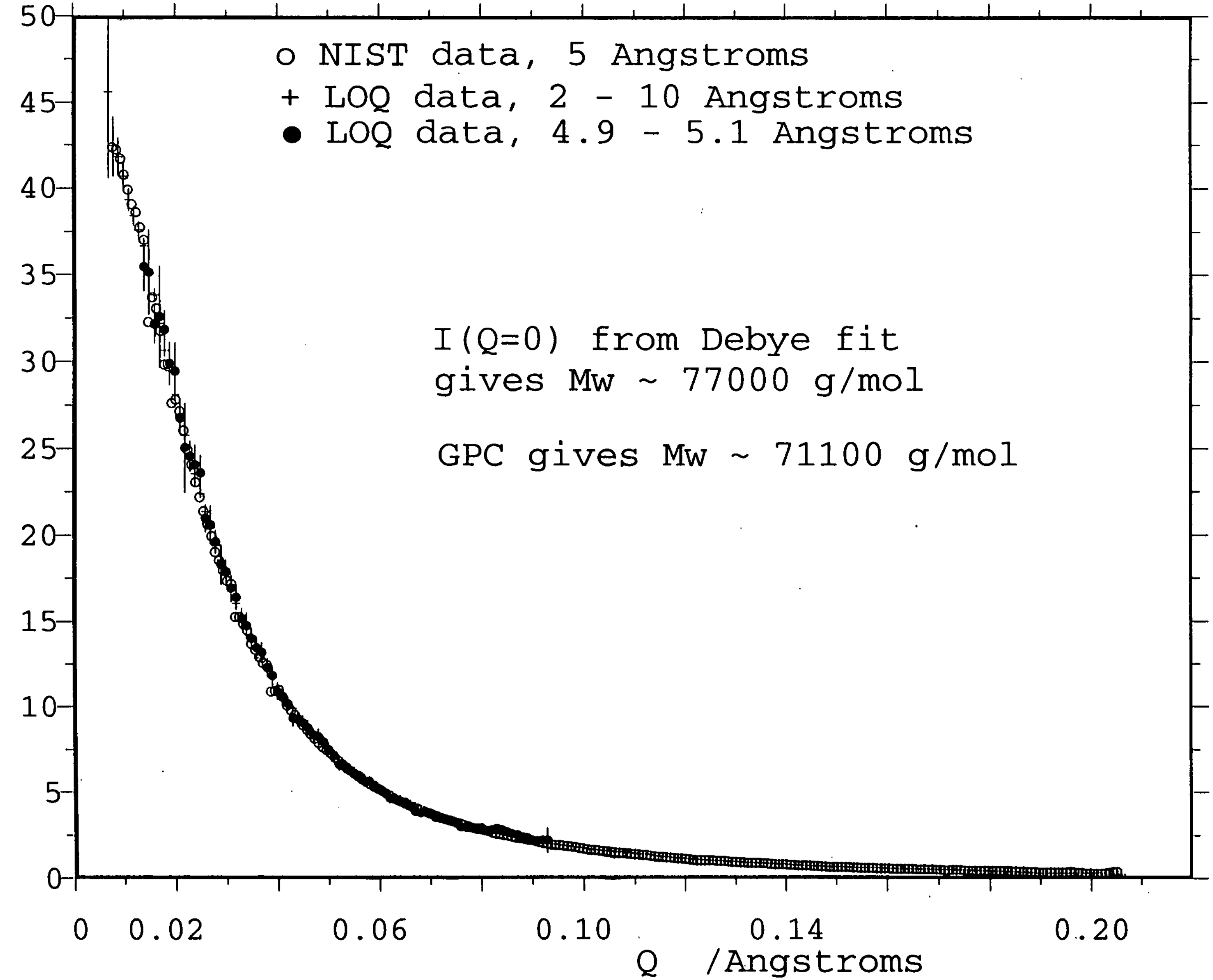
$$K = \frac{\frac{d\sigma}{d\Omega}(Q=0)_{Expected}}{\frac{d\sigma}{d\Omega}(Q=0)_{Measured}} = \frac{I^*}{\frac{d\sigma}{d\Omega}(Q=0)_{Measured}}$$

There are two standards in use on LOQ, imaginatively called "Old" (or "GDW20") and "New" (or "TK48/49")! The table below details their characteristics. "Path" is the sample thickness in mm.

Standard	Path	x	M_w (g mol^{-1})	R_g (Å)	Background	I^* (cm^{-1})
OLD	1.27	0.198	65600	70.8	Empty Beam	42.0
NEW	1.035	0.490	77500	76.7	hPS-dPS random copolymer	78.1

The thickness of the hPS-dPS random copolymer is 1.07 mm.

The transmissions of the "Old" and "New" standards, and the copolymer background, are pre-programmed into COLETTE and may be invoked using the TR/STANDARD, TR/BLEND and TR/COPOLYMER commands, respectively, in place of the normal TR/MEASURED.



o NIST data, 5 Angstroms
+ LOQ data, 2 - 10 Angstroms
● LOQ data, 4.9 - 5.1 Angstroms
I(Q=0) from Debye fit
gives Mw ~ 77000 g/mol
GPC gives Mw ~ 71100 g/mol
50
45
40
35
30
25
20
15
10
5
0
0
0.02
0.06
0.10
0.14
0.20
Q /Angstroms

Notes

Section 2

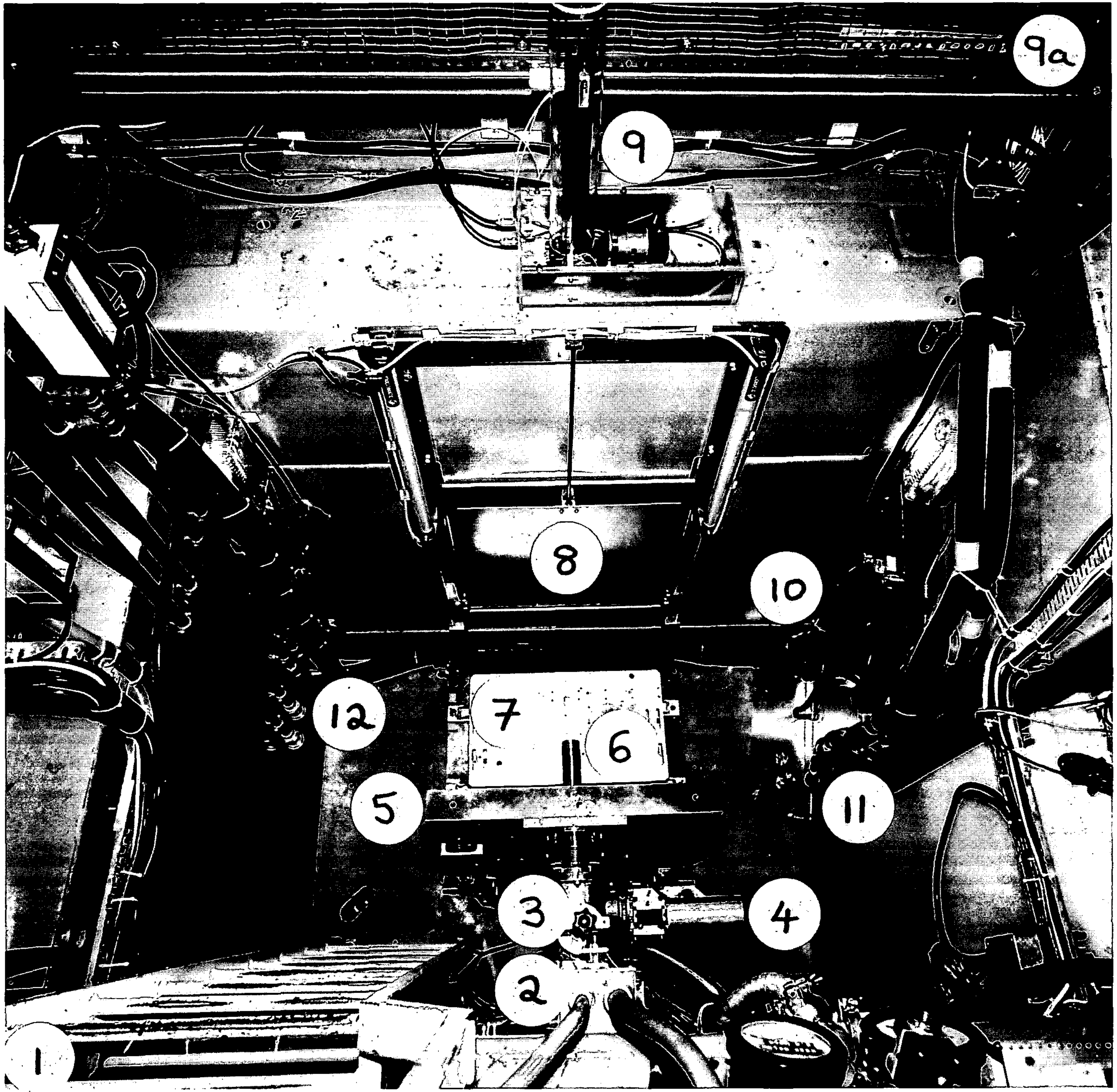
Instrumentation

Contents

Notice to LOQ Users

This section identifies several pieces of calibrated electronic equipment. Unless directed to do so, please do not alter the settings of any equipment you are unfamiliar with.

Thank you



9a
9
8
10
7
6
12
5
11
3
4
2
1

The LOQ Sample Position - I. *[Negative 95RC4891]*

2.1 Access Ladder

2.2 Proton Beam Off Button

- rotate collar to release if accidentally pushed in!
- push this button if the white light goes off, or switches to blue, whilst you are inside the sample enclosure, then climb out and contact the Main Control Room on x6789.

2.3 Incident Beam Monitor #2

2.4 Sample Alignment Laser

- rotate the small handwheel to insert/remove the prism.
- ensure that the prism is <u>out</u> before opening the shutter!

2.5 Aperture Wheel #2

2.6 Collimation Snout

- <u>Warning</u>: this is normally under vacuum! (Also see next page).
- adjustable length with cadmium aperture.
- also see Sections 3.22 and 3.23.

2.7 Sample Mounting Table

- also see Section 3.1.
- shown with "Table Top" in place (thickness 25 mm).

2.8 Safety ("Micro") Shutter

- should be <u>down</u> when you are in the sample enclosure.
- should move up when the Master Personnel Interlock (in the green interlock box on the outside of the enclosure fence) is activated.

2.9 Transmission Monitor

2.9(a) Transmission Monitor Motor Control Box (partially obscured by fence)

- do not interfere with this box!
- in normal operation the lefthand switch should be on "*Stop*" and the righthand switch on "*Remote*".

2.10 Goudsmit Electromagnet Power Cables & Connection Box

- also see Section 3.50.
- access to the "power tails" connection box (the brown box above the cables) is interlocked. The interlock key is on the righthand wall of the LOQ computer room.

2.11 Fluid Bath Hoses

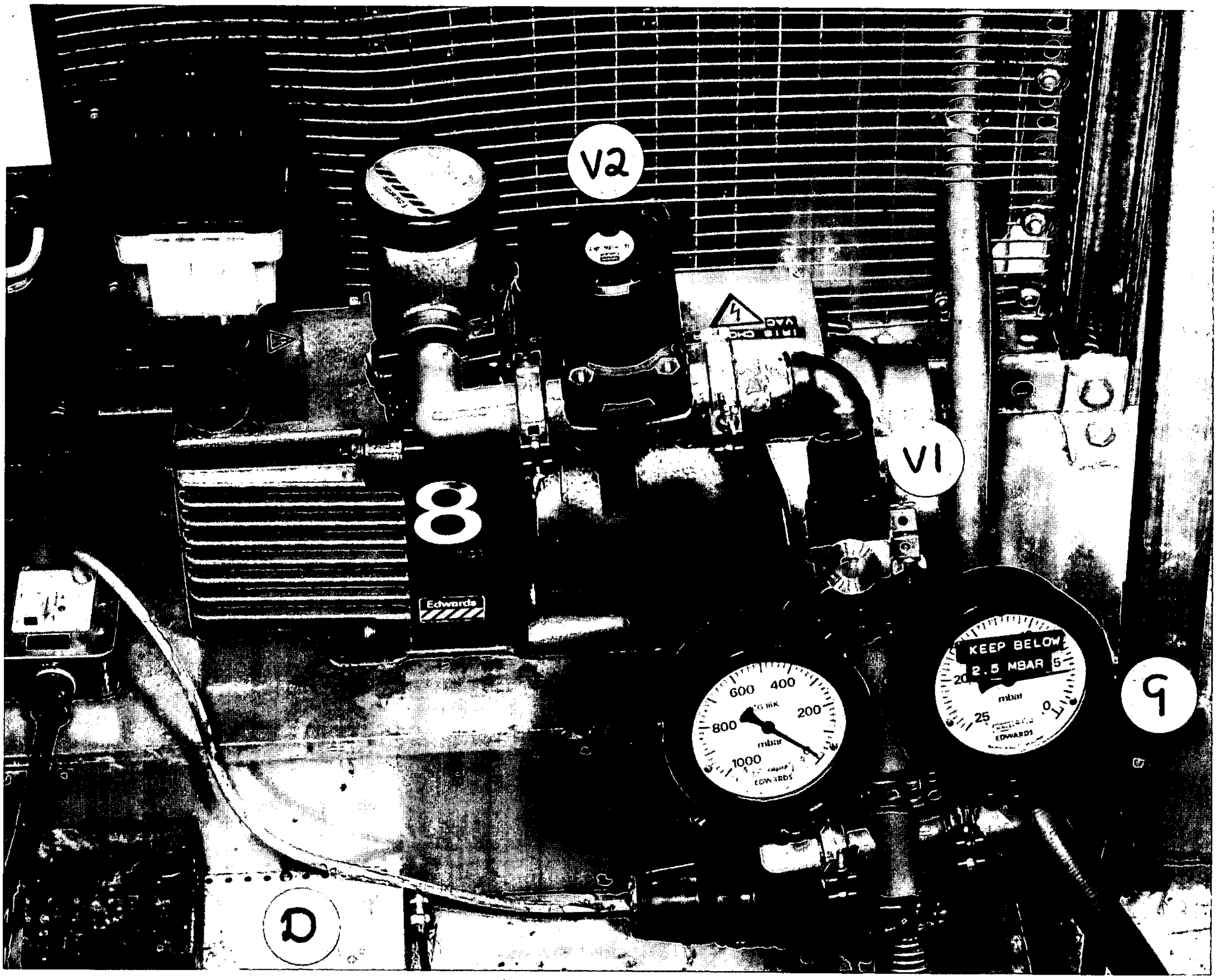
- for connecting circulating fluid baths to the sample changer block, etc.
- <u>yellow</u> for water/glycol, <u>red</u> for oil. Do not mix the two!
- also see Sections 3.41 and 3.47.

2.12 Cooling Water Supplies

- for use with magnets and furnaces, etc.
- there are two sets, terminating in different size connectors to avoid accidental use; a general purpose "tower water" supply (furthest from the ladder), and a "demineralised" supply (nearest to the ladder) which (at the time of writing) is only for use with the Goudsmit Electromagnet (see Section 3.50).
- emergency water shut-off valves are located outside of the enclosure fence; <u>red</u> handles (in plain view) for the <u>demin water</u>, and <u>green</u> handles (under the patch panel, see Section 2.46) for the <u>tower water</u>. The tower water supply also has a pair of red-handled stopcocks inside the sample position (under the flowmeter)

<u>Not shown</u>:

Compressed air (yellow-handled shut-off valves) and *CCR* (see Pages 3-39 and 3-41) *Services Panel* on the left wall of the enclosure (viewed from the ladder).


V2
V1
8
Edwards
KEEP BELOW
2.5 MBAR
mbar
EDWARDS
600
400
200
800
1000
20
25
5
0
9
D

LOQ Sample Position - II. *[Negative 95RC2658]*

The picture shows the region of the LOQ Sample Position immediately adjacent to the gate.

The vacuum pump is responsible for maintaining a rough vacuum in the primary flight path. The vacuum level is indicated on *Gauge G.* As a rule of thumb this should always be < 5 mbar, ideally < 2.5 mbar, if you are collecting data when ISIS is running.

To evacuate the primary flight path
Close *Valve V1* and open *Valve V2.*

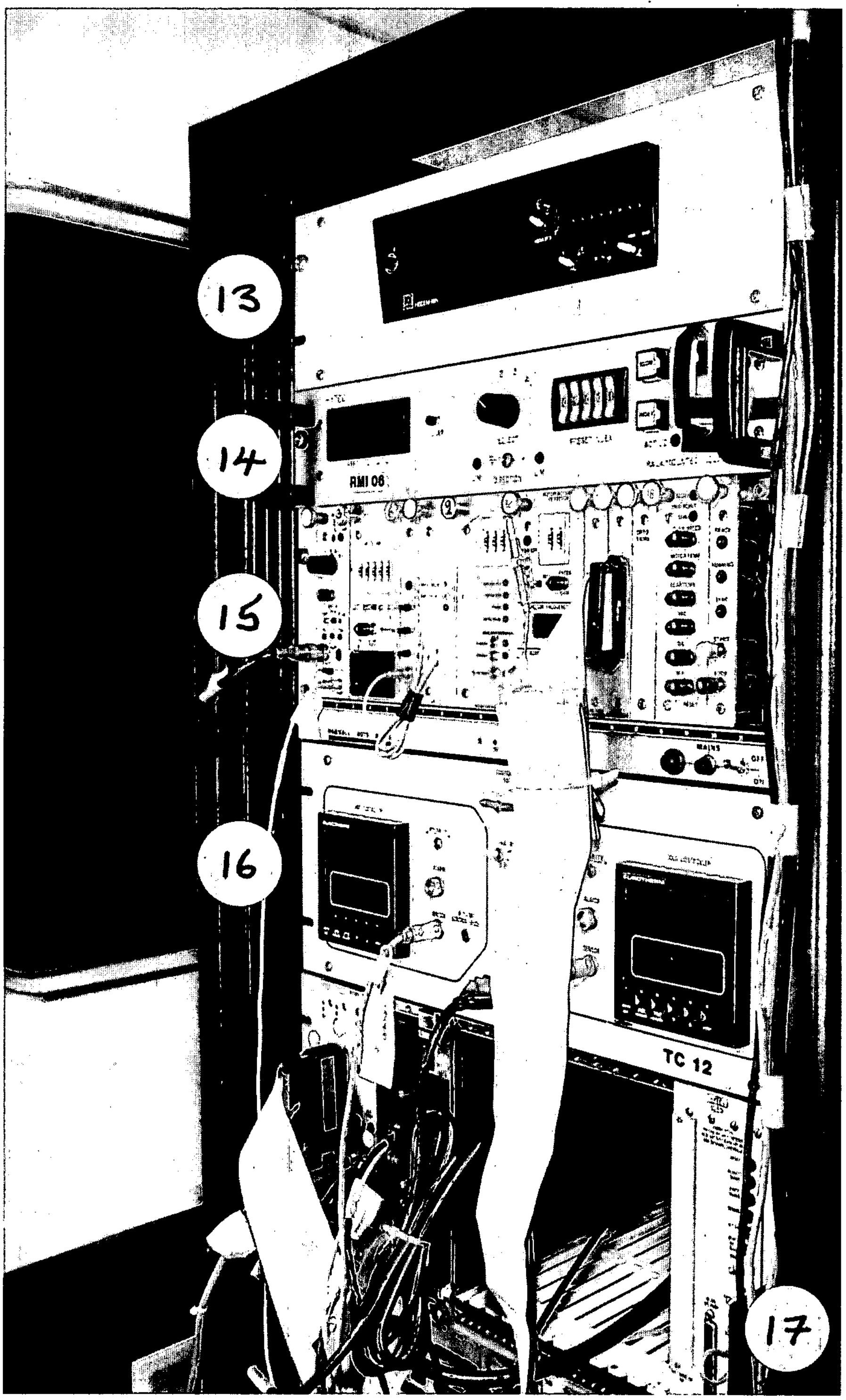
To release the vacuum in the primary flight path
Close *Valve V2* and open *Valve V1.*

Electronics Card D is the discriminator unit for Incident Beam Monitor #2 (see Section 2.3) and the Transmission Monitor (see Section 2.9). Also see Section 2.27(b). The signal cables entering this unit are quite fragile and should be treated with respect!

Not shown:

LOQ *Interlock Boxes* and *Main Shutter Control Panel* (the latter is duplicated inside the LOQ Cabin) on the outside of the enclosure fence immediately to the right of the gate, and

LOQ *Cortina Unit* (the chopper power supply) immediately adjacent to the gate (Do not interfere with the *Cortina Unit*, and be careful not to accidentally knock the main isolator switch at ankle level!)


13
14
15
16
17
RMI 06
TC 12
MAINS

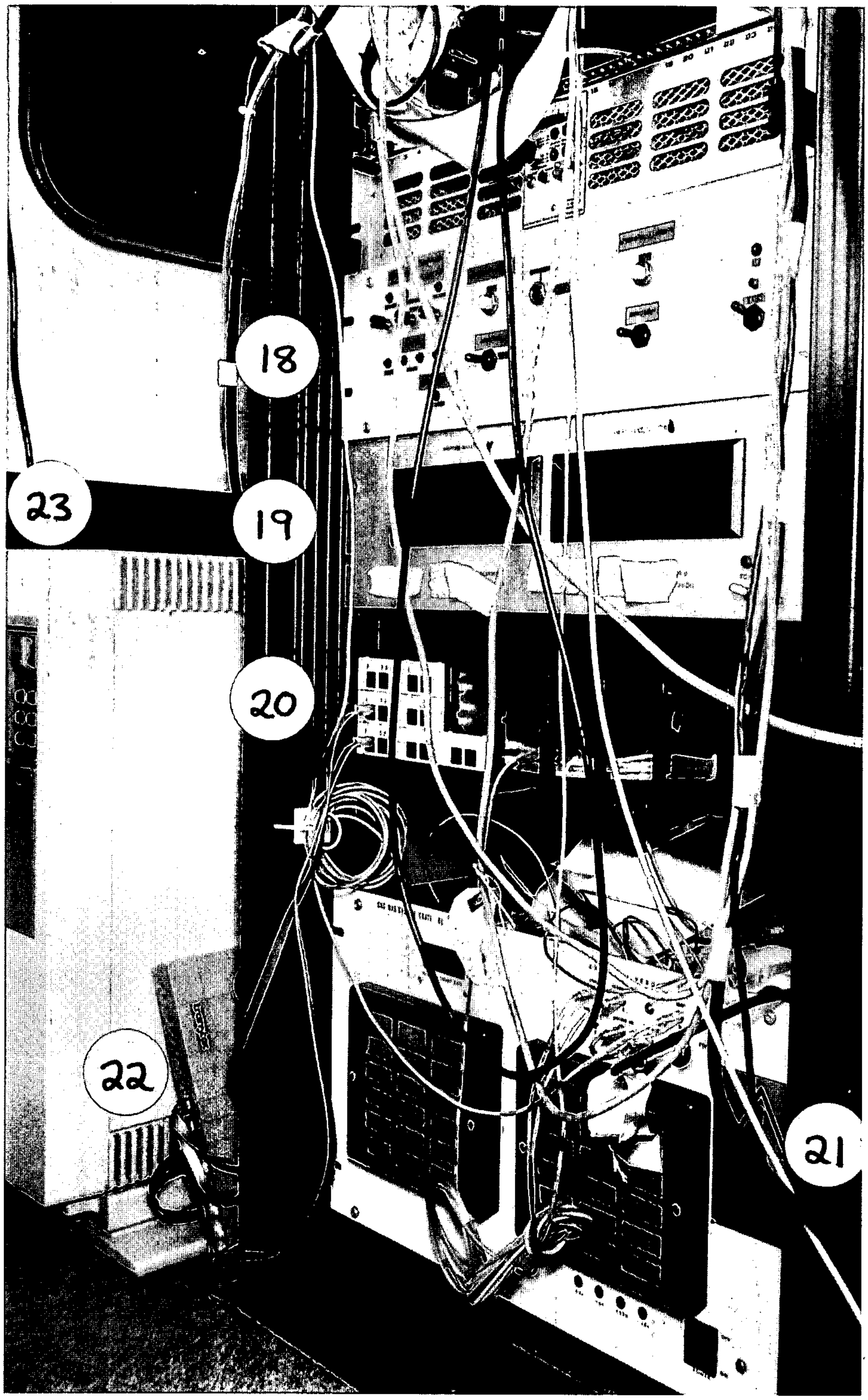
2.13 Digital Readout for Heidenhain Translation Stage
- "old" LOQ Sample Changer. Also see Section 3.5.
- toggle switches should be set to; *green dot, readout 1* and *mm.*
- to set the current position of the translation stage at a particular value, dial in the position (in mm) with the thumbwheel switches (the two rightmost switches are after the decimal point!) and push the black button underneath. Flick the central toggle switch to *readout 2* and push the black button again. Return the toggle switch to the *readout 1* position.

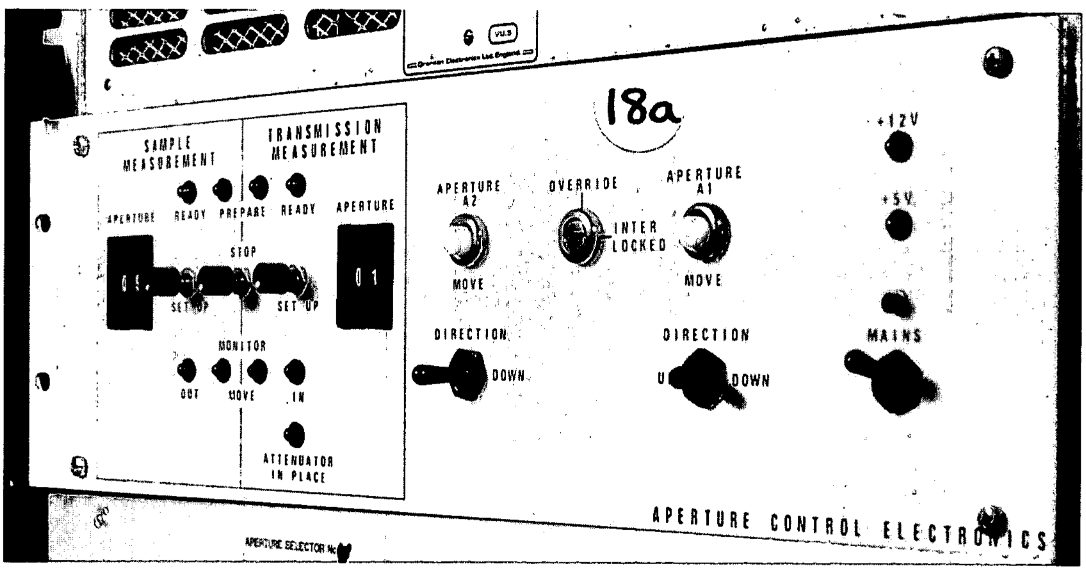
2.14 Beamstop Translation Stage Control Crate
- operates on Channel 2 only.
- readout is in steps. 400 steps = 1 mm.
- the *direction* switch should be set to "—" for in (ie, move towards IRIS) and "+" for out (ie, move towards CRISP).
- best driven using the slow button as there is no encoder on the motor!

2.15 Chopper Control Crate
- in normal operation this will display (left to right):
  a red light above the speed (frequency) selected
  a green SMP / EEP light
  the chopper speed in Hz
  a green "ready" light
  a red "running"light
  a yellow "synch" light

2.16 Eurotherm Temperature Controller
- two channels; *hot* and *cold.*
- displays are in mV, not degrees!
- the *cold* controller can be used to drive heater elements.
- if display indicates an alarm state, push *button 1.*
- ensure that the correct thermocouples are selected, but only operate the switches when the controller is switched off! The thermocouples will be Pt unless you are using a cryostat, CCR or furnace when they will be Rh/Fe.
- also see Pages 3-5 and 3-6.

2.17 CAMAC Crate
- CAMAC stands for Computer-Aided Measurement And Control!
- the device drivers and interface cards are on the left, the Hytec 1050 ("Falcon") minicomputer is on the right.


18
19
20
21
22
23


18a
SAMPLE MEASUREMENT
TRANSMISSION MEASUREMENT
APERTURE
READY
PREPARE
READY
APERTURE
STOP
SET UP
SET UP
01
MONITOR
OUT
MOVE
IN
ATTENUATOR IN PLACE
APERTURE A2
MOVE
OVERRIDE
INTER LOCKED
APERTURE A1
MOVE
+12V
+5V
DIRECTION
DOWN
DIRECTION
U
DOWN
MAINS
APERTURE CONTROL ELECTRONICS
APERTURE SELECTOR

LOQ Cabin Electronics Room - II. *[Negative 94RC5752]*

2.18 Aperture Control Crate

- LOQ has two collimation aperture wheels. Aperture #1 is immediately before the chopper. Aperture #2 is at the sample position (see Section 2.5).
- Aperture #2 is used to attenuate the neutron beam for transmission measurements. This changeover is under computer control but may be initiated manually with the black pushbuttons on the left.
- The white pushbuttons allow independent manual rotation of each aperture wheel from one position to the next. The black toggle switches control the direction of rotation; *up* and *down* relate to increasing or decreasing aperture codes as displayed on Unit 2.19. Also see Section 1.

2.18(a) Aperture Control Crate

- this is the more recent (1995) version of the crate shown in the main picture.
- the two pairs of thumbwheel switches are for presetting the aperture codes that will be used for scattering and transmission measurements. Also see Section 1.

2.19 Aperture Position Readout

- left display for Aperture #2, right display for Aperture #1.
- the white toggle switches (hidden behind masking tape in the main picture) change between the two sets of optos on each aperture. Do not alter the settings of these switches unless you are certain that both sets of optos are functioning correctly!

2.20 RS423 Patch Panel

- allows connections from the LAN terminal server(s) (Section 2.22) to outlets in the main part of the LOQ cabin. These outlets may be identified by the code numbers on this panel.
- since the photograph was taken this unit has been incorporated into the cabin trunking panel to the left of the LOQ FEM (Section 2.23).

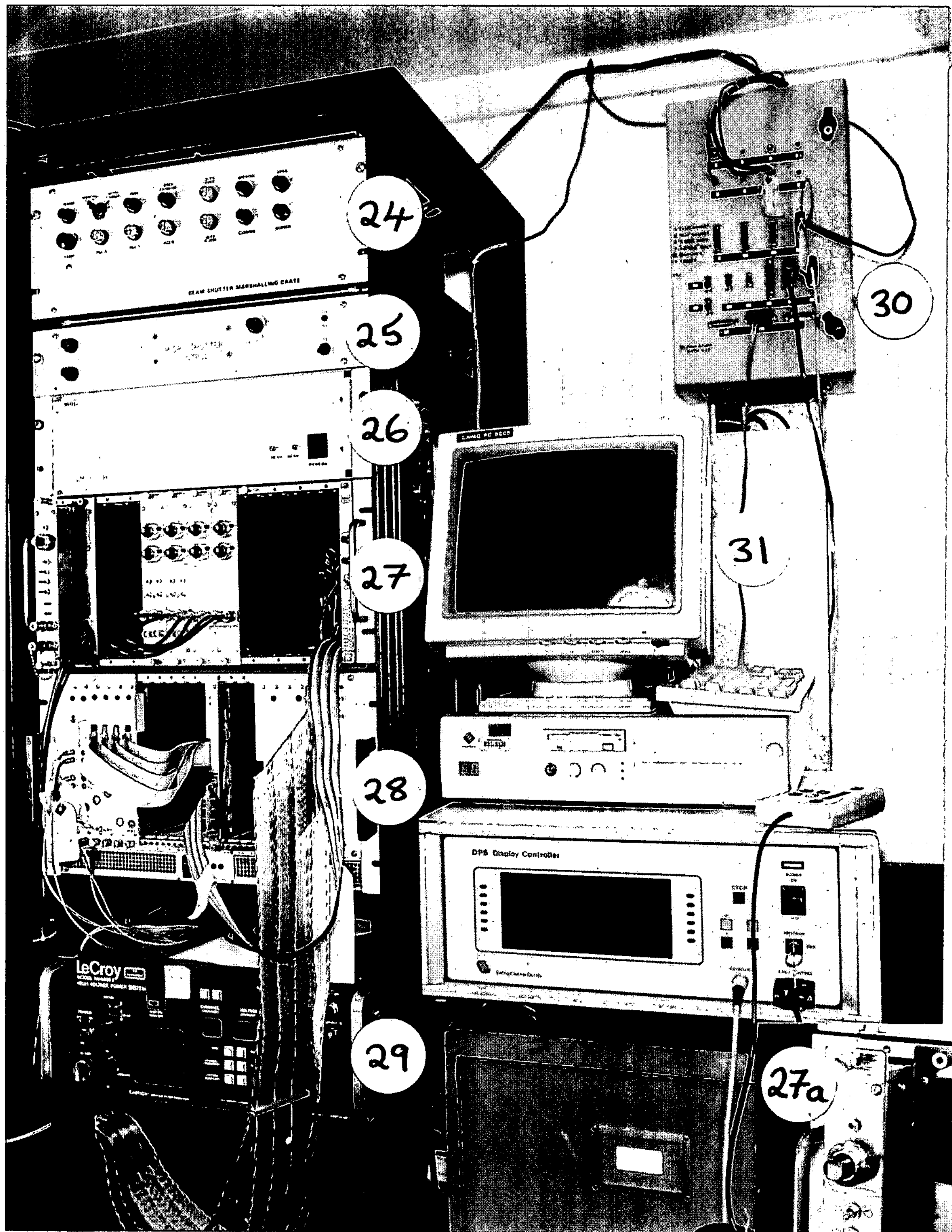
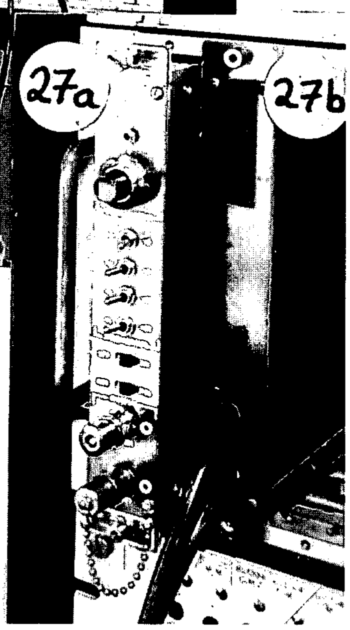
2.21 Data Acquisition Electronics (DAE) System Crate

- this crate histograms the data from the monitors and main detector(s) in each time channel and so is one of the most important pieces of electronics in the LOQ cabin!
- The LCD *count rate* display is the data rate on the computers Q-bus and not the actual count rate at the detector.
- at the top right of the front panel are the inputs from the various timing signals and vetoes.
- turning this crate off will crash the LOQ computer!

2.22 Terminal Server

2.23 DEC MicroVax 3200

- the LOQ Front End Minicomputer (FEM)


24
25
26
27
28
29
30
31
BEAM SHUTTER MARSHALLING CRATE
LeCroy
HIGH VOLTAGE POWER SYSTEM
DPS Display Controller
STOP

27a
27b

LOQ Cabin Electronics Room - III. *[Negative 94RC5150]*

2.24 Beam Shutter Marshalling Crate
- do not interfere with this crate!

2.25 Microshutter Status Display
- also see Section 2.8.

2.26 Power Supply for Beamstop Translation Stage Control Crate
- also see Section 2.14.

2.27 "NIM Bin" with ORDELA Detector Electronics
- please do not touch any of these vernier dials or switches!

2.27(a) ORDELA Pulse Generator
- this is diagnostic equipment and <u>must not</u> be pushed home in the rack until required or the real neutron data may be corrupted!

2.27(b) Low Voltage Power Supply
- supplies the discriminators for monitor #1 and monitor #2.

2.28 Instrument Crate
- contains Monitor Input Modules (MIM's), Detector Input Modules (DIM's), demultiplexers, and the Ping-Pong Frame Memories (PPFM's).
- converts the raw signals from the monitors and main detector(s) into time-coded and position-coded counts the DAE (see Section 2.21) can histogram.

2.29 Lecroy High Voltage Power Supply Unit
- see the separate instructions below.

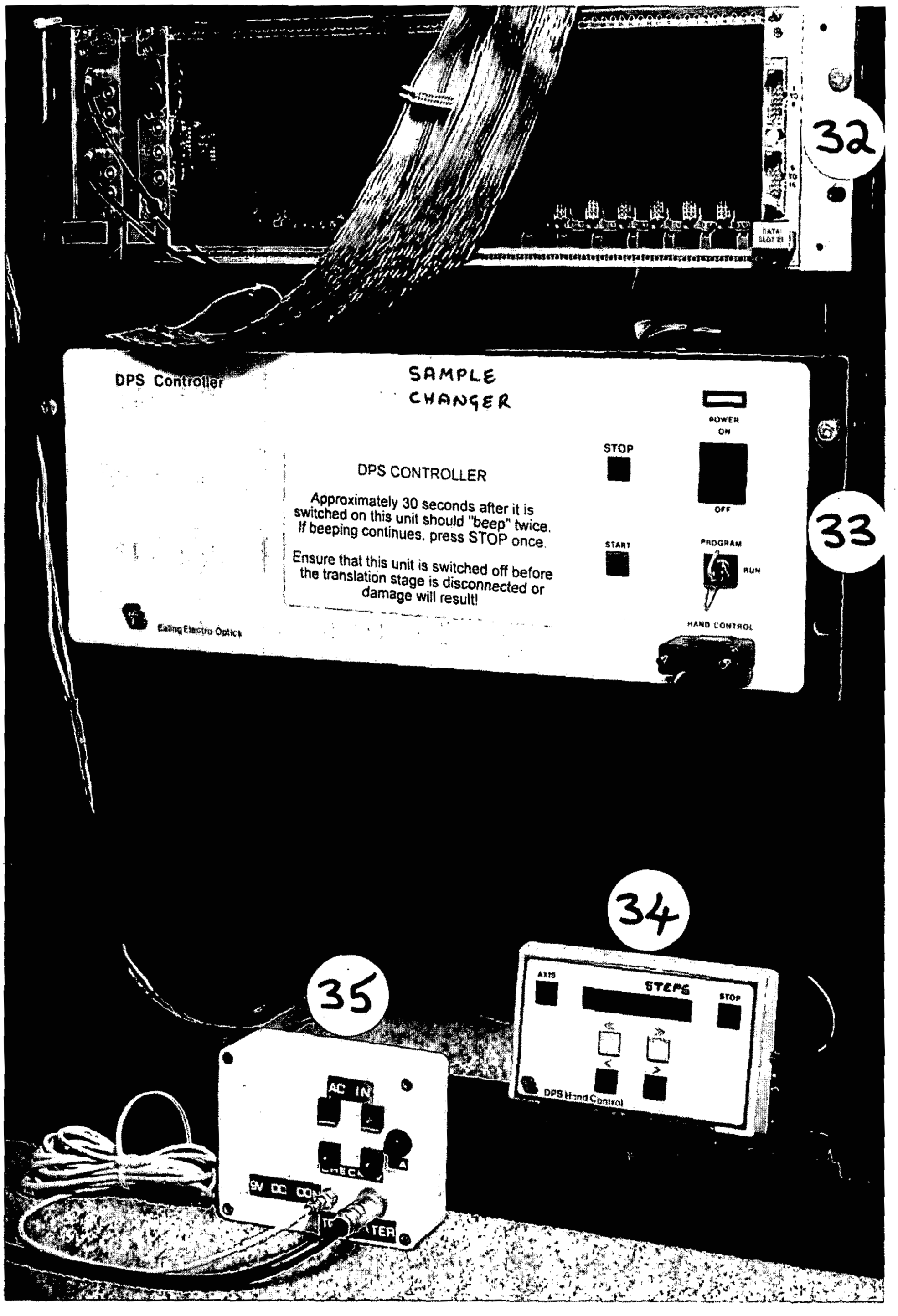
2.30 LOQ Patch Panel
- allows easy connection of equipment between the cabin and sample position (where there is a duplicate panel).
- also see Section 2.46.

2.31 PC
- used to control various pieces of equipment (eg; the *Lauda Circulating Fluid Bath,* see Section 3.41).

If the Lecroy Unit Dies (loss of HV to Detectors)..

- Turn the Lecroy Unit off at the keyswitch.
- Wait 1 minute.
- Turn the Lecroy Unit back on again at the keyswitch.
- Go to Channel 99 (push in the *C* button and use the arrow buttons) to set the Ramp Rate.
- Set Channel 99 to "0004" (push in the *V* button and use the arrow buttons).
- Push the *HV On* button.
- Go to Channel 14c to set the Current Limit on the main detector channel.
- Set Channel 14c to "0050" using the *V* button.
- Go to Channel 14 to set the HV for the main detector.
- Set Channel 14 to "4000" in steps of "1000" (release the *V* button after each thousand).

- Check, and set if necessary, Channel 1 to "975" (HV for monitor #1)
- Check, and set if necessary, Channel 2 to "1100" (HV for monitor #2)
- Check, and set if necessary, Channel 3 to "1000" (HV for the Tx monitor)
- Check, and set if necessary, Channel 7 to "1300" (HV for the High-angle bank)


32
DPS Controller
SAMPLE CHANGER
POWER
ON
OFF
STOP
DPS CONTROLLER
Approximately 30 seconds after it is switched on this unit should "beep" twice. If beeping continues, press STOP once.
Ensure that this unit is switched off before the translation stage is disconnected or damage will result!
START
PROGRAM
RUN
HAND CONTROL
Ealing Electro-Optics
33
34
AXIS
STEPS
STOP
DPS Hand Control
35
AC IN
9V DC

LOQ Cabin Electronics Room - IV. *[Negative 95RC2659]*

2.32 Eurocrate Electronics Bin
- newer-style *NIM Bin* (see Section 2.27)

2.33 Ealing Digital Positioning System (DPS) Control Unit
- "new" LOQ translation stage. Also see Section 3.14.

Note:

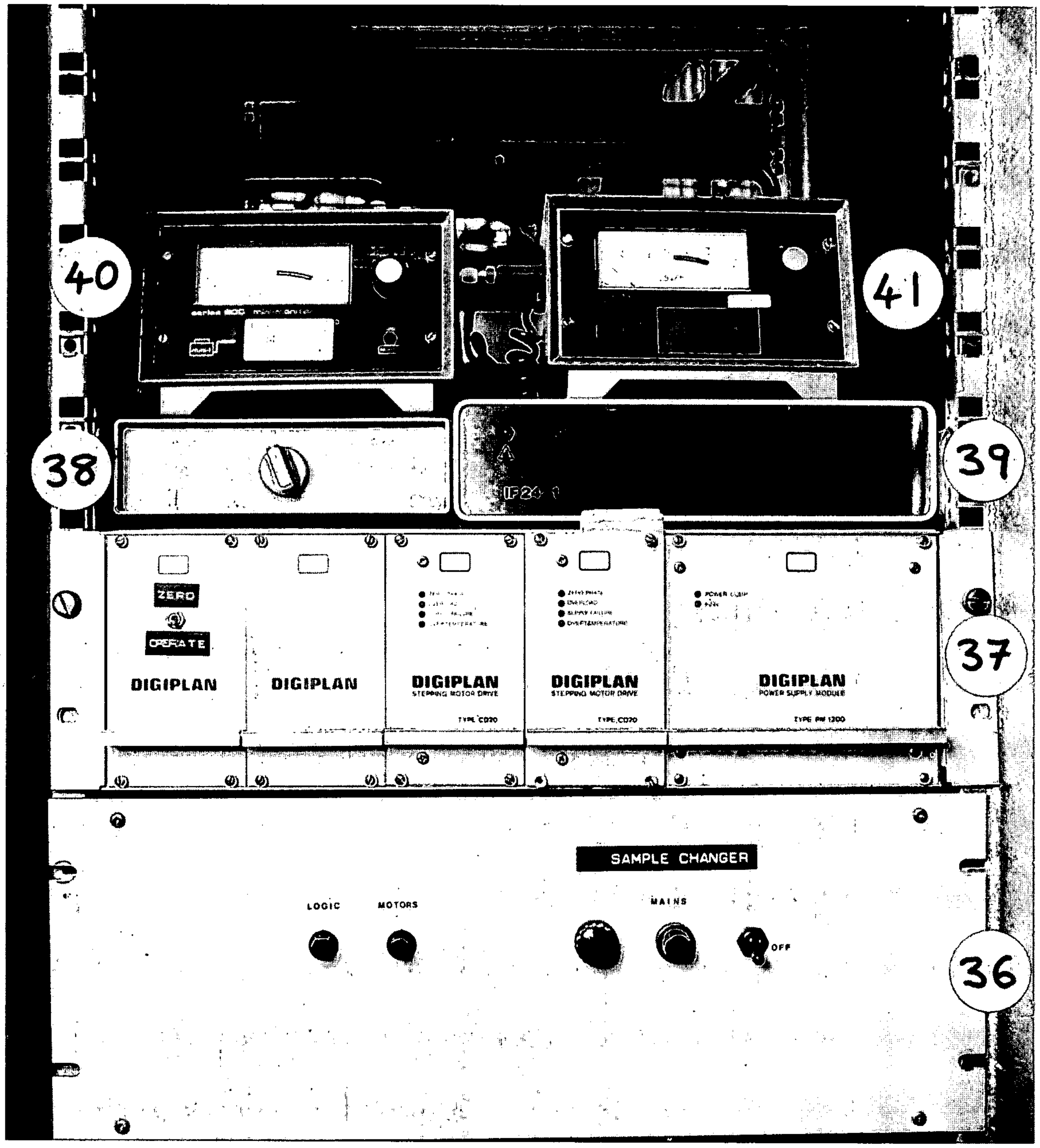
- this unit must be switched off before (a) the motor drive cable to the *Ealing Translation Stage* (see Sections 3.14 and 3.15) is unplugged or (b) the power to the LOQ FEM is cycled (see Section 2.23) otherwise damage will result!

2.34 Ealing DPS Remote Control Handset
- "new" LOQ translation stage.
- allows manual control of the position of the *Ealing Translation Stage.* Also see Section 3.14.

2.35 Cartridge Heater Power Booster Box
- used when the "cold controller" of the *Eurotherm Temperature Controller* (see Section 2.16) is unable to supply sufficient electrical power to the cartridge heater(s) in use.

The box contains a solid-state relay which switches power from an external power source, such as a variac, in synchronisation with a Eurotherm control signal. Note that the polarity of the control signal reverses between the Eurotherm "hot" and "cold" controllers (the level also changes). Care must therefore be taken to ensure that the appropriate booster box is being used.

To use the booster box with the "cold" controller:
- connect the "9 VDC" cable from the *Eurotherm Temperature Controller* to the box.
- unplug the "heater" cable from the *Eurotherm Temperature Controller* and plug it into the box.
- plug the output from an AC variac into the sockets on the box marked " AC In".
- limit the variac output to the rating of the cartridge heaters.


40
41
38
39
37
36
ZERO
OPERATE
DIGIPLAN
DIGIPLAN
DIGIPLAN
STEPPING MOTOR DRIVE
DIGIPLAN
STEPPING MOTOR DRIVE
DIGIPLAN
POWER SUPPLY MODULE
SAMPLE CHANGER
LOGIC
MOTORS
MAINS
OFF

Electronics Rack by LOQ Sample Position (IRIS Side). *[Negative 94RC5157]*

This rack was removed during a reorganisation in May 1996 following the LOQ and IRIS / OSIRIS upgrades. Items 2.36 and 2.37 have been transferred to ALF on extended loan, but because they may still be used on LOQ in the event of problems with the new translation stage their descriptions have been retained. Item 2.38 has been abolished as it is unnecessary for short-term use of the old translation stage. Item 2.39 has been relocated on top of the patch panel (see Section 2.46) on the CRISP side of the sample position.

2.36 Power Supply for Heidenhain Translation Stage
- "old" LOQ translation stage. Also see Section 3.5.

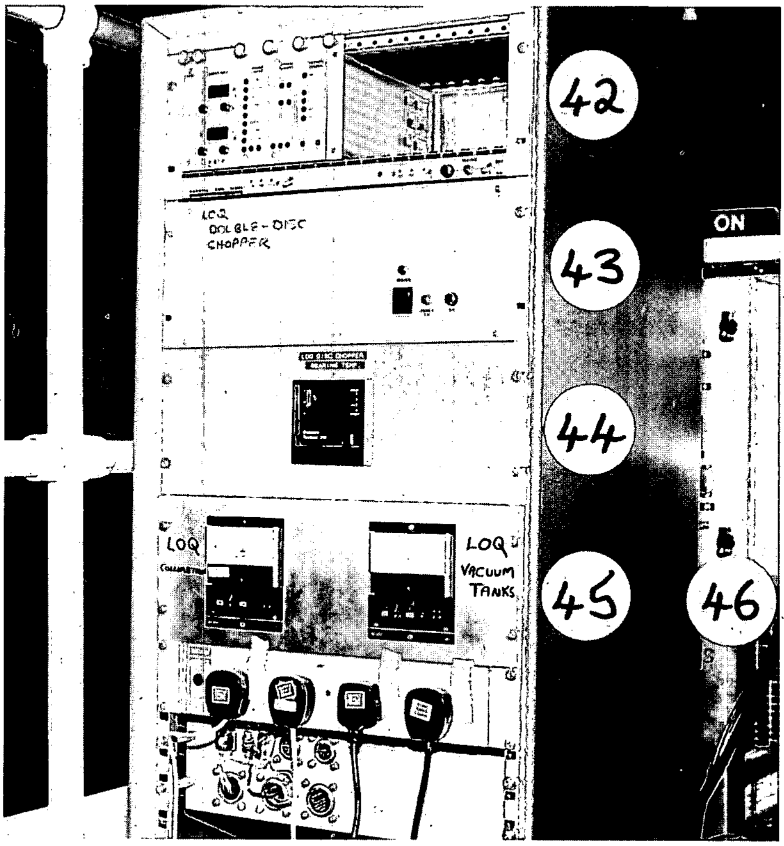
2.37 Digiplan Stepper Motor Drive Crate
- "old" LOQ translation stage. Also see Section 3.5.
- cards are (left to right):
  mode switch
  blank plate
  stepper motor drive card
  spare stepper motor drive card
  power supply
- mode switch operation:
  Set to *zero* the translation stage can be driven to locate itself between the two central (reference) microswitches on its incident beam side. Set to *operate* for normal use.
- also see Section 2.38.

2.38 Computer / Remote Handset Selector Switch
- "old" LOQ translation stage. Also see Section 3.5.
- switches between computer control and manual control (from the remote handset) of the translation stage.
- set to *computer* for normal use.
- also see Section 3.6.

2.39 Haake IF 24-1 RS232 Interface
- interfaces between the LOQ FEM (see Section 2.23) and the *Haake Circulating Fluid Bath* (see Section 3.47).

2.40 Portable β-Radiation Monitor
- used to monitor samples for induced beta radioactivity after irradiation in the neutron beam.

2.41 Portable γ-Radiation Monitor
- used to monitor samples for induced gamma radioactivity after irradiation in the neutron beam.



42
LOQ
DOUBLE-DISC
CHOPPER
43
44
ON
LOQ
LOQ
VACUUM
TANKS
45
46

Electronics Rack by LOQ Sample Position (CRISP Side). *[Negative 94RB5936]*

2.42 Double-Disc Chopper Indexer Crate

- do not attempt to operate any of the controls on this crate whilst the chopper is running except the computer / manual *(COMP / MAN)* toggle switch!
- operates a clutch mechanism which determines the sector opening of a double-disc chopper (like that on LOQ).
- in normal operation this crate will display several coloured LED's. It also has two, three-digit, LED readouts. The top readout displays the requested sector opening in degrees. In normal operation this will be "126" or "000" (which is also equivalent to 126º). The bottom readout, which displays the position (or index) of the disc, is meaningless (and unreadable!) except when the chopper is stopped or at very low speed.
- to manually set the sector opening of the double-disc:
    - Stop the chopper!
    - Slide the toggle switch to *Man.*
    - Press the *Reset* button.
    - Use the *Inc* (increment) and *Dec* (decrement) buttons to select the required opening (2 to 126 degrees in 2 degree steps).
    - Press the *Init* button. Various coloured LED's will illuminate and then go off as the indexer locates its reference position, engages the clutch, sets the requested opening and then disengages the clutch. When the operation is complete the two LED readouts will both display the requested opening and the green *Chop Drive En* and *Chop Stopped* LED's will be illuminated.

2.43 Indexer Power Supply

- see Section 2.42.

2.44 LOQ Disc Chopper Bearing Temperature Display

- displays the temperature of the chopper bearings.
- the chopper can trip off if the bearing temperature is too high.
- the trip level is set by the ISIS Chopper Group and may vary with the type of bearings and/or lubrication system employed. The trip level must not be altered except on the advice of either a chopper technician or a LOQ instrument scientist as irreparable damage could result.
- to view the current trip level push in the button just below the temperature readout.
- if you believe that the chopper is in danger of tripping because the trip level has been set too low, contact a LOQ instrument scientist.
- under normal operation, if the chopper is running at 25 Hz the bearing temperature will probably be around 45°C. At 50 Hz it may rise to around 75°C.

2.45 Vacuum Gauges

- these gauges display the vacuum level in the primary and secondary flight paths.
- the lefthand gauge registers the vacuum in the primary flight path (or what is generally referred to as just "the collimation"). However, due to the frequency with which the vacuum in the primary flight path is cycled (for sample and collimation changes, etc) this electrical gauge is normally left switched off in favour of the mechanical gauges on the collimation vacuum pump (see Page 2-5).
- the righthand gauge registers the vacuum in the secondary flight path (or what is generally referred to as just "the vacuum tank"), the region between the sample and the main detectors.

2.46 LOQ Patch Panel (partially obscured)

- allows easy connection of equipment between the sample position and cabin (where there is a duplicate panel).
- also see Section 2.30.

Notes

Section 3

Sample Environment

Contents

Safety Note: Cadmium

Cadmium sheeting (1 - 2 mm thick) is widely used on LOQ for shielding our own and user-provided sample holders, etc. However please note that this places restrictions on the use of such sample environment.

- **If possible, always wear gloves when handling cadmium. If skin contact is necessary then ensure that you wash well afterwards.**

- **NEVER let cadmium come into direct contact with heater elements.**

- **Cadmium must NEVER be used in furnaces.**

- **Cadmium-faced or cadmium-plated equipment must NEVER be heated above 150°C in air, or above 90°C in a CCR (or other such vacuum vessel). This is because cadmium oxide fumes are very toxic!**

- **If, by accident, the above limits are exceeded DO NOT approach the apparatus until it has thoroughly cooled down. Notify your local contact immediately so that appropriate decontamination procedures can be implemented.**

- **Though rare on LOQ, cadmium can become active after irradiation by the neutron beam so always monitor the equipment before handling it. If the radiation level is greater than 75 μSv Hr^{-1} then contact the Duty Officer (extension 6789).**

Where shielding of high temperature components is necessary, gadolinium foil may be more suitable. Please discuss your requirements with the instrument scientists.

Guidance Note EH 1 from the Health and Safety Executive

Cadmium: health and safety precautions

Environmental Hygiene Series EH 1 (revised June 1986)

These Guidance Notes are published under five subject headings: Environmental Hygiene, Chemical Safety, Plant and Machinery and General and Medical.

DESCRIPTION

1 Cadmium (Cd) is a silver white ductile metal which melts at 321°C and when heated to a sufficiently high temperature emits highly toxic fumes of cadmium and cadmium oxide. It can react vigorously with oxidising materials.

EXPOSURE LIMITS

2 Occupational exposure to cadmium and its compounds should be kept as low as is reasonably practicable and should not exceed the exposure limits published in the Health and Safety Executive's (HSE), Guidance Note EH40, Occupational Exposure Limits. At present the control limit for cadmium dust and salts (other than cadmium sulphide pigments) is 0.05 mg m^{-3}, 8-hour time-weighted average. Cadmium oxide fume is subject to an additional short term control limit of 0.05 mg m^{-3}, 10-minute time-weighted average.

3 Cadmium sulphide pigments are assigned a recommended limit of 0.05 mg m^{-3}, 8-hour time-weighted average, and 0.2 mg m^{-3}, 10-minute time-weighted average. These recommended limits are currently under review.

INDUSTRIAL USES

4 Cadmium and its compounds are present in a number of industrial raw materials and they are used for a variety of industrial applications. Potential exposure to cadmium may occur in the use of cadmium compounds in the manufacture of various products and also in the recovery, recycling or disposal of materials containing cadmium, eg breaking nickel-cadmium batteries and refining copper scrap containing cadmium.

5 Metallic cadmium is used

(a) as a protective coating for metals, usually applied by electroplating, though sometimes mechanical plating or vacuum processes are used;

(b) as an alloy with copper to which it imparts strength without impairing electrical conductivity (this alloy is used in the production of high conductivity cable for the electrical industry);

(c) as a constituent of low melting point alloys used in the tips of fire sprinklers, safety plugs, steam boilers and electrical fuses;

(d) as a constituent of alloys used for bearings in combustion engines, and for soft solders;

(e) as a neutron absorber in nuclear reactors and in radiation detecting/measuring instruments, and

(f) as a constituent (about 25%) in some brazing alloys (also known as hard solders or silver solders).

6 Cadmium compounds are used

(a) as pigments in the production of paints, plastics, rubber, printing inks, glass and ceramic glazings (notably cadmium sulphide and cadmium sulphide-selenide);

(b) in the negative plates of alkaline storage batteries (cadmium hydroxide);

(c) as the source of the metal ion in electrolytic plating baths (cadmium oxide and cadmium cyanide);

(d) as a starting point in the preparation of other cadmium compounds (cadmium oxide);

(e) as a consitituent of photo-electric cells and rectifiers (cadmium selenide, cadmium sulphide and cadmium telluride);

(f) to form contact breaker points for electrical systems (cadmium oxide in silver), and

(g) as stabilisers for plastics (cadmium salts of organic acids).

HEALTH EFFECTS

7 Absorption occurs mainly through inhalation of cadmium dust and fume, although ingestion is also possible. Non-occupational intake mainly results from cigarette smoking although some may be attributable to diet.

Acute effects

8 The main acute effects are respiratory, after inhalation of cadmium oxide fume, and gastro-intestinal, after ingestion. The effects of freshly generated fume generally include irritation of the eyes, nose and throat, followed by cough, headache, dizziness, weakness, chills, fever, chest pains and breathlessness, although fatal chemical pneumonitis may occur in severe cases. Pulmonary damage may occur in the absence of such symptoms and may be delayed for several hours or days. Following ingestion the first symptoms to occur are severe nausea, vomiting, diarrhoea, muscular cramps and salivation. In more severe cases shock, acute renal failure, cardiopulmonary depression and death may occur within 7 to 14 days. Liver damage may also occur.

9 Acute posioning usually results from failure to recognise that metal being heated is cadmium or cadmium-plated. Cases have resulted from welding cadmium-plated steel, from brazing or cutting cadmium-plated nuts and from using brazing alloys containing cadmium without proper ventilation. Exposure to dust or fume during melting or pouring of cadmium metal or alloys has produced cases of acute poisoning, as has the accidental ignition of cadmium powder.

Chronic effects

10 Repeated exposure to cadmium is associated with a slow accumulation in the renal cortex (the outer layer of the kidneys). This can happen over several years of occupational exposure. Renal abnormality occurs when the concentration of cadmium in the cortex exceeds a critical value which is thought to be between 200 and 400 parts per million. One of the abnormalities, a change in calcium handling, is associated with the formation of kidney stones. Progression from minor biochemical changes to more severe changes is slow but may not be reversible. However, there is no clear epidemiological evidence that these changes in kidney function lead to an increase in mortality from renal conditions.

11 A number of chronic effects following prolonged exposure to cadmium at work have been described. The kidney is the organ primarily affected, though damage to the respiratory system can also occur.

12 Obstructive airways disease and pulmonary fibrosis have been described in workers exposed to cadmium dust or fume for prolonged periods. Some deaths due to emphysema have been reported in workers repeatedly exposed to very high levels of cadmium fume.

13 Heavy exposure in the past has produced chronic poisoning in workers employed in the casting of copper-cadmium alloys and in the manufacture of cadmium pigments and alkaline storage batteries.

Sensors And Heaters

Sensors and heaters are used extensively in *orange cryostats (OC's), closed cycle refrigerators (CCR's)* and on *centre sticks (CS's).* These devices are normally used for measuring and controlling cryogenic temperatures as low as 4 K, and on occasions down to 1.5 K, and up to room temperature. The sensors and heaters are capable of working up to about 400 K. For temperatures around ambient *circulating fluid baths (FB's)* are used which work over the temperature range of about -50 to +100°C. Furnaces are used from room temperatures to around 1000°C. Plans are in hand to extend the top temperature limit for furnaces to 1500 °C.

1. Sensors

For low temperature work **RHODIUM / IRON sensors** are used in a four wire configuration, that is, a constant current of 1 mA is passed down two wires to the sensor and the resulting voltage in mV is measured on the other two wires and displayed on the Temperature Controller (see Section 2.16). Because the RHODIUM / IRON sensors are calibrated over a minimum of 30 points, by spline fitting and extrapolation, temperatures can be determined very accurately over the range 1.5 K to 600 K.

For work involving temperatures from ambient up to 200 or 300°C, standard PT-100 **PLATINUM resistance thermometers** are used. These devices are again used in a four wire configuration with a constant current of 0.25 mA. PLATINUM resistance thermometers are also used in the circulating fluid baths.

For furnace work, sheathed **Type K thermocouples** are used with an external cold junction compensation box. Millivolts generated by the thermocouple are read by the temperature controller and converted by the computer to a display of the temperature being measured.

2. Heaters

Except in furnaces, cylindrical **cartridge heaters** are used extensively. These heaters are 3.1 mm in diameter (the same diameter as the RHODIUM / IRON sensors) and 50.8 mm in length. The heater rating varies depending on the experiment, but 100 W heaters are used wherever possible but derated to a maximum of 70 W to give a longer working life.

In furnaces **vanadium foil** is normally used as the heater element. This requires low voltages but high currents and is provided by special power supply units capable of delivering upto about 3 kW of power. For very high temperature work **niobium foil** needs to be used.

3. Wiring Arrangements

All wiring is standardised for quick changeover of units, ease of operation and maintenance, though many experiments require modifications to suit the requirements of that particular experiment.

For sensor wiring cotton-covered self-fluxing constantan wire is used. Four lengths of such wire are twisted in a loom of appropriate length for the unit being wired. For heaters enamel-covered copper wire 0.5 mm in diameter is used.

On OC's, CCR's, CS's and furnaces, connections from the sensors and heaters are brought to the outside world through a vacuum tight, hermetically-sealed, circular 12-way Jaeger connector. The pin connections are specified below. This is a standard arrangement which caters for two sensors, a heater and an earthing connection if required.

On OC's and CCR's a sensor, called a *fixed sensor,* is wired near the heat exchanger along with a heater. The heater and the associated wiring is protected by a thermal switch which is normally closed but opens if the temperature of the copper block to which it and the heater are attached exceeds 70°C. The thermal trip automatically resets - closes - when the temperature falls below 65°C.

Most CS's have a fixed sensor housed in the metalwork as near the sample as is possible. Not far from there, and away from the neutron beam, are placed two Lemo connectors on a circular metal bracket attached to the CS. These allow a *floating sensor* and a *floating heater* to be physically attached to a sample under investigation, if required.

The above paragraphs describe a basic system which operates successfully. However experiments sometimes require a different arrangement of sensors and heaters. These requirements can often be accomodated **providing that sufficient notice is given.**

4. Connectors

The wiring arrangement for a **12-way Jeager connector** at ISIS is:

Pin No.	Function	
1	Current –)	
6	Voltage –)	*fixed*
3	Voltage +)	*sensor*
4	Current +)	
8	Current –)	
9	Voltage –)	*floating*
10	Voltage +)	*sensor*
11	Current +)	
7	Heater	
12	Heater	
2 or 5	Chassis earth	

Note:

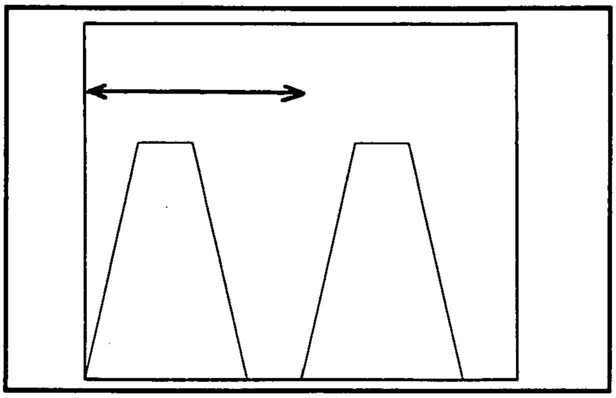
By using a polarity switch on the Temperature Controller Crate (see Section 2.16) the current and voltage can be swapped. This facility is a great aid in pinpointing possible wiring breaks and faults.

A mating 12-way Jaeger connector is used to bring out the connections which are then converted into 4-way Lemo connectors for the sensors and a 3-way Lemo connector for the heater. On an OC these connections are brought out on a right angled plate attached to the top of the OC in such a way that Lemo connectors are perpendicular to the body of the OC. On a CCR the mating 12-way Jeager connector is terminated in appropriate Lemo connectors which are housed in a black metal box secured to the top flange of the CCR head. The same type of black box is used for connections from CS's. This arrangement allows sensors and heaters to be connected via the patch panels (see Sections 2.30 and 2.46) to the Temperature Controller Crate (see Section 2.16) in the instrument cabin.

Steve King, 30 May 1995

Tips On Configuring A Eurotherm Temperature Controller

For a much more detailed guide to the Eurotherm Temperature Controller, how to operate it and how to configure it, please consult Hari Shah's green manual!

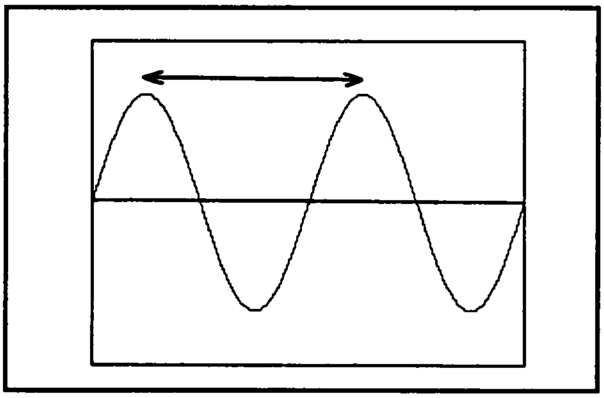


The Eurotherm Controller supplies electrical power to cartridge heaters in pulses. The actual power supplied is determined by three factors:

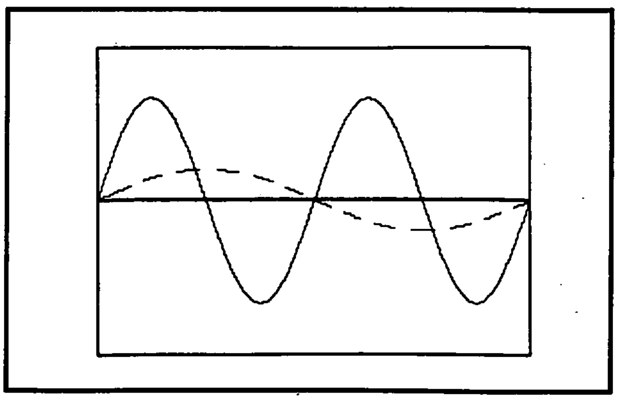
1) the voltage supply (eg; the variac setting).

2) the *MAX POWER* setting of the Controller.

3) the *CYCLE TIME* setting of the Controller (see graph).



The actual temperature of the sample oscillates about the requested temperature setpoint with a characteristic frequency determined by the configuration of the Controller and the heat capacity of the sample, sample holder, etc. The Eurotherm "tunes" itself so as to try and minimise this oscillation. This tuning takes place over an *INTEGRATION TIME* (see graph). It is also necessary to set a *DERIVATIVE TIME.* This is simply the (*INTEGRATION TIME* / 5).



This graph shows the effect that two different settings of the *PROP BAND* have on the temperature oscillation. The continuous line corresponds to a large *PROP BAND,* whilst the dashed line corresponds to a small *PROP BAND.*

As you increase the power rating of the heater:
reduce the PROP BAND
reduce the CYCLE TIME
increase the INTEGRATION TIME

The Eurotherm Temperature Controller Crate in the LOQ cabin (see Section 2.16) contains two Eurotherm 820 Controllers - one designated "HOT", and one designated "COLD".

The COLD Controller has a logic output which operates two solid state relays; one of which can be used to provide the appropriate amount of electrical power to a heater and the other to provide 0 or 9 V d.c. as a synchronised control signal. The HOT Controller has an analogue output which may be used to provide 0 to 5 V d.c. for driving ancillary equipment (eg, a thyristor unit). Both controllers are highly accurate and stable.

The controllers are normally configured in the Sample Environment Electronics Laboratory by skilled technicians. Users are therefore requested not to interfere with controller configuration settings. Users may, however, adjust a controllers PID parameters (such as those described on the previous page) to suit the apparatus being used. This procedure is described later.

When a sensor input is connected and the crate is turned on both controllers initialise. Within a minute or so the upper, green, LED digital display of each will show the temperature (or, more often, a sensor voltage in mV) and some analogue error indicator bars (sometimes mistaken for a negative reading!). Note that a sensible temperature (or mV) reading will only be obtained if the toggle switches at the top of the crate are set for the correct type of sensor (but only change these switches with the power off!).

The lower display on each controller is a 15 x 54 dot matrix display which shows the status of the controller. The controller should normally initialise in "Automatic Remote" mode which means that the status display should show a real-time indication of the mean output power.

The nine bars situated to the left of the upper digital readout provide an indication of the difference between the measured value and the setpoint as a percentage of the full range. When only the centre bar is illuminated the controlled value is within 0.5% of the full scale setpoint.

Above the dot matrix display on each controller are 6 push-buttons. Their functions are:

- Button 1 - To acknowledge an alarm.
- Button 2 - To toggle between "Automatic" and "Manual" control of heater power.
- Button 3 - To toggle between "Local" and "Remote" signals.
- Button 4 - To decrease PID parameter values.
- Button 5 - To increase PID parameter values.
- Button 6 - To set PID parameter values OR to return to the normal display.

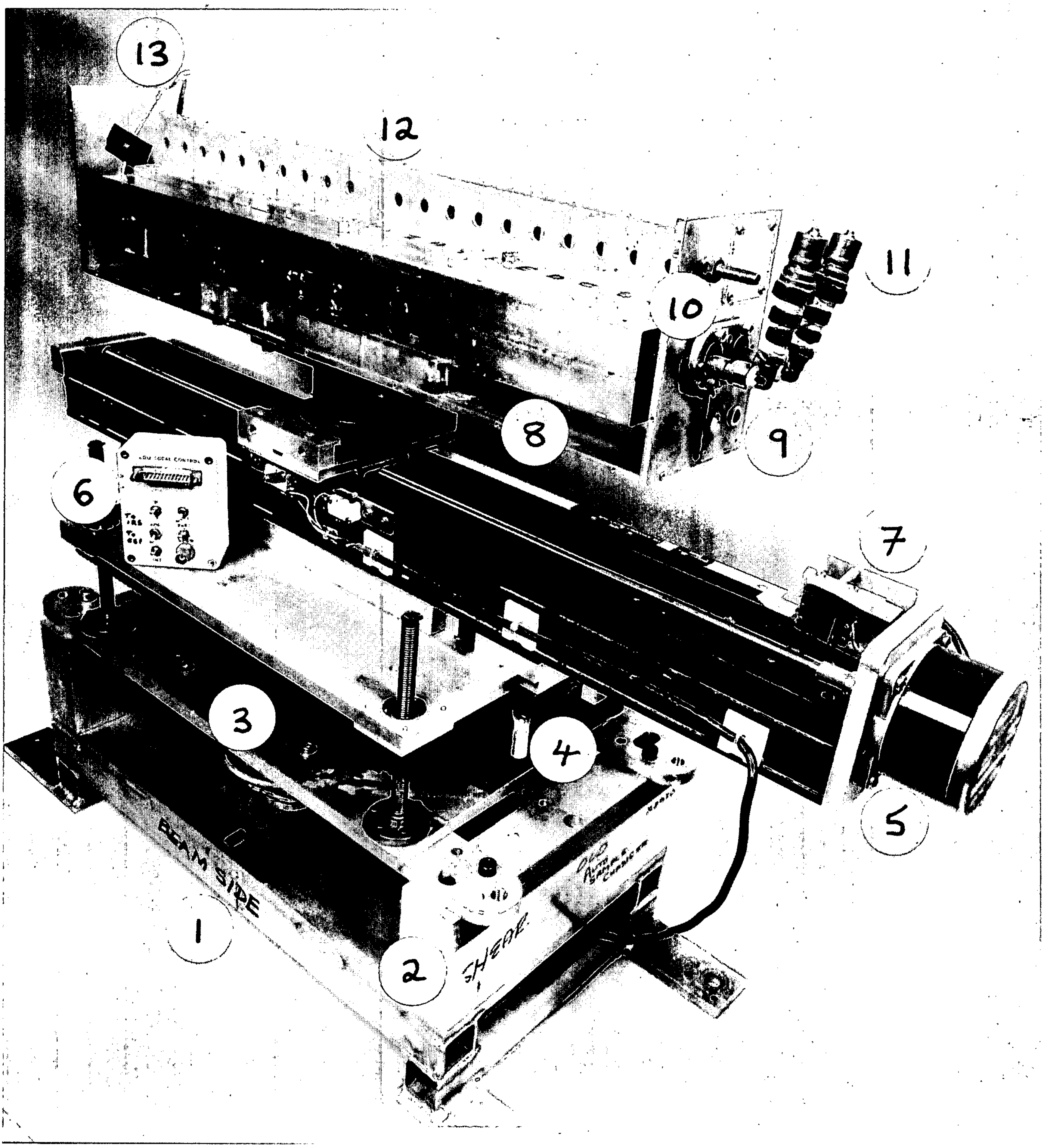
There are two sets of PID parameter values (as distinct from configuration settings) that can be changed. Most of these can be changed by issuing appropriate CAMAC commands (see the *PUNCH Manual*) but they can also be altered using the push-buttons on a controller. To access "Level 1" PID values, push buttons 1 and 6 simultaneously. To access "Level 2" PID values, push buttons 1 and 6 a second time. In each case, use button 6 to step through the various parameters and, ultimately, to return to the normal display.

Sample environment equipment (eg, cryostats and furnaces) are sometimes marked with PID values to aid controller tuning. The values are:

- P - Propband (%)
- I - Integration time (seconds)
- D - Derivative time (seconds)

ISIS Sample Environment Codes

SE Code	Description
A	Ambient experiment
CS	Cryostat centre stick
CS/ROT	Rotating cryostat centre stick
CS/RHP	Rotating cryostat centre stick for pressure cell
CS/GH	Cryostat centre stick for gas cell
CS/RF	Rotating cryostat centre stick for furnace
CC	McWhan clamped high pressure cell
CC/PE	Paris-Edinburgh clamped high pressure cell
CCR	Closed cycle refrigerator **(see Pages 3-39 & 3-41)**
CCR/ROT	Rotating closed cycle refrigerator
CCR/B	Bottom loading closed cycle refrigerator
CCR/TL	Top loading closed cycle refrigerator
DRI	Dilution refrigerator insert
DRI/Mu	Muon instruments dilution refrigerator cryostat
E/F	Electric field
FB/H	Haake circulating fluid bath **(see Page 3-27)**
FB/L	Lauda circulating fluid bath **(see Page 3-21)**
FB/P	Parma circulating fluid bath
FB/T	Techne circulating fluid bath
F3S	RAL 3 furnace **(see Page 3-43)**
F3S/Nb	RAL 3 furnace with niobium windows
F/BL	Furnace with blockheater
FL	Leicester furnace
F/PRIS	PRISMA furnace
F/RR	Resonance radiography furnace
F/SXD	SXD furnace
GC/P	Gas pressure cell
GH	Gas handling rig
G/FH	Franke-Heidrich goniometer
G/C	CRISP goniometer
G6	6-degree goniometer
HPI	High pressure intensifier
LT	Langmuir trough
MG	Magnet **(see Page 3-33)**
MG/C	Cryomagnet
OC/100	Orange cryostat (100 mm bore)
OC/50	Orange cryostat (50 mm bore)
OC/LAD	Orange cryostat (50 mm bore - special for LAD)
OC/LQ	Orange cryostat (50 mm bore - special for LOQ) **(see Page 3-43)**
OC/Mu	Orange cryostat (special for muon instruments with kapton windows)
OC/PRS	Orange cryostat (special for PRISMA)
OC/SXD	Orange cryostat (50 mm bore - special for SXD)
OC/D	Orange cryostat (dome tails)
OC/OPT	Ornage cryostat (optical windows)
OC/V	Orange cryostat (vanadium tails)
P/S	Pump set
RP	Rootes pump set
R/STR	Stress rig
SCH/CR	CRISP sample changer
SCH/HR	HRPD sample changer
SCH/LD	LAD sample changer
SCH/LQ	LOQ sample changer **(see Pages 3-9 & 3-11)**
SR	Sorption cryostat
TR/XYZ	XYZ translation stage
UPE	User provided equipment
W/ROT	ω-rotation stage
W/SXD	ω-orienter for SXD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3
4
5
BEAM SIDE
1
2
SHEAR

LOQ Sample Changer Assembly - I. *[Negative 94RB5154]*

3.1 Base Table
- *low table* is shown (height 100 mm). A *high table* is available (height 241 mm).

3.2 Spacer Blocks
- 4 sizes available: 19 mm, 29 mm, 38 mm & 76 mm.

3.3 Jacking Table
- height range is 90 to 200 mm.

3.4 Height Adjuster For Jacking Table

3.5 Unislide Translation Stage
- "old" LOQ translation stage
- fitted with a Heidenhain encoder
- also see Sections 2.13 and 3.14.
- usually carries the *Sample Changer Block* (see Section 3.8) but may also carry specialised sample racks (see Sections 3.24 and 3.31).

3.6 Remote Control Handset
- "old" LOQ translation stage
- also see Section 2.38.

3.7 Stepper Motor Power And Limit Switch Connection
- "old" LOQ translation stage
- also see Sections 2.36 and 2.37.

3.8 Sample Changer Block
- shown without its side walls.
- takes up to 2 sample racks; see Sections 3.17, 3.18 and 3.19.

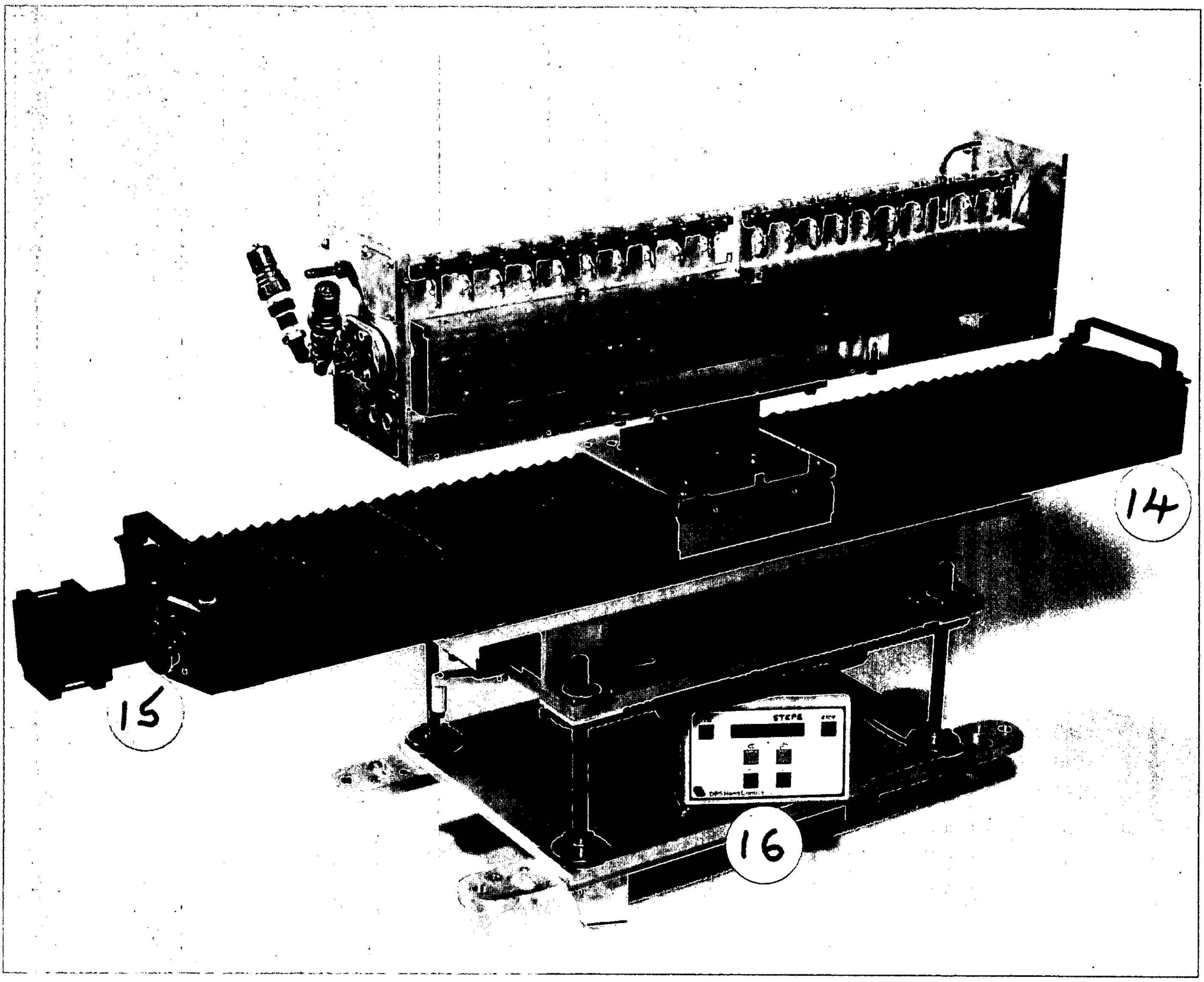
3.9 Thermocouple Connection
- 4-pin LEMO socket for connecting the Pt-100 temperature sensor (see Section 3.13) to the sample position patch panel (Section 2.46) or the *Lauda Circulating Fluid Bath* (see Section 3.41).

3.10 Gas Inlet Connection
- to prevent atmospheric water vapour condensing on sample cells at below ambient temperatures it is standard practice to pass dry nitrogen gas around the sample racks.

3.11 Circulating Fluid Bath Connections
- quick release couplings.
- also see Sections 2.11, 3.41 and 3.47.

3.12 Sample Racks
- also see Sections 3.17, 3.18 and 3.19.

3.13 Platinum Resistance Thermometer
- Pt-100 sensor.
- these are very fragile; please do not put sharp bends in the cable and be careful not to snap the ceramic tip!
- also see Pages 3-3 and 3-4.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16
STEPS

LOQ Sample Changer Assembly - II. *[Negative 95RC2655]*

3.14 Ealing Translation Stage

- "new" LOQ translation stage.
- fitted with an encoder.
- also see Sections 2.33 and 2.34.
- usually carries the *Sample Changer Block* (see Section 3.8) but may also carry specialised sample racks (see Sections 3.24 and 3.31).

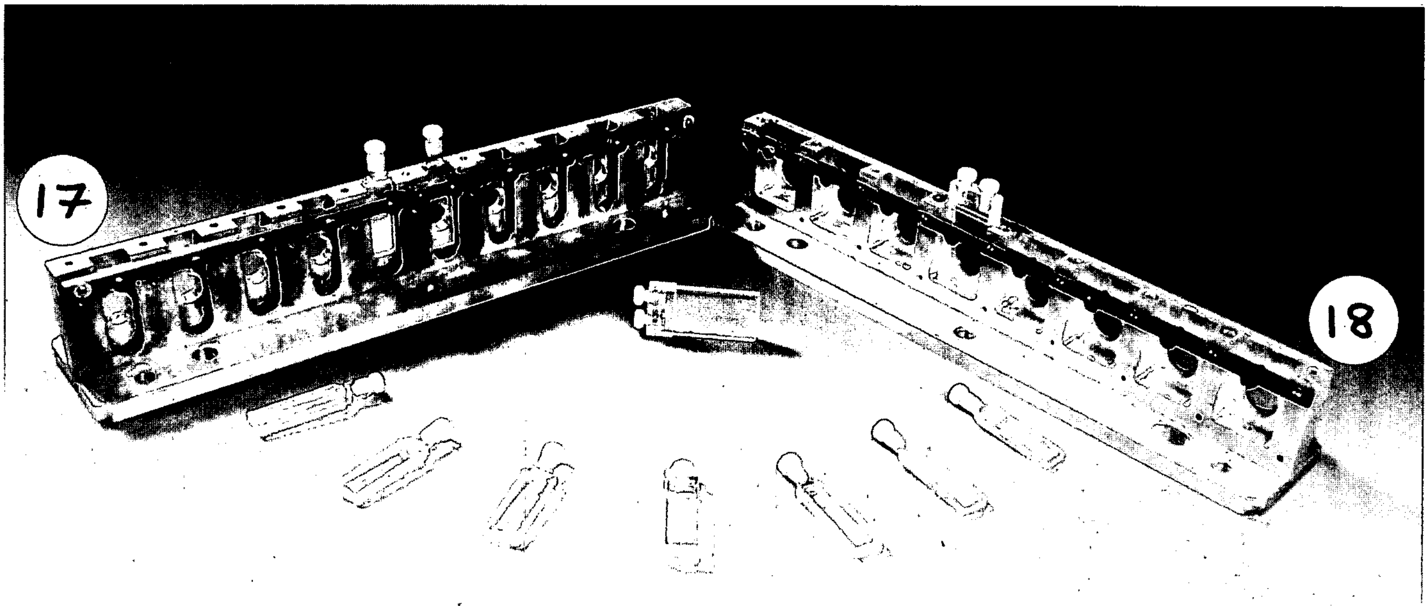
3.15 Stepper Motor Power And Limit Switch Connection

Note:

- the motor drive cable that connects to this socket must not be unplugged unless the *Ealing DPS Control Unit* (see Section 2.33) has been switched off first or damage will result!

3.16 Ealing DPS Remote Control Handset

- "new" LOQ translation stage.
- allows manual control of the position of the *Ealing Translation Stage.*
- also see Sections 2.33, 2.34 and 3.14.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18

LOQ Sample Racks - I. *[Negative 94RC5155]*

There are a variety of sample racks designed to hold most types of commonly available cuvettes and other sample containers.

For SANS, cuvettes should be made of quartz or quartz glass. All other types of glass are unsuitable due to their boron content (boron is a neutron absorber).

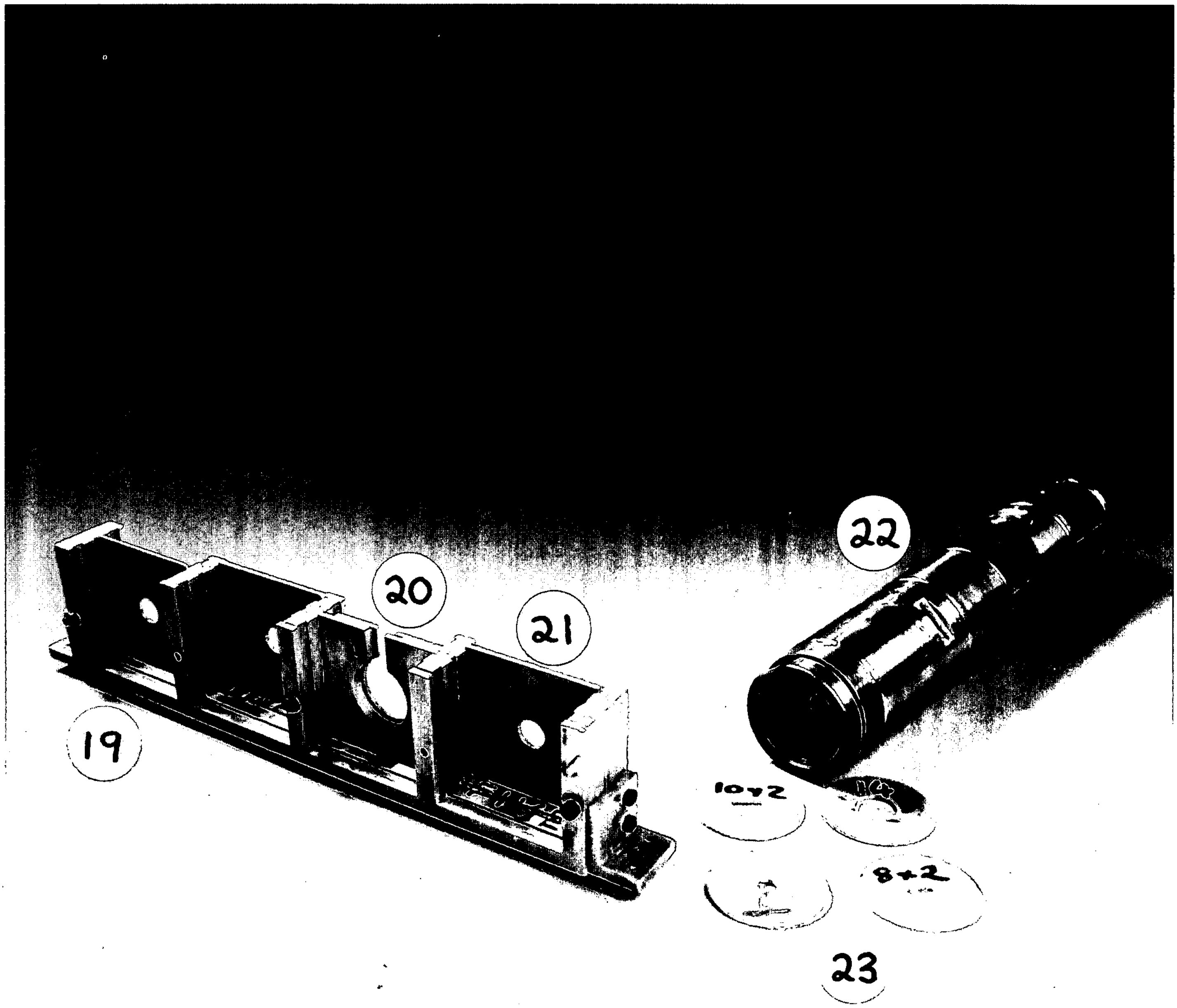
Suitable cuvettes can be purchased from Hellma (who have member companies in the UK, Europe and North and South America) and Optiglass (UK only). Please note that this information is supplied without any endorsement on the part of ISIS, the Rutherford Appleton Laboratory or the Central Laboratory of the Research Councils.

The accompanying photograph shows examples of cuvettes stocked by LOQ and which are available for loan to users, together with the available sample racks.

3.17 10-position x 1 cm wide rack (2 racks)
3.18 8-position x 2 cm wide rack (1 rack)

Cuvettes with pathlengths of 0.1 cm or 0.2 cm will suffice for most experiments, though 0.5 cm or even 1.0 cm pathlength cells may prove useful with very dilute samples so long as the solvent is not hydrogenous! The cuvettes shown are approximately 3.0 cm in height but need not be filled beyond $^{2}/_{3}$ of this.

The tops of the racks have 3 mm diameter holes drilled in them which accomodate cartridge heaters and/or thermocouple sensors.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10+2
8+2
23

LOQ Sample Racks - II. *[Negative 94RC5153]*

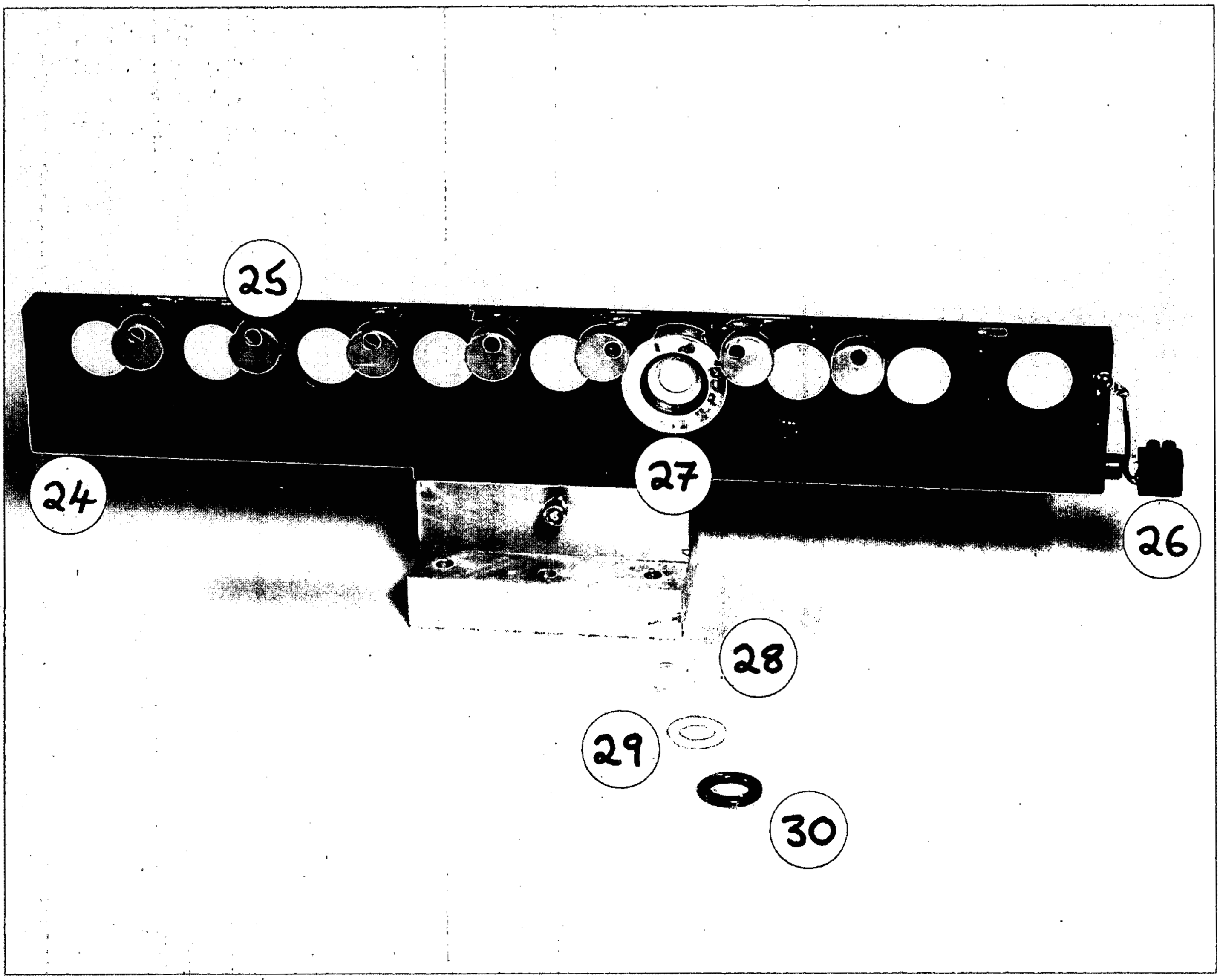
3.19 4-position x 5 cm wide rack (2 racks)

3.20 Cell Holder
- for disc-shaped cells with necks.
- also see Section 3.32.

3.21 Cadmium Masks
- a number of these are available with a range of central hole sizes from 6 mm diameter up to 12 mm diameter.
- these masks are often useful for mounting samples that cannot be contained in cuvettes; for example, the sample can be wrapped in thin aluminium foil and simply taped to the back of a mask.

3.22 Collimation Snout
- screws together in vacuum-tight sections to allow collimation of the incident beam right up to the sample.
- the brass ring right on the end nearest the camera may be unscrewed without loss of vacuum to reveal a quartz window in front of which cadmium apertures (see Section 3.23) may be inserted.
- also see Section 2.6.

3.23 Cadmium Apertures
- these apertures provide the final beam definition before the sample.
- there are a range of apertures; those with central holes from 6 mm diameter up to 15 mm diameter and those with 6 x 2, 8 x 2 and 10 x 2 mm slits.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LOQ Sample Racks - III. *[Negative 95RC2657]*

This rack, together with its specialised sample cells, was designed and built by Prof R W Richards and his research group at the University of Durham for SANS studies of molten polymers. Temperatures of up to 230 °C are possible. The rack is normally resident at ISIS and available for use by other users, though they will need to provide their own cells.

3.24 Durham High Temperature Sample Rack
- 9-position brass rack with integral high power cartridge heater.

3.25 Retaining Device
- quick-release retaining device to keep the sample cells in place.

3.26 Cartridge Heater Power Connection
- the heater is rated to 240 VAC.
- also see Section 2.35.

3.27 Sample Cell
- the cell consists of an aluminium annulus with a brass centrepiece. A hole in the aluminium allows a thermocouple to be placed in direct contact with the brass centrepiece for monitoring of the sample temperature.
- the sample is sandwiched between the quartz windows and contained inside a third PTFE washer. The pathlength of the sample is determined by the thickness of the washer, typically 1 mm. Maxium pathlength is 5 mm.
- the outer diameter of the cell is 39.5 mm.

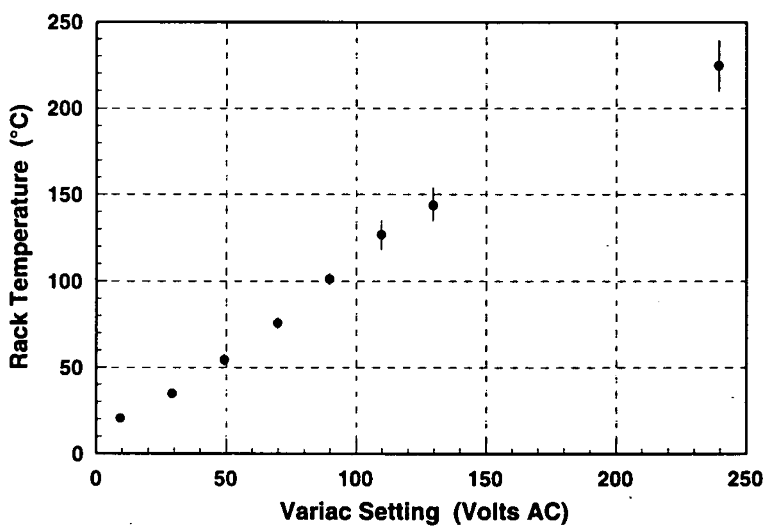
3.28 Quartz Window (1 of 2)
- 1 mm thick and 20.5 mm in diameter.

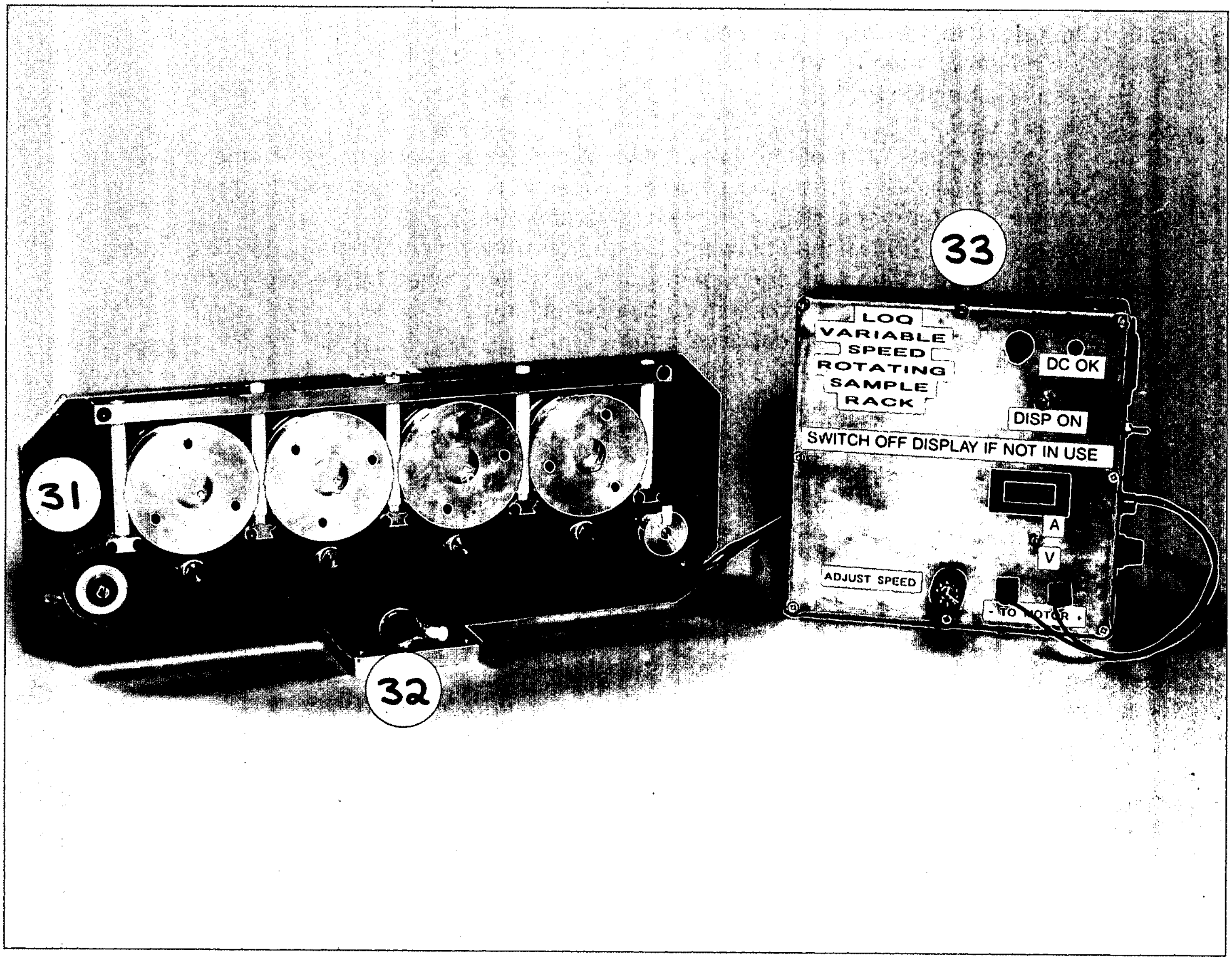
3.29 PTFE Washer (1 of 2)
- 1 mm thick and 20.5 mm in diameter.

3.30 Threaded Locking Ring
- used to clamp the cell components encasing the sample together.
- the inner diameter of the ring (and the brass back to the cell) is 14 mm.

Eurotherm Temperature Controller Settings:

Maximum Output Power	: 50%	Propband	: 0.5%
Cycle Time	: 1.0s	Integration Time	: 200s
Setpoint High	: 50.00	Derivative Time	: 40s





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LOQ
VARIABLE
SPEED
ROTATING
SAMPLE
RACK
DC OK
DISP ON
SWITCH OFF DISPLAY IF NOT IN USE
A
V
ADJUST SPEED
- TO MOTOR +

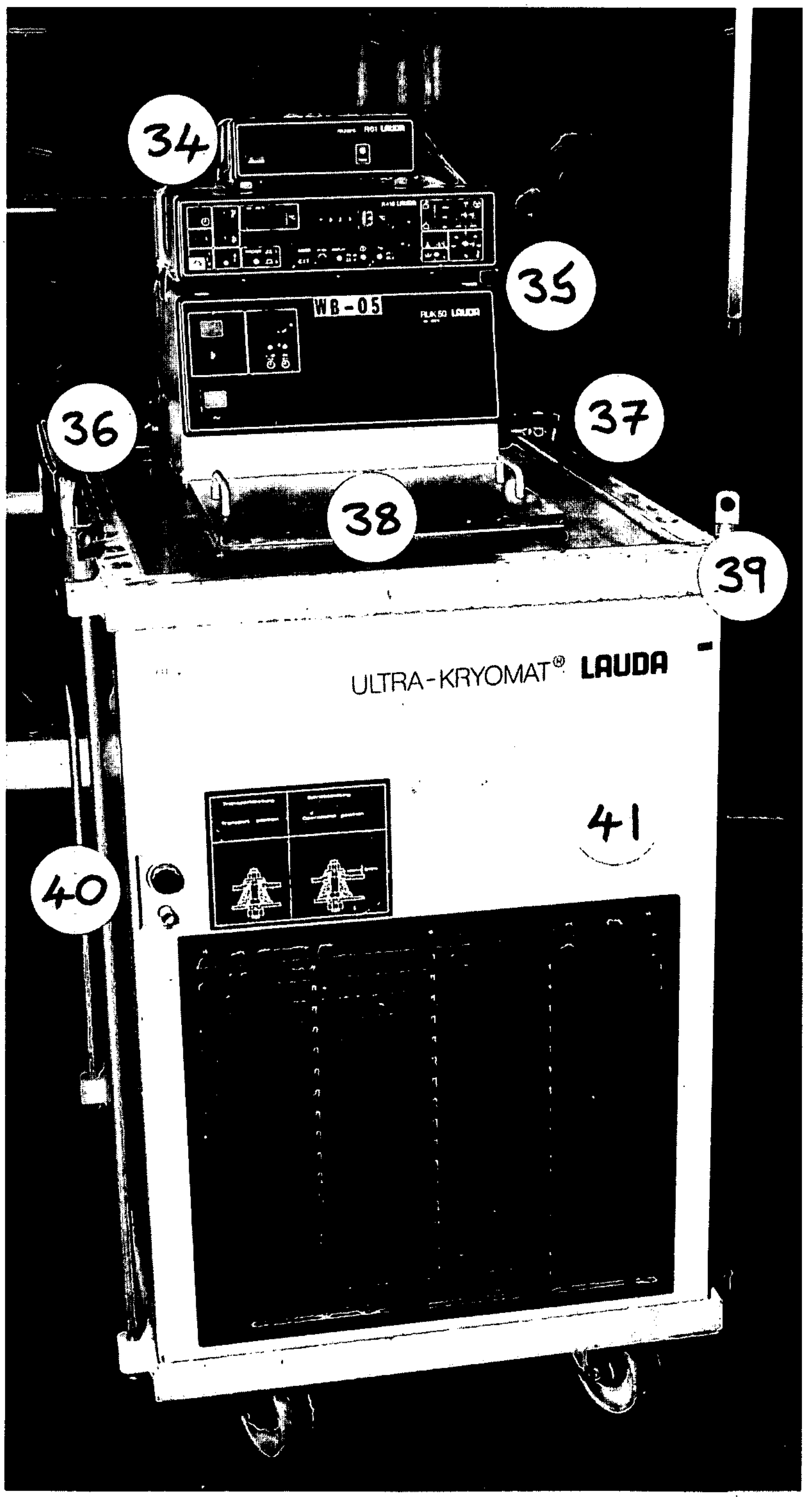
LOQ Sample Racks - IV. *[Negative 95RC2656]*

This rack is designed to rotate samples about the axis of the neutron beam. Typical applications are in the study of systems that sediment on the timescales of normal SANS measurements (eg; less than 1 hour). There is no provision for temperature control.

3.31 Rotating Sample Rack
- 4-position rack with rotating sample holders.
- choice of three different motors for optimum control of rotational speed.

3.32 Disc-shaped Quartz Cuvette
- the rack can accomodate cells with pathlengths of 0.1 or 0.2 cm. The cuvette shown has a diameter of 22 mm and overall height of 39 mm.

3.33 Speed Control Box
- provides variable control of the rotational speed between 0 to 40 rpm, 0 to 65 rpm or 0 to 130 rpm, depending on the motor installed.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WB-05
ULTRA-KRYOMAT® LAUDA

Lauda RUK50 Ultra-Kryomat® Circulating Fluid Bath. *[Negative 94RC5158]*

(ISIS Water Bath WB-05)

3.34 Lauda R61 RS232 Communications Interface

3.35 Lauda R410 Control Unit

- this is described in more detail overleaf.

3.36 Main Power Switch

- to turn the bath ON, rotate the switch until a "1" appears in the hole.
- to turn the bath OFF, rotate the switch until a "0" appears in the hole.

3.37 Hose Connections And Flow Control

- the external flow and return connections off the bath are fitted with approximately 0.5 m of lagged hose terminating in male quick release connectors. The bath should be connected to the apparatus to be thermostatted (such as the sample changer block) using additional hoses.
- also see Sections 2.11 and 3.11.
- the flowrate in the external circuit may be controlled by the sliding lever; move towards the front of the bath to increase the flowrate and move towards the rear of the bath to decrease the flowrate. Normally this control is left in the fully open position except when connecting/disconnecting the fluid bath hoses.

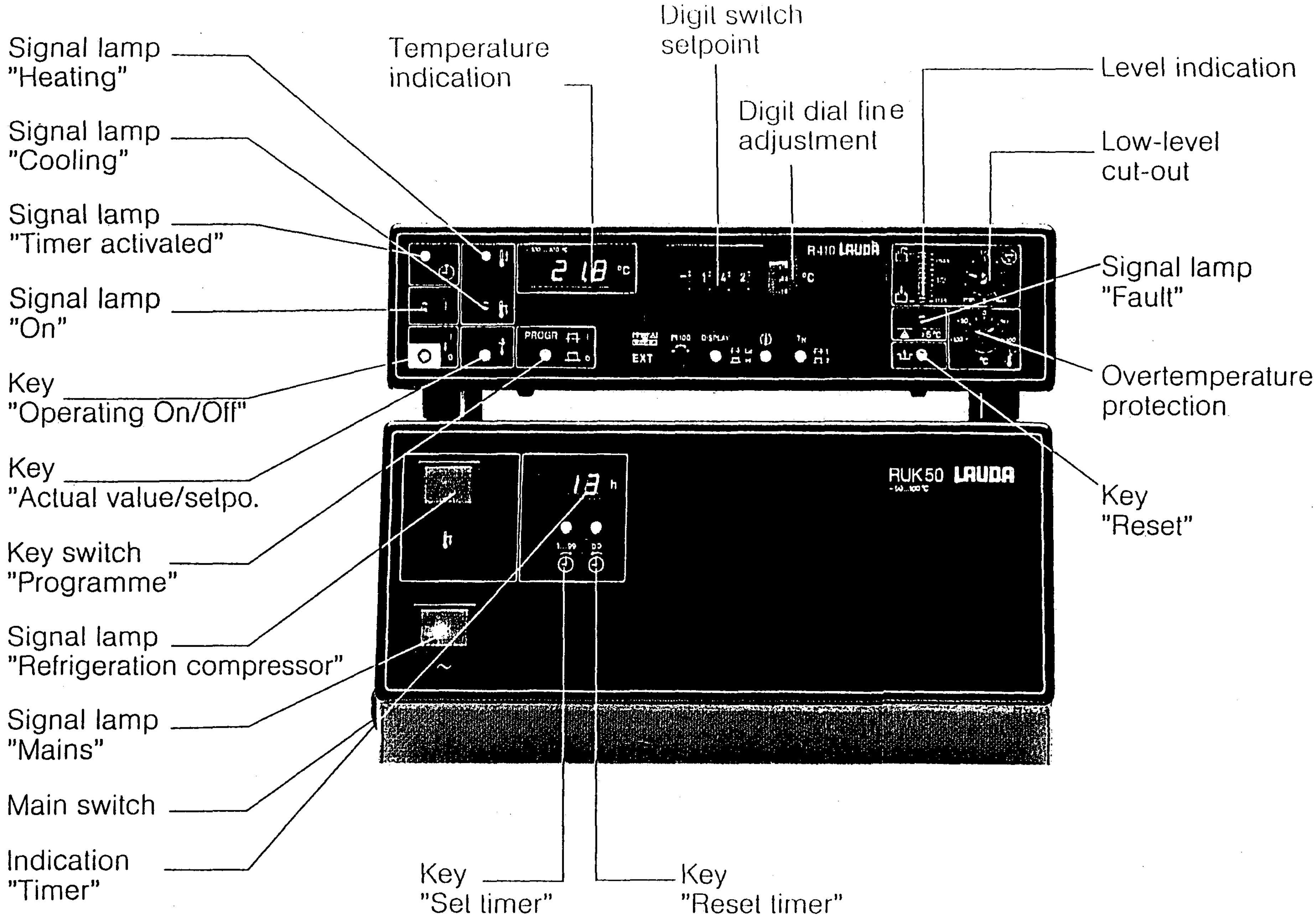
3.38 Bath Cover

3.39 Lifting Point

- the bath sits in a self-contained lifting frame. During crane operations the bath should be slung evenly from these four lifting points.

3.40 Drain Cock

3.41 RUK50 Circulation Thermostat



Controls and indications on R 410 and unit deck

IID/SE/N1/92, Hari Shah, 26 May 1992, Revised Steve King, 10 May 1995

Operating Instructions For The Lauda Circulating Fluid Bath

1. Description

The *Lauda Circulating Fluid Bath* consists of four different pieces of equipment connected together to form a single package (hereafter referred to as the Lauda bath).

The main unit is the *RUK 50 Ultra Kryomat* which is a floor standing model of compact construction mounted on four swivelling castors. The main unit is a low temperature bath/circulation thermostat with an operating range of -50 to +100 °C depending on the fluid fill. The entire bath is fitted with a lifting framework so that it can be craned to the point of use without draining.

Suspended from the stainless steel top plate is a large, highly insulated, bath containing the evaporator coil of the refrigeration system. Mounted at the back of the bath are the circulating pump, heater, and probes for temperature and level measurement. The front part of the bath is freely accessible but is normally enclosed by the bath cover. The circulating pump is of the pressure/suction type with automatic level control.

The controller that operates the Lauda bath and maintains the bath temperature sits on top of the main structure near the bath cover and comes in two parts; the bottom unit contains signal lamps and the timer controls whilst the top, *R 410*, unit contains the remainder of the controls. On top of the *R 410* unit sits an RS232 interface called the *R 61*. This allows computer control and monitoring of the Lauda bath via the CAMAC system. The figure opposite shows all the indicators and controls on the two units.

2. Circulating Fluid

Normally the Lauda bath should be supplied with an appropriate fluid fill and the various controls preset as required. If not, then make sure that the bath is filled to a level approximately 1 cm below the bath cover with the appropriate fluid for the temperature range to be used for the experiment. The Lauda bath is normally operated with a 50:50 mix of water and ethylene glycol. This gives an operational temperature range of about -20 to +95 °C. For temperatures below -20°C it is necessary to use special silicone oil and hoses. For temperatures above +95°C it is necessary to either reduce the water content of the glycol mixture or to use a special mineral oil. <u>Never mix water/glycol and oil, in the bath, the sample environment being used or in the hoses</u>! Please check the fluid level again after the bath has been connected up with the appropriate hoses as long hoses will obviously reduce the level of the fluid in the bath and cause poor flow and inefficient temperature control.

3. Manual Operation

(a) Turn the Main Power Switch on. The green "Mains" signal lamp should come on, as will various other indicator lamps.
(b) For manual control ensure that the pushbutton "Programme" is out ("0").

(c) Select the desired temperature with the thumbwheel switches marked "Digit switch setpoint". These switches give 1 °C resolution. (Don't forget to select the sign, + / -, of the temperature you are setting!). The decimal places are set in analogue form with the knurled wheel marked "Digit dial fine adjustment" with a resolution of 0.01 °C.
(d) The actual bath temperature, or the bath setpoint, can be read digitally on the green LED display marked "Temperature indication" to a resolution of 0.1 °C depending on whether the pushbutton marked "Actual value/setpo" is out or in respectively. The "Heating" and "Cooling" lamps switch on and off as needed by the bath controller.

An external platinum resistance thermometer can also be used to to monitor the temperature of a sample which may be situated at some distance away. If connected, the temperature it registers can be displayed on the LED readout by pushing the button marked "Display" in ("Ext"). To revert to a temperature display from the baths internal sensor simply release this pushbutton ("Int").

Note:

It is inadvisable to attempt to control the Lauda bath on its external sensor. This is because the controller is unable to use full PID control with the external sensor.

4. Computer Control (Lauda ⟷ CAMAC)

Note:

Negative temperatures cannot be set directly from CAMAC. If it is necessary to set temperatures below 0.00°C then a PC must be used to translate Kelvin setpoints into Celsius setpoints. See below.

(a) Ensure that the RS232 cable from the back of the R 61 interface connects to a SP232 card in slot 3 of the CAMAC crate (see Section 2.17).
(b) Turn the Main Power Switch on. The green "Mains" signal lamp should come on, as will various other indicator lamps.
(c) Turn on the R 61 interface and push the white button marked "Reset".
(d) For computer control ensure that the pushbutton "Programme" is in ("1").
(e) Set the thumbwheel switches marked "Digit switch setpoint" to zero. Failure to do this will result in a permanent temperature offset being applied to whatever temperature setpoint is issued by computer. Also set the knurled wheel marked "Digit dial fine adjustment" to "0,10".
(f) The actual bath temperature, or the bath setpoint, can be read digitally on the green LED display marked "Temperature indication" to a resolution of 0.1 °C depending on whether the pushbutton marked "Actual value/setpo" is out or in respectively. The "Heating" and "Cooling" lamps switch on and off as needed by the bath controller.
(g) Set the desired temperature. See below.

5. Computer Control (Lauda ⟷ PC ⟷ CAMAC)

Also see the previous section.

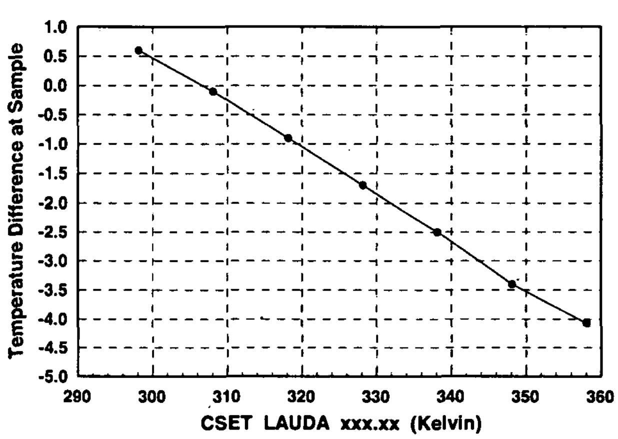
(a) Ensure that the RS232 cable from the back of the R 61 interface connects to COM2: on a PC (see Section 2.31). Ensure that COM1: on a PC connects to a SP232 card in slot 3 of the CAMAC crate (see Section 2.17).

(b) Turn on the PC. After it has booted, type *L* for the LOQ setup and then:
CD LOQ
CD LAUDA
QBASIC LAUDA.BAS
[Shift]-F5
(c) Turn the Main Power Switch of the Lauda bath on. The green "Mains" signal lamp should come on, as will various other indicator lamps.
(d) Turn on the R 61 interface and push the white button marked "Reset".
(e) For computer control ensure that the pushbutton "Programme" is in ("1").
(f) Set the thumbwheel switches marked "Digit switch setpoint" to "025" and the knurled wheel marked "Digit dial fine adjustment" to "0,10".
(g) The actual bath temperature, or the bath setpoint, can be read digitally on the green LED display marked "Temperature indication" to a resolution of 0.1 °C depending on whether the pushbutton marked "Actual value/setpo" is out or in respectively. The "Heating" and "Cooling" lamps switch on and off as needed by the bath controller.
(h) Press *[Return]* on the PC as instructed. Eventually a message will appear which says that the interface has been calibrated and that the setpoint has been set to 298.16 K.
(I) Set the desired temperature. See below.

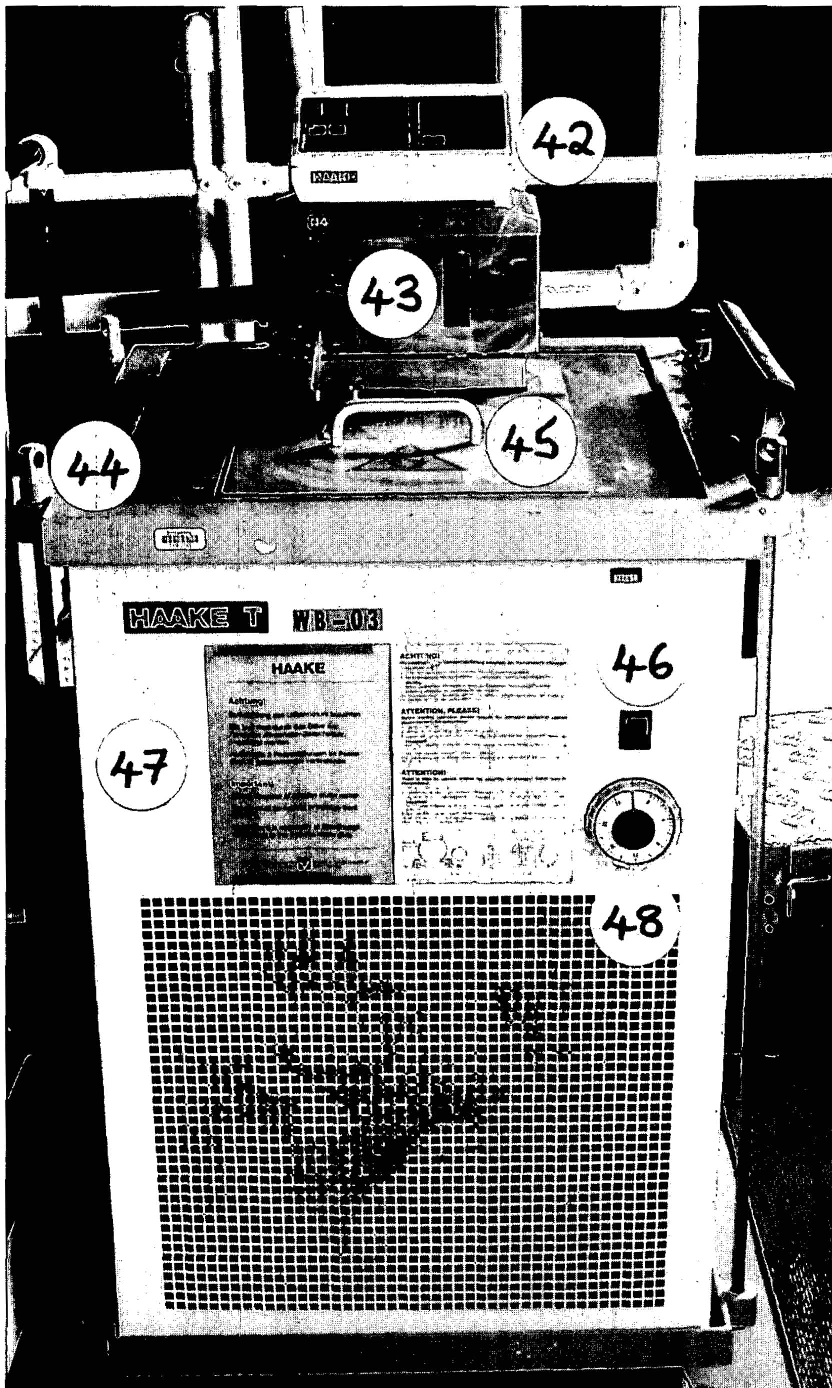
6. CAMAC Commands For Setting / Logging The Lauda Bath Temperature

CSET LAUDA xxx.xx	sets the bath temperature to xxx.xx degrees <u>Kelvin</u>
CSET LAUDA/LOG=n	starts logging of the bath sensor every n seconds
CSET LAUDA/NOLOG	stops logging of the bath sensor
CSET BLOCK/LOG=n	starts logging of the external sensor every n seconds
CSET BLOCK/NOLOG	stops logging of the external sensor
CSHOW BPAR/ENQ	reads and displays the setpoint value

The following figure shows the difference between the requested bath setpoint and the temperature of a light oil in a 10 x 1 mm quartz cuvette in the appropriate sample rack (see Section 3.17) mounted on the sample changer block (see Section 3.8). The sample changer block had both sides and its lid fitted. Equilibration times ranged from 20 minutes to 40 minutes at the higher temperatures. The difference primarily arises as a result of heat loss from the fluid hoses (see Section 2.11) connecting the bath and sample changer block.



Over the range shown: *{Actual Temperature} = 0.921 x {Setpoint} + 24.306*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HAAKE T
HAAKE
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Haake N4 Circulating Fluid Bath. *[Negative 94RC5156]*

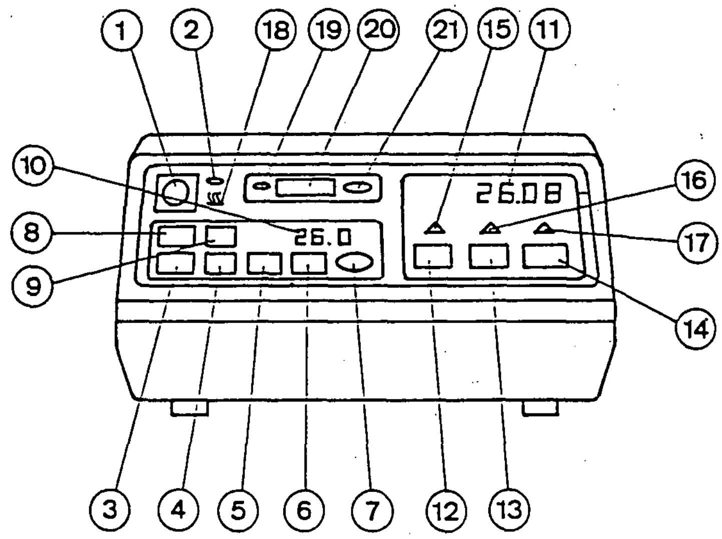
(ISIS Water Bath WB-03)

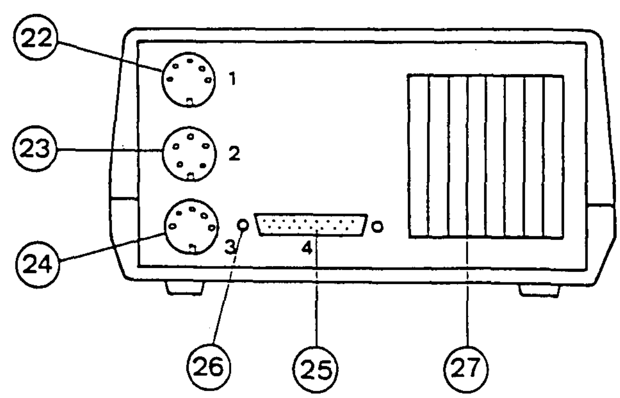
Also see Section 2.39.

The flow control is on the left side of the unit housing the level meter (see Page 3-29).

3.42 Haake N4 Control Unit
3.43 Fluid Level Meter
3.44 Bath Cover
3.45 Lifting Point
- the bath sits in a self-contained lifting frame. During crane operations the b ath should be slung evenly from these four lifting points.

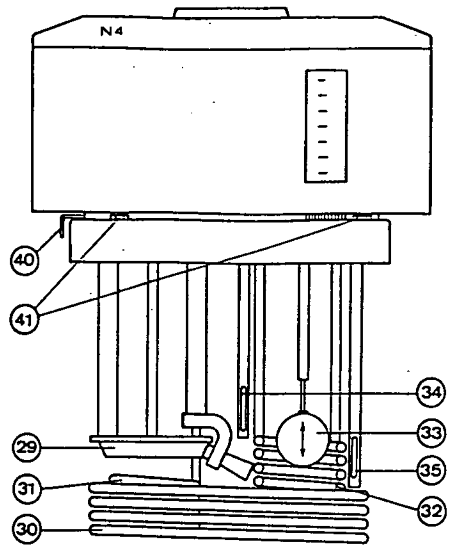
3.46 Main Power Switch
3.47 Haake N4-T Circulation Thermostat
3.48 Timer
- not normally used.





(1) Main Switch
(2) Power Control Light (Green)
(3) Preset Value Key '100' (Values in °C)
(4) Preset Value Key '10' (Values in °C)
(5) Preset Value Key '1' (Values in °C)
(6) Preset Value Key '0.1' (Values in °C)
(7) Preset Value Key - fine Adjustment '0.01' (Values in °C)
(8) Remote Control Key 'EXT' - External Temperature Presetting
(9) Preset Value Sign '-'
(10) Digital Indication of Preset Value
(11) Digital Indication of Actual Temperature
(12) Selection Key - Actual Bath Temperature 'INT'
(13) Selection Key - Actual Bath Temperature 'EXT'
(14) Selection Key - Excess Temperature Cut-Off 'ALARM'
(15) Switch Position Indicator - Key 12
(16) Switch Position Indicator - Key 13
(17) Switch Position Indicator - Key 14
(18) Heater Control Light
(19) Potentiometer for setting Excess Temperature Cut-Off Point
(20) Breakdown Control Indicator 'ALARM'
(21) Release Switch - Restarts Instrument after Breakdown
(22) Socket for connecting a Remote Temperature Controller
e.g. HAAKE Temperature Programmer
(23) Socket - Monitor Functions
(24) Socket for connecting an External Temperature Sensor Pt-100
(25) Male Plug (25 Pins) - For connecting Control Head to Module N-4
(26) Tapped Holes for securing Flat Cable Plug
(27) Heat Sink

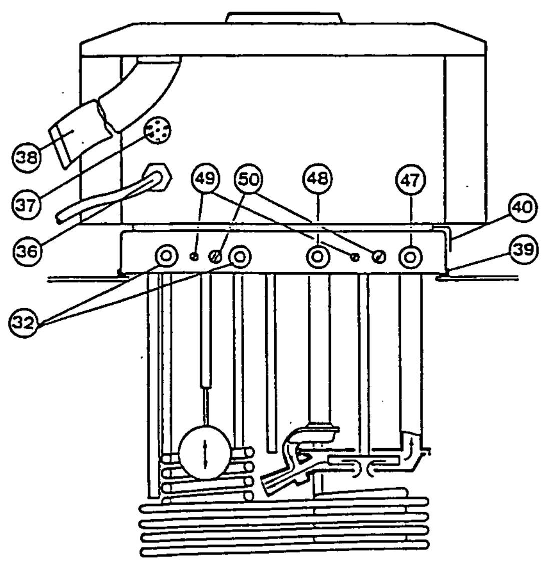
Front Side



(29) Pump
(30) Heating Coil 2,000 W
(31) Heating Coil 1,000 W •
(32) Cooling Coil
(33) Float of Level Switch
(34) Actual Temperature Sensor Pt-100
(35) Control Temperature Sensor Pt-100
(40) Flow Rate Control Lever
(41) Front Clamping Screws
(Spanner Width: 8 mm)

• Heating coil rendered inactive by removing the fuse.

Rear Side



(32) Cooling Coil Fittings
(33) Power Cable for Module N 4
(37) Socket for Control Cable to connect 'Cooling Baths' as well as optional 'Control Box ZS 1'
(38) Flat Cable for connecting Control Head
(39) Gasket
(40) Flow Rate Control Lever for Pressure Pump
(47) Pressure Port Fitting
(48) Suction Port Fitting
(49) Fastening Screws
(50) Eccentric Locking Screws

Steve King, 12 May 1995

Operating Instructions For The Haake Circulating Fluid Bath

1. Description

The *Haake Circulating Fluid Bath* consists of two different pieces of equipment connected together to form a single package (hereafter referred to as the Haake bath).

The main unit is the *T Cryostat,* a floor-standing low temperature bath/circulation thermostat mounted on four swivelling castors. The operating range is -50 to +50°C depending on the fluid fill. The entire bath is fitted with a lifting framework so that it can be craned to the point of use without draining.

Suspended from the stainless steel top plate is a large, highly insulated, bath containing the evaporator coil of the refrigeration system. Mounted at the back of the bath are the circulating pump, heater, and probes for temperature and level measurement. The front part of the bath is freely accessible but is normally enclosed by the bath cover. The circulating pump is of the pressure/suction type with automatic level control.

The controller that operates the Haake bath and maintains the bath temperature, called the *N4 Module,* sits on top of the circulation thermostat and contains signal lamps, temperature setting controls and two red LED readouts; one of the bath temperature (always displayed) and one of the bath setpoint (only displayed when the bath is being controlled manually). Computer control and monitoring of the Haake bath is possible. Note however that, unlike the Lauda bath, the Haake bath is controlled directly by the LOQ FEM (see Section 2.23) and not by the CAMAC system.

2. Circulating Fluid

Normally the Haake bath should be supplied with an appropriate fluid fill and the various controls preset as required. If not, then make sure that the bath is filled to a level approximately 1 cm below the bath cover with the appropriate fluid for the temperature range to be used for the experiment. The Haake bath is normally operated with a 50:50 mix of water and ethylene glycol. This gives an operational temperature range of about -5 to +50°C. For temperatures below -5°C it is necessary to use special silicone oil and hoses. The bath is physically unable to attain temperatures above +50°C. Never mix water/glycol and oil, in the bath, the sample environment being used or in the hoses! Please check the fluid level again after the bath has been connected up with the appropriate hoses as long hoses will obviously reduce the level of the fluid in the bath and cause poor flow and inefficient temperature control.

3. Manual Operation

(a) Turn the Main Power Switch on. The green signal lamp in the switch should illuminate, as will various other indicator lamps on the *N4 Module*. If they don't, turn the *N4 Module* on as well!

(b) For manual control ensure that the red signal lamp on the button marked "EXT" is extinguished.

(c) Select the desired temperature by holding down any of the five "Preset Value Keys" until the desired temperature is displayed on the smaller of the two red LED readouts. These buttons allow the temperature to be set to a resolution of 0.01 °C. (Don't forget to select the sign, + / -, of the temperature you are setting!).

(d) The actual bath temperature can be read digitally on the larger red LED display to a resolution of 0.01 °C. The "Heater Control Lamp" will switch on and off as needed by the bath controller.

An external platinum resistance thermometer can also be used to to monitor the temperature of a sample which may be situated at some distance away. If connected, the temperature it registers can be displayed on the LED readout by pushing the button marked "EXT" until the red signal lamp illuminates. To revert to a temperature display from the baths internal sensor simply push this button again until the red signal lamp goes out.

4. Computer Control (Haake ←→ LOQ FEM)

(a) Ensure that the RS232 cable from the back of the *Haake IF 24-1 interface* (see Section 2.39) connects to terminal line TTA1: on the back of the LOQ FEM (see Section 2.23).
(b) Ensure that the 4-way signal cable from the back of the *Haake IF 24-1 interface* connects to sockets "1" and "2" on the back of the *N4 Module.*
(c) Turn the Main Power Switch on. The green signal lamp in the switch should illuminate, as will various other indicator lamps on the *N4 Module*. If they don't, turn the *N4 Module* on as well!
(d) For computer control ensure that the red signal lamp on the button marked "EXT" is illuminated. The smaller red LED display of the bath setpoint will be extinguished.
(e) The actual bath temperature can be read digitally on the larger red LED display to a resolution of 0.01 °C. The "Heater Control Lamp" will switch on and off as needed by the bath controller.
(f) Set the desired temperature. See below.

5. CAMAC Commands For Setting / Logging The Haake Bath Temperature

CSET HAAKE xx.xx	sets the bath temperature to xx.xx degrees Celsius
CSET HAAKE/LOG=n	starts logging of the bath sensor every n seconds
CSET HAAKE/NOLOG	stops logging of the bath sensor
CSHOW HAAKE	displays the bath setpoint, temperature, limits, etc

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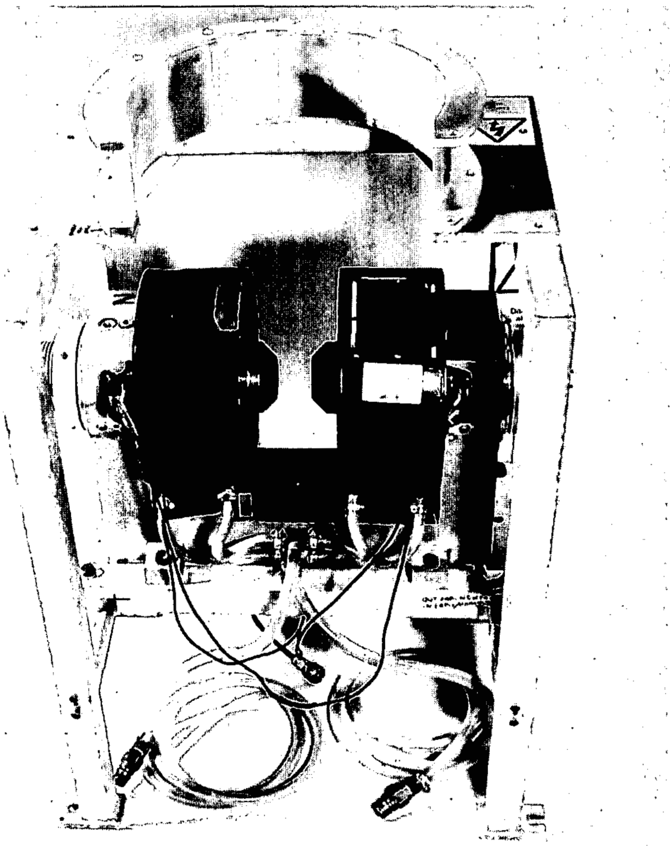

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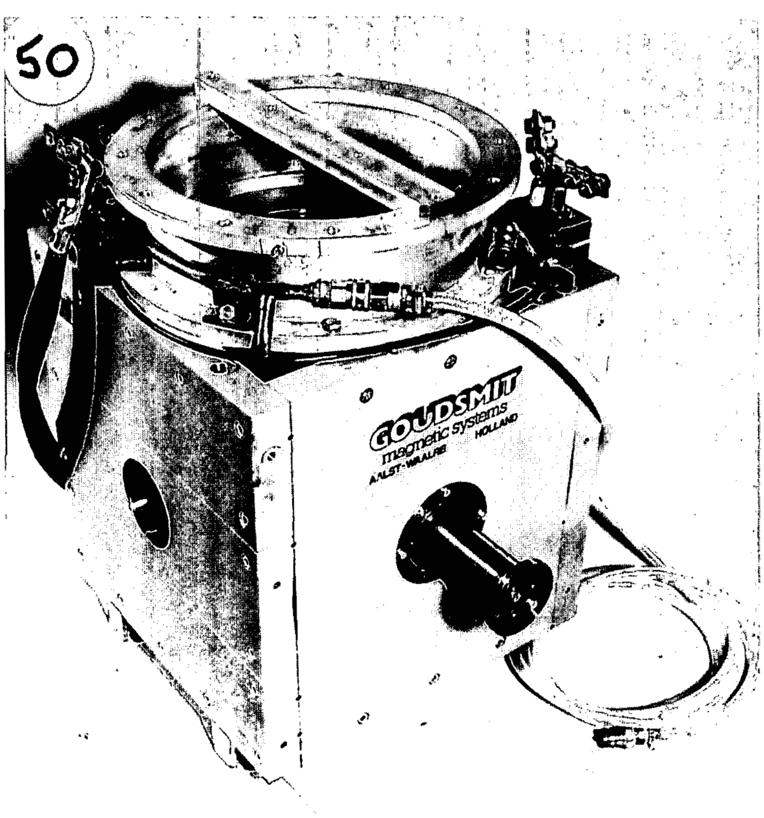


Electromagnets

There is a choice of two water-cooled electromagnets; the *Newport* and the *Goudsmit.* Both accept ISIS 50 mm bore "Orange" *helium cryostats* and *Leybold helium closed-cycle refrigerators* in addition to a LOQ-specific single cell holder. This latter device accepts most types of quartz cuvette normally used with the LOQ sample changer whilst a combination of thermostatted circulating fluid and electrical heaters provides a usable temperature range of approximately +15 to +250°C. Neither magnet can accept a RAL furnace (see Page 3-43).

3.49 Newport Electromagnet. *[Negative 94RB5935]*

Transverse magnetic field only.

The magnet is shown fitted in the ISIS Tomkinson Frame and ready for installation on LOQ. The semicircular flange at the top is an adaptor ring used with orange cryostats.

This magnet provides a magnetic field of approximately 1.0 T with a pole gap of 20 mm, decreasing to about 0.5 T at the maximum pole gap of 100 mm (see overleaf).

The time required for installation is about ¾ hour.

3.50 Goudsmit Electromagnet. *[Negative 94RB5934]*

Note: This magnet weighs 0.8 tonnes!
Furthermore, because it may be used on CRISP, use of this magnet must be requested on a proposal form so that it can be scheduled accordingly.

Transverse or solenoidal magnetic field.

The magnet is depicted ready for installation. Note one of the two removable soft iron pole pieces in the foreground. These are removed when the magnet is used in its solenoidal mode. *Note: This magnet is limited to 600 Amps when used on CRISP.*

This magnet can generate a magnetic field of 2.0 T with pole gaps of less than 50 mm. The magnetic field strength versus power supplied graph for a pole gap of 90 mm (the maximum possible) is shown overleaf, for both the transverse and solenoidal modes of operation.

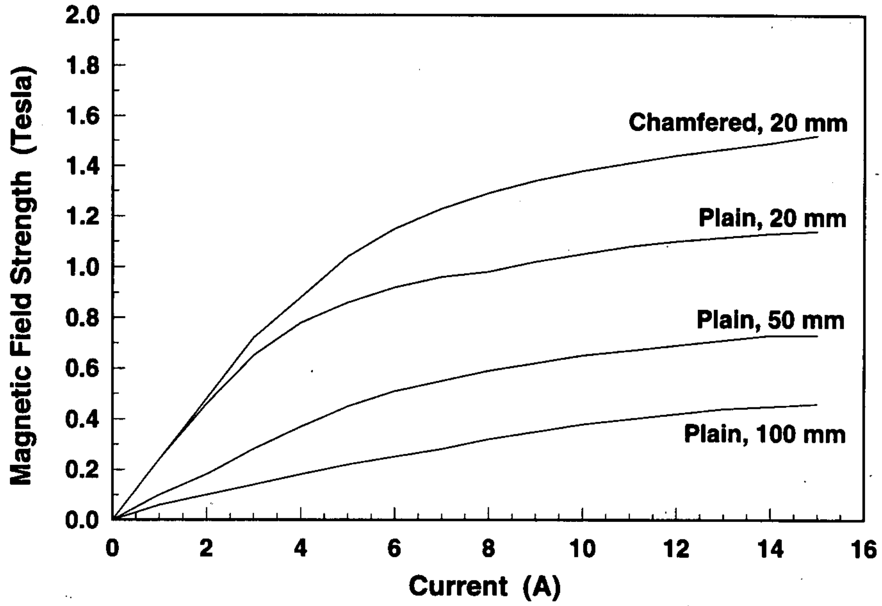
The time required for installation is about 2 hours.

Note:

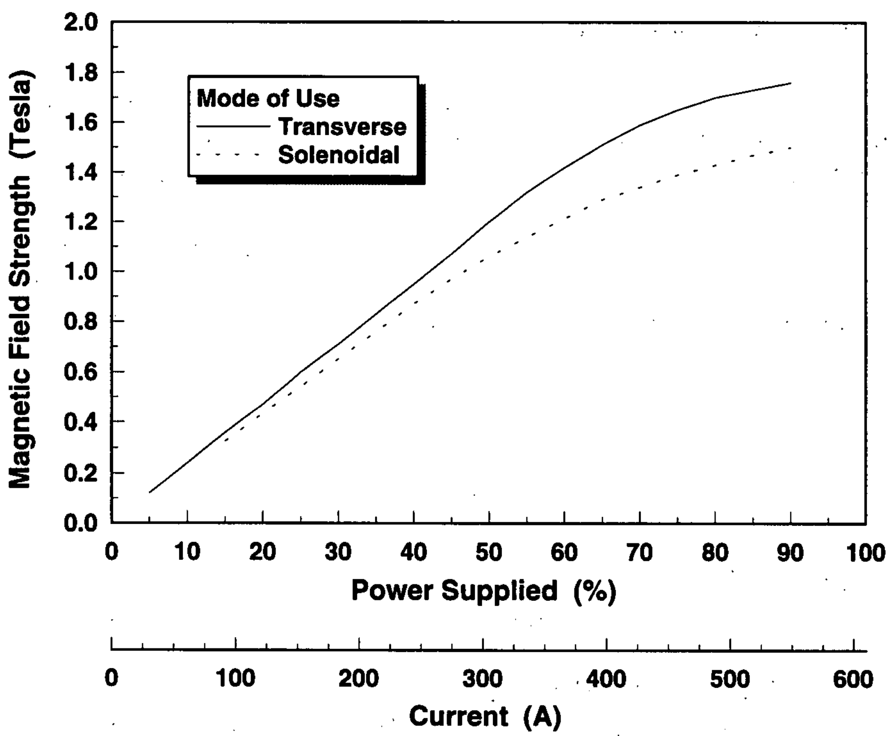
TRANSVERSE MODE
Neutron beam in horizontal plane, perpendicular to horizontal magnetic field.

SOLENOIDAL (OR LONGITUDINAL) MODE
Neutron beam in horizontal plane and along the direction of the magnetic field.

Newport Electromagnet
(pole piece type & gap specified, transverse mode)



Goudsmit Electromagnet
(90 mm pole gap in transverse mode)



Richard Heenan, November 1994, Revised Steve King, 5 December 1995

Operating Instructions For The Goudsmit Electromagnet

1. Safety
The power supply for this magnet is situated on the balcony above the LOQ Cabin. It is a modified 50kW inverter capable of providing 615 Amps at up to 100 Volts! Though this voltage would not kill a healthy person the stored energy is extremely dangerous! A short circuit would cause violent sparks and severe burns.

Electrical connections to the magnet are interlocked and covered so that there are no exposed live terminals. Under no circumstances should users interfere with the electrical connections; these will be made by an instrument scientist or some other approved person.

The high magnetic field inside the magnet is a potential hazard! Access to the magnet is possible whilst the magnet is live, but should not be necessary in most circumstances. Access may be required, for example, for measurements of the field strength; or to adjust a cryostat if turning off the field would irreversibly destroy the sample alignment or if hysteresis effects are important. If you do need to approach the magnet whilst it is running then remove all loose metal objects from your person - such as keys, watches, steel rulers etc., and <u>never</u> attempt to use tools such as spanners or screwdrivers either near to or within the poles of the magnet.

The “MAGNET ON” light remains on, even if the magnet has tripped off. Users must ascertain separately (by visiting the power supply on the balcony if need be) whether the magnet is live before approaching it. <u>If in doubt assume that the magnet is in fact live</u>! The local control (black) box inside the sample enclosure has a red “EMERGENCY OFF” button. This box also has an orange LED which indicates if the interlock circuits are on. If this LED is not lit then the magnet has tripped off. There is also a blue interlock box on the wall of the LOQ Cabin computer room (immediately on the right as you open the door). The key in this box unlocks the case surrounding the electrical connections in the sample enclosure. Removing this key isolates the magnet from the power supply and so can also be used in an emergency.

Close up to the live magnet the stray fields may exceed the 0.5 mT (50 Gauss) limit recommended as safe for pacemakers and so anyone wearing such a device is advised to not to enter the sample enclosure. Stray fields on the footbridge above LOQ sample position are well within this limit.

At present, with a demineralised water flowrate of 20+ Lmin^{-1}, the maximum operating power is around 80%. Above this setting the magnet will ultimately trip off but the magnet can be safely run to full power for short periods.

2. Installation
Only those persons with valid RAL Lifting Permits are allowed to operate the crane.

If the magnet is to be rotated 90° from its *transverse* to its *solenoidal* orientation then it is necessary to completely disconnect it - this operation requires the presence of an instrument scientist, or other approved person - and to remove it from the pit. This is because the steel plate on top of the magnet base table (not shown in the photograph) must also be rotated by 90°. In addition removal of the soft iron pole pieces is a lot easier outside of the confines of the sample enclosure.

Ensure that the separate earth cable is bolted to the casing of the magnet.

Ensure that the trip leads are properly and fully connected to the local control box in the sample enclosure.

Ensure that the demineralised cooling water is connected and turned on. The taps (which have red handles) are situated at foot level behind the electronics rack on the CRISP side of the LOQ sample position (see Page 2-16). Please watch for water leaks; a flood could be catastrophic and would not make you popular as the demineralised water supply is also used by the ISIS accelerator magnets!

3. Local Control Box

This black box in the sample enclosure (which must be within easy reach) features:

- a mains power lead coming in from the power supply on the balcony.
- connections for water flow and magnet temperature trips (these are simple switches and so the three connectors are interchangeable).
- a mains lead going out to the "MAGNET ON" light.
- an orange LED next to a white "RESET" button. The magnet can only be s uccessfully powered up if this LED is lit.
- a large red "EMERGENCY OFF" button.

4. Switching the Magnet ON

The telephone on the balcony next to the power supply is extension 5199.

Warning: do not interfere with any of the other power supplies on the balcony and if in doubt about any aspect of the operation of the power supply please consult an instrument scientist or the ISIS MCR crew.

Locate Power Supply R67 T5/11. Turn on the *Main Isolator* by swinging the long-handled rotary switch on the front of the power supply (this is stiff to operate).

The Control Panel display will say "INITIALISE" for some time before the %Power Indicator appears as "SET 000000 ADC 00000"

Press the "REM" button so that the yellow "LOCAL / REMOTE" LED above it goes out. Select LOQ with the "POLARITY" button (check the lights under the *T-switch* on the shelf in the walkway between the LOQ and CRISP cabins).

Check to see if any of the four red LED's under "INTERLOCK STATUS", indicating trips, are lit. If "MAG", for magnet trip, is lit, pushing the appropriate button will cause the identities of the active trips to be displayed on the display.

Have someone press the white button on the *Local Control Box* by the magnet, so that the orange LED on the Local Control Box lights up. This will clear any trips. Then press the "OFF / RESET" button on the Control Panel. The amber LED by this button should not be lit now.

Press the "MAIN POWER" button firmly. There will be a noise as contactors operate.

Check again that no trip lights have appeared as it is not unknown for the contactors to "bounce" off setting an "MPS" trip light! If this happens, press the "OFF / RESET" button to clear the trip and try again.

Press the "COARSE" button next to the *Current Adjustment Knob* and then rotate the knob clockwise to increase the current to the magnet.

The left display on the Control Panel shows the requested power setting as 10000 times the % of the maximum (615 Amps), whilst the right display shows the actual power setting (ie; for 10% power, about 60 A, set the readout to 100000 and for 80% power, about 490 A, set the readout to 800000). A green "READY" LED shows when the requested setting has been achieved. Analogue meters on the front of the power supply indicate the actual current and voltage.

5. Clearing a Magnet Trip
If the magnet trips off during operation, press "OFF / RESET" button on the balcony Control Panel to clear the trips and then the "MAIN POWER" button. Ramp the power back up.

6. Switching the Magnet OFF
The magnet should switched off as follows in order to avoid damaging induction effects.

Rotate the *Current Adjustment Knob* anticlockwise until the requested power setting reduces to zero.

Press the "OFF / RESET" button to release the main power contactors.

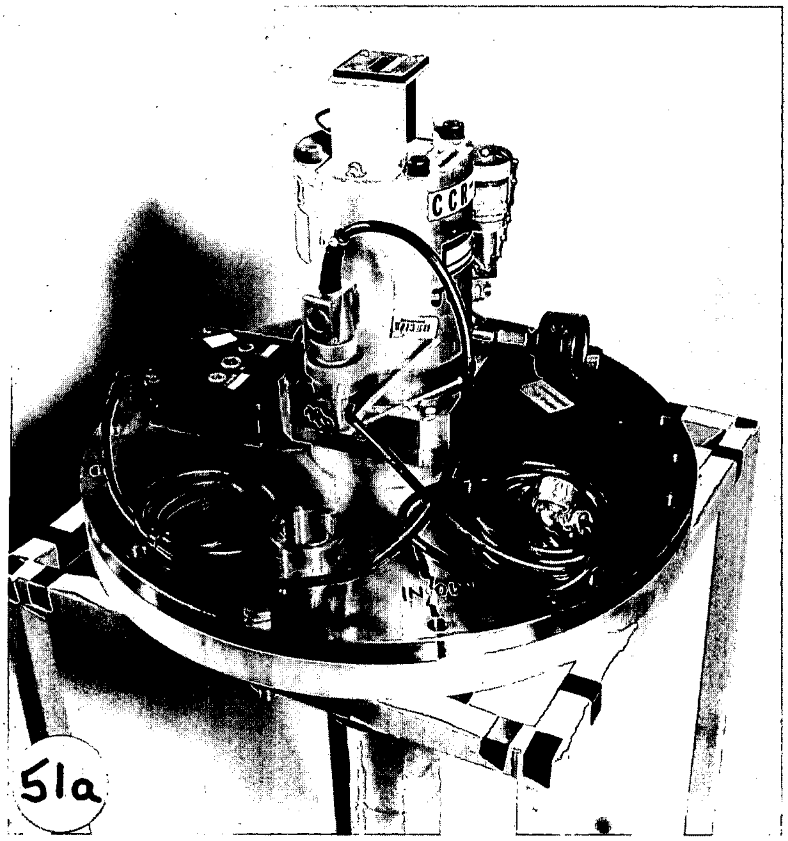
Turn off the *Main Isolator* rotary switch on the front of the power supply.

7. Computer Control of the Magnet
Please contact SMK for tuition before attempting computer control of the magnet.

(a) Ensure that the Amplicon 485Fi Interface is connected to terminal line TTA1: on the back of the LOQ FEM (see Section 2.23) and that its power supply is switched on.
(b) Ensure that the 4-way RS422 signal cable is connected to the Amplicon interface.
(c) Ensure that the *T-switch* on the shelf in the walkway between the LOQ and CRISP cabins is switched to LOQ.
(d) Follow the instructions in Part 4 above for turning the magnet on <u>but do not set a power level with the *Current Adjustment Knob*</u>.
(e) Press the "REM" button so that the yellow "LOCAL / REMOTE" LED is illuminated.

8. CAMAC Commands For Controlling The Goudsmit Magnet

CSET MAGNET/ON	turns the magnet on (equivalent to pushing the "MAIN POWER" button)
CSET MAGNET/OFF	turns the magnet off
CSET MAGNET xx.x	sets the power level to xx.x% (see graph on Page 3-34)
CSET MAGNET/LOG=n	starts logging of the magnet power level every n seconds
CSET MAGNET/NOLOG	stops logging of the magnet power level
CSHOW MAGNET	displays the magnet power level and controller status



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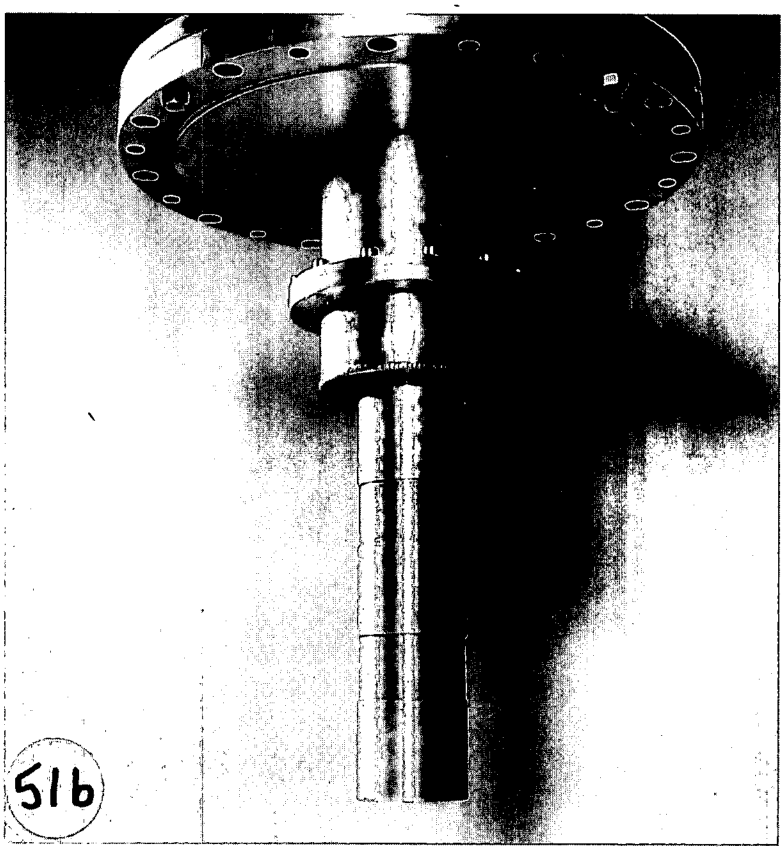


51b

Closed Cycle Refrigerators - I

For experiments which require a base temperature of not less than 10 K (-263°C), a number of Leybold helium *closed cycle refrigerators*, either Type RG1040 or RG1245, are available.

CCR's are entirely mechanical devices and so can be directly mounted into an instruments own, or suitable user-provided, evacuated sample chamber. Doing so eliminates the need for an additional cryogenic vacuum chamber and needs only a single aluminium thermal shield mounted on to the CCR's first stage (cooled to about 50 K). Normally on LOQ however, CCR's are operated with purpose-designed, thin-walled, vacuum "tails" as the sample position is in air. There is a special LOQ CCR, with a longer *cold finger* and corresponding vacuum tail, for use with the *Goudsmit Electromagnet* (see Section 3.50).

Special top-loading CCR's with ω-rotation stages or a 6° goniometer, and bottom-loading CCR's with gas condensation cells, are also available.

The cooldown time for a basic CCR unit is typically about 45 minutes.

CCR's can run up to ambient room temperatures, however to do so requires a compressor unit fitted with a *Bypass Valve*. If necessary, please state this requirement on the proposal form.

Also see the section on *Helium Cryostats.*

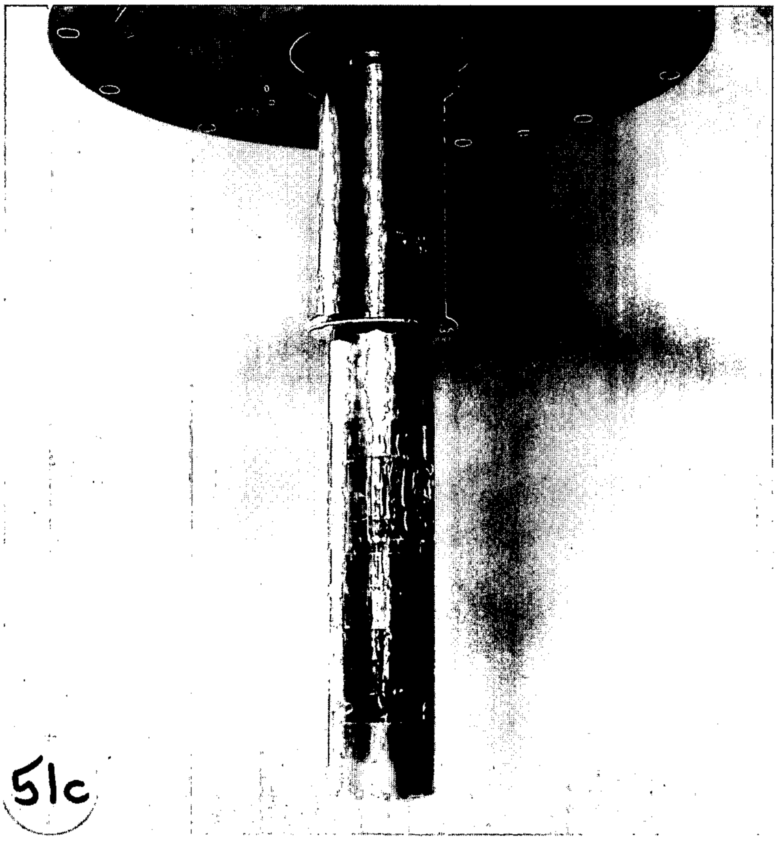
3.51(a) Closed Cycle Refrigerator Unit. *[Negative 94RB5933]*

The picture shows a typical CCR unit. The CCR head, in the centre, is mounted on a vacuum flange. Electrical connections between the head and the remote compressor unit (not shown) are made via the cable in the foreground. The helium lines from the compressor attach to two special fittings at the rear of the head, one of which is just visible. On the right is a mechanical temperature gauge. Moving clockwise around the vacuum flange we see the pumping port for evacuating the tail and the connection box for the two platinum resistance thermometers and cartridge heater.

3.51(b) 50mm Diameter Vacuum Tail. *[Negative 94RB5931]*

The picture shows the underside of the previous picture and, in particular, the CCR vacuum tail. The tail shown is specific to LOQ and allows the use of CCR's with two different lengths of *cold finger.* The aluminium has been machined away at the two different sample heights to reduce background scattering. The longer position is that used with the *Goudsmit Electromagnet.*

To change a sample it is either necessary to crane the CCR head (to which the *cold finger* is attached) up and out of the vacuum tail, or alternatively, to undo the Allen headed bolts shown and remove the lower $^2/_3$ of the tail. Whichever procedure is used, great care must be taken so as not to distort the delicate thermal shield inside (see Section 3.51(c)).



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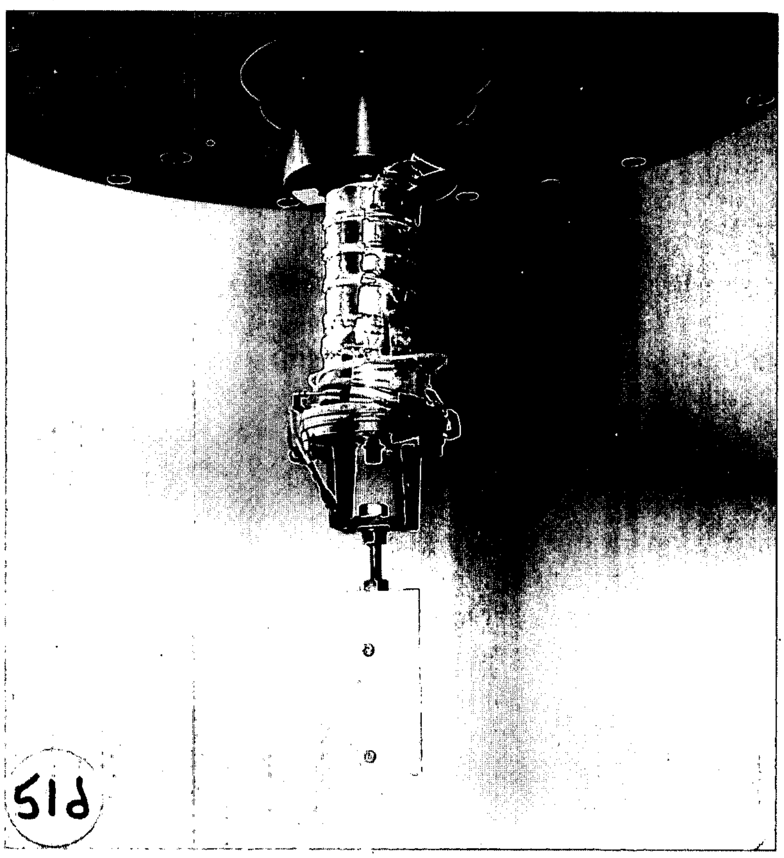


51d

Closed Cycle Refrigerators - II

3.51(c) Thermal Shield. *[Negative 94RB5930]*

The picture shows what is inside the vacuum tail. Without this thermal shield it is impossible to cool the CCR below about 70 K.

The shield is made from thin aluminium sheeting, but in the regions of the two sample positions holes have been cut in the sheeting and then covered with aluminium foil to reduce background scattering. For these reasons the thermal shield is very delicate.

<u>To prevent "heat leaks" it is important that no part of the thermal shield touches the inside wall of the vacuum tail. The clearance between shield and tail is only a few mm!</u>

3.51(d) Sample Mount. *[Negative 94RB5932]*

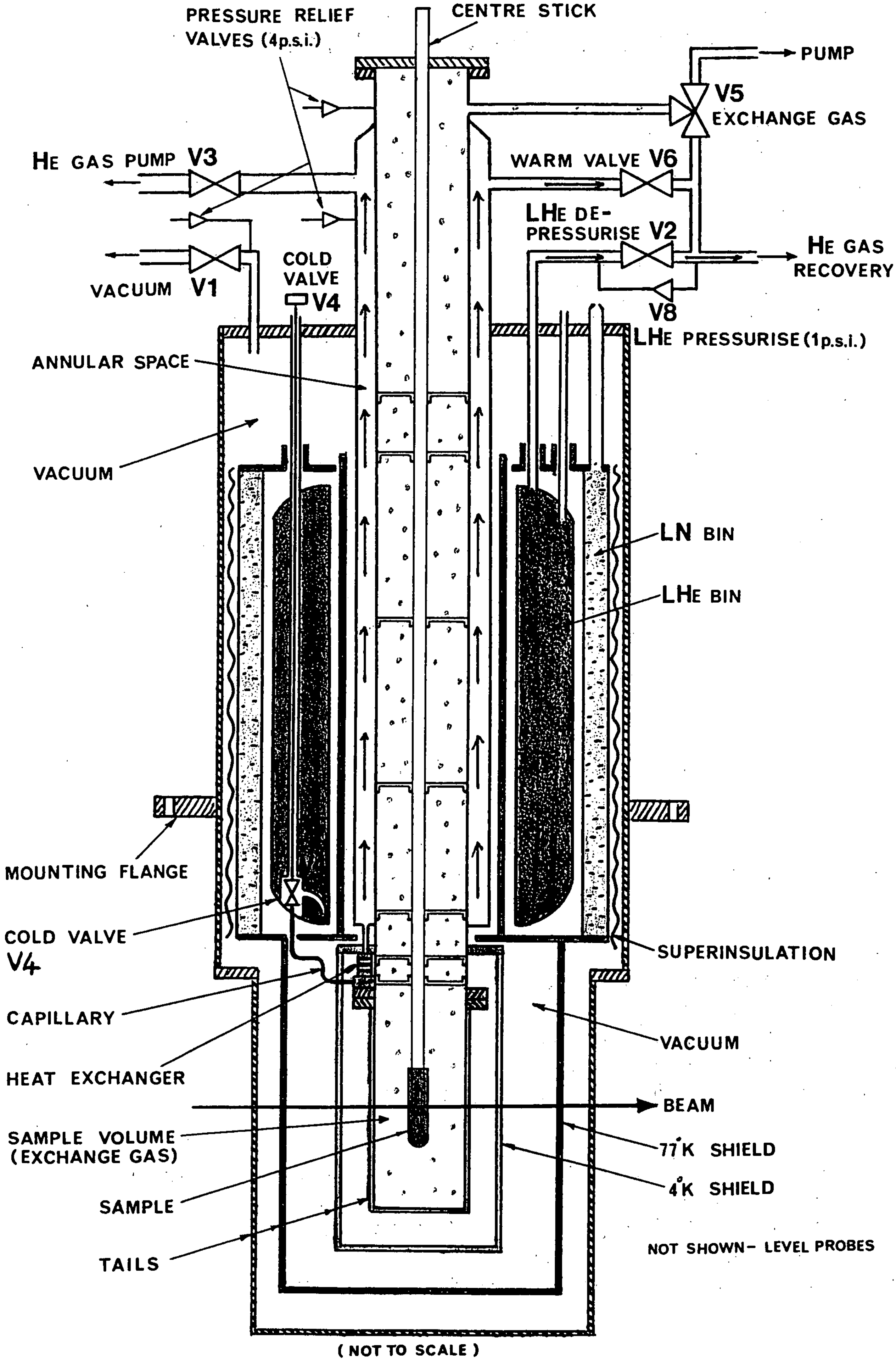
The picture shows what is inside the thermal shield. The *cold finger* protrudes from the bottom of the CCR head. Wrapped around it and held in place my alum inium tape are the wires from the platinum resistance thermometers and the cartridge heater. At the bottom of the cold finger is a brass U-bracket with a tapped 8mm diameter hole. Threaded brass rod is screwed into this hole and then into a user-provided sample holder.

CCR's versus Cryostat's

In principle, both *CCR's* and *Cryostats* (see overleaf) can be used to cool samples to temperatures between 10 K (-263ºC) and room temperature. Each has advantages and disadvantages.

CCR's
- present lower backgrounds than cryostats
- offer better near-ambient temperature control; say, between 220 K (-50ºC) and ambient
- are relatively straightforward to operate
- have long sample cool-down times
- require use of the LOQ pillar crane to change sample

Cryostat's
- can be used to quench-cool samples (though care must be taken to prevent the sample icing-up on removal)
- do not require use of the LOQ pillar crane to change sample
- present higher backgrounds than CCR's
- can be tricky to operate (eg, prone to blocking)



"ORANGE" CRYOSTAT

Helium Cryostats

For experiments which require a base temperature of <u>less than 10 K</u> (-263°C) but <u>not less than 1.2 K</u> (-272°C), a number of "orange" *helium cryostats* are available with bores of 50 mm and 100 mm. Operation of the cryostat at temperatures below 4 K requires a special (Rootes) vacuum pump set.

Normally on LOQ 50 mm bore cryostats are used in conjunction with special thin-walled "tails". A 50 mm bore cryostat (OC 50-03) <u>must</u> be used with the *Newport* and *Goudsmit Electromagnets* (see Sections 3.49 & 3.50).

A schematic diagram of an orange cryostat is shown opposite.

Charging the cryostat with helium
Depressurise the helium bin by opening spanner valve V2. When the helium flow ceases (check the flowmeter), remove the threaded bung on the helium fill pipe and insert the siphon hose from a helium dewar. Fill the helium bin. Close valve V2.

Working at temperatures below 4 K
Attach a Rootes pump to the cryostat pumping port and open valve V3.

Regulating the temperature of the cryostat
Ensure that the "cold valve" (V4) is open $^{1}/_{4}$ - $^{1}/_{2}$ of a turn. Control the helium flow rate with the "warm valve" (V6) with reference to the flowmeter on the helium recovery panel. Greater flow gives greater cooling.

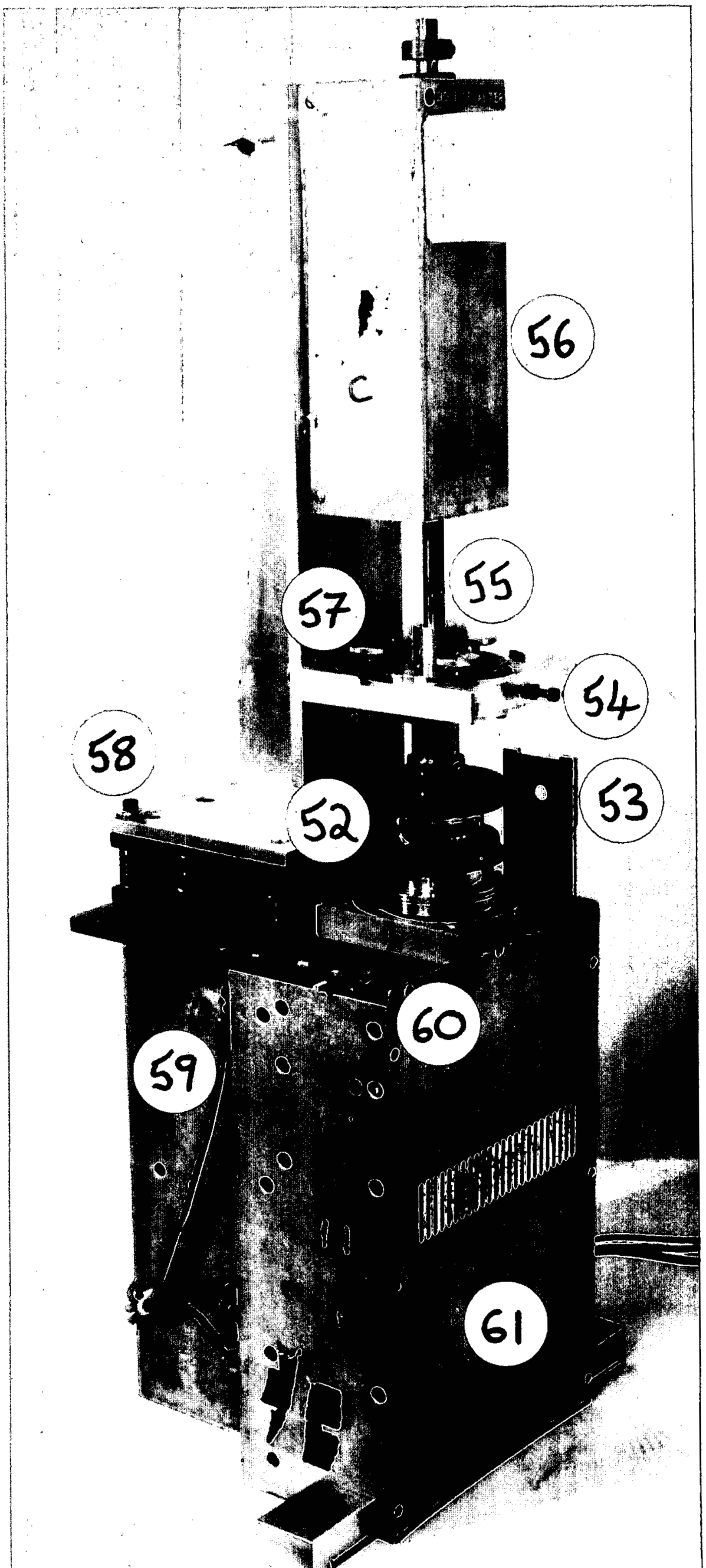
Changing samples
Create a helium overpressure in the centre stick volume by turning valve V5 until it points downwards. Disconnect any temperature sensor that might be attached. Remove the Allen headed bolts clamping the sensor flange to the cryostat body. Quickly, but carefully, lift the centre stick up and out of the cryostat making sure that you keep it vertical (stand on a chair if it helps!). Insert the new centre stick or cover the hole with a blanking plate. Replace the Allen headed bolts. Turn valve V5 until it points upwards. Turn on the small pump to exchange the contents of the centre stick volume. Depressurise the centre stick volume to 3 - 4 mbar (2 - 3 minutes). Close valve V5 by turning it horizontal.

<u>Note</u>:
Before inserting a new centre stick, ensure that it has been correctly adjusted. The distance from the centre of the sample to the underside of the centre stick top mounting flange will be given on the outside of the cryostat. The distance from the underside of the same flange to the last thermal collar on the centre stick above the sample is about 1.1 m.

Is the cryostat blocked?
If the flowmeter on the helium recovery panel does not indicate any flow then it is likely that the cryostat is blocked (with ice). Recovery procedures will take <u>several hours</u> so call out the experts and clear off!

Furnaces

There are two RAL furnaces, either of which can be used on LOQ. Both cover the temperature range 200 - 1400°C and can be provided with either vanadium or niobium elements and shields depending on the requirements of the experiment. Temperatures above 1200 °C require a furnace having niobium elements and shields. The maximum sample diameter is 35 mm.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Couette Shear Flow Cell

[Negative 89RC2460]

This is a shear flow cell of the Taylor-Couette design with a static, thermostatted, inner stator and a concentric, revolving, outer cylinder. The stator and outer cylinder are both made from the same type of synthetic quartz as standard sample cuvettes (see Pages 3-12 and 3-13), but this, and the method of assembly, limits the apparatus to an operational temperature range of 10 - 50°C. The cell is <u>not</u> fitted with a temperature sensor. The stator can be lifted up and down to allow access to, and removal of, the outer cylinder for cleaning and sample loading. The apparatus is fitted with a two-part lid to prevent sample spillage and to reduce evaporative losses. The total pathlength through the sample is 1 mm and the volume of sample required is ~7 cm^3. In some instances this can be reduced to ~4 cm^3.

The outer cylinder is driven by a d.c. motor with a feedback mechanism to maintain accurate speed control as sample-generated torque on the motor varies. The range of shear rate accessible with this apparatus is approximately 0 - 25000 s^{-1}, though the actual limits are obviously sample dependent. At the time of writing there is no provision for computer control of the shear rate. For further information please consult the technical paper on the following pages. The apparatus was designed and constructed by Unilever, but is available for use by other users.

3.52 Sample Cell
- shown assembled but with the lower portion of the lid (in black) released.

3.53 Cadmium Collimation Aperture
- optional, depending on the final collimation of the instrument (see Section 2.6)

3.54 Locking Screw
- used to hold the stator in place during operation.

<u>Note</u>:
- this screw must be tightened with the torque screwdriver provided before any attempt is made to spin the outer cylinder!

3.55 Thermostatting Fluid Hoses
- for connection to a circulating fluid bath (see Sections 3.41 and 3.47).
- terminated with quick release connectors. *Note: the connectors are smaller than those on the fluid baths and so adapters are necessary. These are provided.*

3.56 Stator Winding Handle (out of view)
- used to raise or lower the stator when the cell lid and locking screw (Section 3.54) are released.

3.57 Concentricity Adjustment Screws (3 off)
- fine adjustment screws used to position the stator in the centre of the outer cylinder.
- do not touch the screws unless you have been trained in how to perform the alignment!

3.58 Transit Screw (one of several)
- the apparatus is fitted with several transit screws and adjustment micrometers. Please do not interfere with them; the results of bad alignment could be dangerous!

3.59 Motor Power Supply & Control Cables

3.60 Mounting for Oscillatory Shear Option
- see Page 3-51.

3.61 Motor Housing

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A Couette shear flow cell for small-angle neutron scattering studies

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Abstract. Shear flow alignment is now an established technique for aligning anisotropically shaped molecules and colloids in small-angle neutron scattering studies. We present in this paper details of the construction of a Couette shear flow cell designed for such studies. Examples of small-angle neutron scattering studies using this cell are included.

1. Introduction

Following the pioneering work in optical flow birefringence on micellar systems [1], shear flow alignment of anisotropically shaped micelles is now receiving much attention in the context of small-angle neutron scattering [2–6] (SANS) studies.

The alignment of anisotropic micelles removes much of the insensitivity of scattering data from orientationally averaged systems. Additional and more detailed information on micellar geometry, size distribution and conformation is available than from the traditional rotationally averaged radius of gyration measurements. A number of groups have reported measurements on dilute rod-like micelles [3,7], mixed surfactant phases [8], shear-induced structures [9] and concentrated/viscoelastic systems [2]; and effects due to polydispersity, turbulence, hindered rotation and rod flexibility have been discussed [3].

Optical flow birefringence was extensively developed [1,10] to study anisotropic micelles, colloids and polymers. A suspended elongated particle subjected to a viscous shear gradient G will precess in the flow, the instantaneous angular velocity being a function of the orientation relative to the local streamlines. Simultaneously, Brownian motion, characterised by a rotational diffusion coefficient D_r, will tend to randomise the orientations. The relative importance of the two competing effects can be characterised by the parameter $\Gamma = G/D_r$; full alignment will occur only when $\Gamma \gg 1$. In practice, perfect alignment is never achieved, and to understand the scattering pattern from a partially aligned system, an estimate of the orientational distribution $p(\theta, \varphi; \Gamma)$ (where θ, φ are the normal polar angles, see figure 1) must be made. The recent neutron scattering studies [2,5] have derived distribution functions based on the original optical work [11].

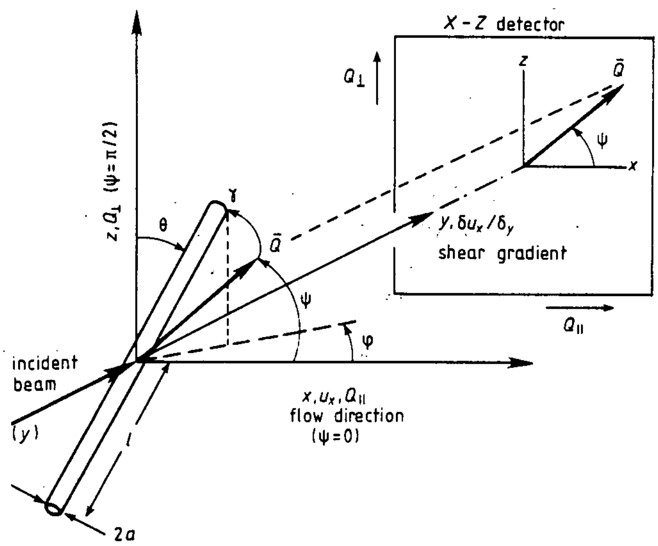

Figure 1. Scattering geometry and coordinate system for small-angle neutron scattering studies on shear aligned samples.

2. Couette shear cell

The typical unhindered rotational diffusion coefficients of the micellar/colloidal particles of interest to us are in the range 10^2–10^3 s^{-1}; hence shear gradients $>10^3$ s^{-1} are needed. Such shear gradients can be achieved in a relatively straightforward way by either Poiseuille or Couette flow (see figure 2).

Rotating concentric cylinders are commonly used to produce shear in optical experiments [1,10,11], the observation axis being parallel to the rotation axis. For neutron scattering much shorter path lengths, which can be obtained perpendicular to the rotation axis, are required to avoid multiple scattering and ensure adequate transmission. Alternatively, the sample can be contained

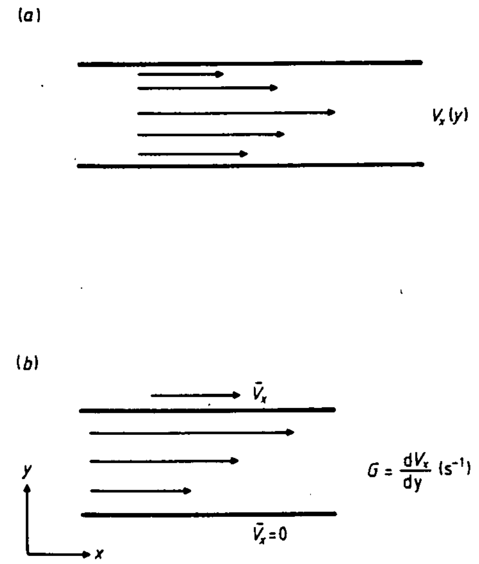

Figure 2. (*a*) Poiseuille and (*b*) Couette flow between two parallel plates.

between parallel plates and sheared by either a rotating disc or by flow between the plates. All these geometries have been used [5,12] to produce either Couette or Poiseuille flow. However, Couette flow is more preferable because if $d \ll r$ (where r is the Couette diameter and d the gap width), then a constant gradient across the gap is achieved, whereas Poiseuille flow will always have the characteristic parabolic form. A number of flow apparatus have been constructed for SANS measurements [12,13] and they predominantly use shear flow. The main requirements are as follows:

(i) transparent to neutrons;
(ii) no small-angle scattering from cell;
(iii) small sample volume and path length ($\leqslant 1$ mm);
(iv) shear gradient range 10^3–2×10^4 s^{-1};
(v) sealed system with no pumping action to prevent foaming in surfactant systems.

These main design requirements have been incorporated into the Couette shear cell designed by us, it is shown in figures 3 and 4. The outer glass rotor rotates whilst the inner stator is stationary. This geometry inhibits the production of Taylor vortices and establishes a constant shear gradient across the gap [11].

The main design features of Couette cells for neutron scattering studies have been discussed by Lindner *et al* [13]; we present here, in detail, only the aspects of design specific to this shear cell.

The Couette shear cell consists of an outer rotating cylinder and an inner fixed spigot, with a cylindrical gap of 0.5 mm; the main components are shown in figures 3

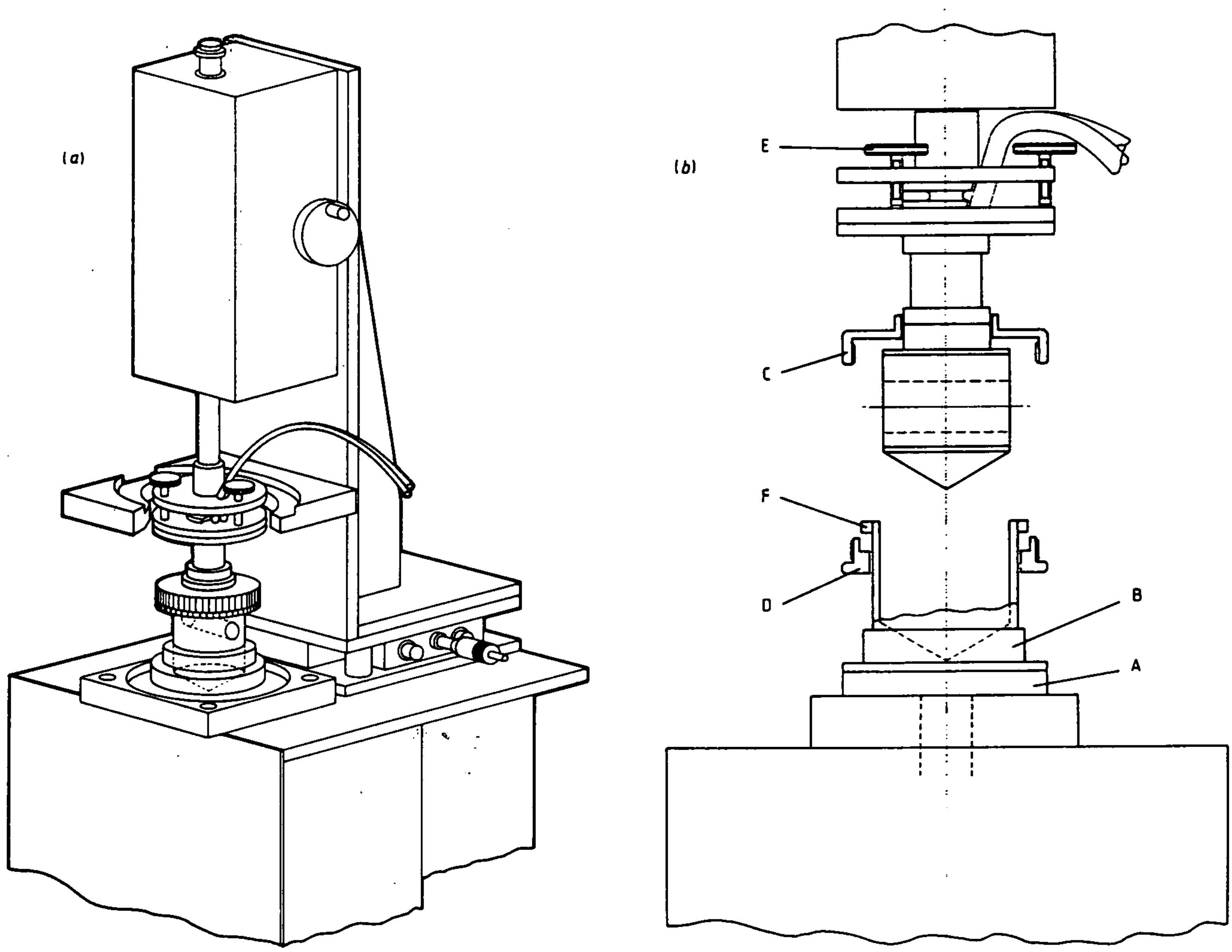

Figure 3. (*a*) Schematic view of shear cell assembly and (*b*) detail of stator and rotor.

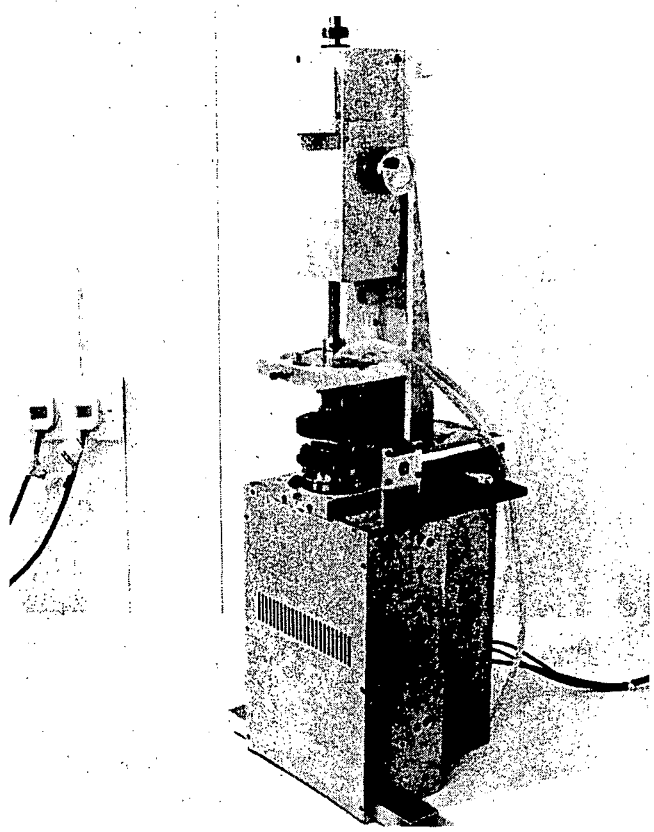

Figure 4. View of shear cell assembly.

and 4. The outer and inner cylinders are made of 3 mm suprasil quartz polished to $\pm 5\ \mu$m. The quartz central spigot is bonded with Araldite to Invar 36, which has the same coefficient of expansion as the quartz. The bonding of the separate components was performed after preparation of the quartz and care was taken to ensure that all components were concentric.

The conical profile of the spigot base reduces the possibility of air entrapment when samples are introduced and, in conjunction with the shaped PTFE base of the outer cylinder, results in a small total sample volume (7 ml). This geometry also produces a shear field within the base which is comparable to that generated within the cylindrical region (this reduces the possibility of osmotically induced mass transport processes). Water is circulated through the inner cell to provide temperature control, and an air-filled tube (figures 3 and 4) provides a low attenuation path for the neutron beam.

The outer cylinder is coaxially bonded (with Araldite) to an Invar 36 collar (B). This collar has a precisely machined locating ring that allows removal and precise concentric relocation onto the main shaft of the instrument. This main shaft axis provides the reference for the subsequent alignment of the inner cell, and no adjustment is provided as the precise location of the assembly is determined during construction.

Although a simple centripetal force may not be sufficient to directly eject liquid from the rotating outer cylinder, wetting of the cell surface with surfactants may cause a loss of material at low rotation speeds. To prevent this and to reduce solvent loss, a sealing mechanism is used. A silicon rubber ring (F) is cast/bonded to the top outside edge of the outer cell. Two threaded PVC collars (C and D) are arranged such that, whilst they can be readily separated for disassembly, very high local pressure (and hence a good seal) is achieved at the knife-edged contact point. The spigot passes through a simple clearance hole in C.

To ease cleaning and sample changing, the inner cell assembly is mounted on a rack and pinion. In its 'working' position the inner assembly is located and locked by a 'three-point mounting' yoke. The yoke and the rack and pinion can be translated using an x–y stage (NRC) that has been modified to allow direct locking in position. With this arrangement the inner cell can be withdrawn and replaced to within $\pm 2.5\ \mu$m. The central spigot axis is aligned parallel to that of the main shaft by adjustment of the three adjusting screws (E). Correct alignment in the working position is determined using two clock gauges mounted on the rotating section (A) in place of the cell outer. The main shaft of the instrument is vertically mounted in two ball races that are located in a rigid aluminium frame. The shaft is connected via a rubber coupling to a DC motor (Dunker Motor, Bonndorf, FRG) whose speed is controlled in the range 2–6000 RPM using its tachogenerator.

The shear gradient is related to the rotation speed of the rotor, and the gap width, in the approximate expression

$$G = \frac{2\pi N}{60}\frac{r}{d} \quad (\mathrm{s}^{-1}) \tag{1}$$

where N is the rotor speed in RPM, r is the mean cylinder radius in centimetres and d is the gap width in centimetres. For the cell described in this paper this gives

$$G = 5.28N \tag{2}$$

and hence G can range from approximately 10–$25\,000\ \mathrm{s}^{-1}$.

At low shear gradients the limitation will be the stability of the motor whereas the high gradient limit will be the onset of turbulence (we want to operate in a well defined laminar flow regime). The onset of turbulent flow is characterised by the Reynolds number Re, which for planar Couette flow is given by

$$Re = \frac{\rho d^2 G}{\eta} \tag{3}$$

where ρ is the density, η is the viscosity, d the gap width and G is the shear gradient. Turbulence occurs for Reynolds numbers in the range 1500–2000 and implies that, for a fluid with the viscosity of water, turbulence starts at shear gradients $\sim 8000\ \mathrm{s}^{-1}$.

3. Experimental applications

A wide range of different phases of anisotropically shaped micelles, aligned by Couette shear flow, have been investigated by small-angle scattering using the shear cell described in this paper.

Effects due to polydispersity, turbulence, hindered rotation and rod flexibility have been observed [3,4]. A number of dilute rod-like micelles have been characterised [3], details of the nature of clouding in non-ionic micelles have been obtained [7], and the effects of 'salting in' and 'salting out' electrolytes have been observed [14]. The nature of the mixed surfactant phases has been investigated [8], and preliminary measurements on concentrated and viscoelastic phases have been made [2].

We present in this section some recent experimental results for 3% hexaethylene glycol monohexadecyl ether $C_{16}E_6$ and 3% $C_{16}E_6$/0.3% cetyltrimethyl ammonium bromide C_{16}TAB in D_2O; these results are representative of the wide range of results obtained in recent years using the shear cell. The results have been obtained on the small-angle scattering diffractometers at the high flux reactor of the Institut Laue–Langevin. The 3% $C_{16}E_6$ data were measured on the D17 diffractometer in the Q range 0.08 to 0.4 nm^{-1}, whereas the 3% $C_{16}E_6$/0.3% C_{16}TAB data were measured on the D11 diffractometer in the Q range 0.1–0.6 nm^{-1}.

Figure 5(*a*) shows the intensity contour plot for a 3% solution of $C_{16}E_6$ in D_2O at a shear gradient of 5000 s^{-1} and in figure 5(*b*) we show the $Q_\perp$, $Q_\parallel$ intensity profiles (where ⊥, ‖ refer to the directions relative to the direction of orientation). These profiles are typical of the data obtained for long rod-like micelles in dilute solution; the data have been fitted for rods of $2l = 285$ nm and $2a = 6$ nm [7] (where l and a are the rod half-length and radius). As previously observed [3] some aspects of the contour plot indicate that the rods have a subtle degree of flexibility.

These data contrast sharply with the data presented in figures 6(*a*) and (*b*) for 3% $C_{16}E_6$/0.3% C_{16}TAB in D_2O at 40°C. The addition of the small amount of cationic surfactant has caused a dramatic change in the rheology of the solution, and correspondingly the scattering pattern is markedly different. The pattern is now dominated by the consequences of strong inter-micellar interactions which give rise to the pronounced maximum in the scattering at $Q = 0.26$ nm^{-1}; this is reminiscent of the scattering patterns observed for other interacting systems such as SDS/TDPS [2].

From the position of the correlation peak at $Q = 0.26$ nm^{-1} we can estimate a mean particle spacing $D = 2\pi/Q = 25.1$ nm. If we assume that the particles are arranged on a cubic lattice, and from the known surfactant molecular volumes and surfactant concentration, we can estimate that the scattering arises from rods/ellipses of

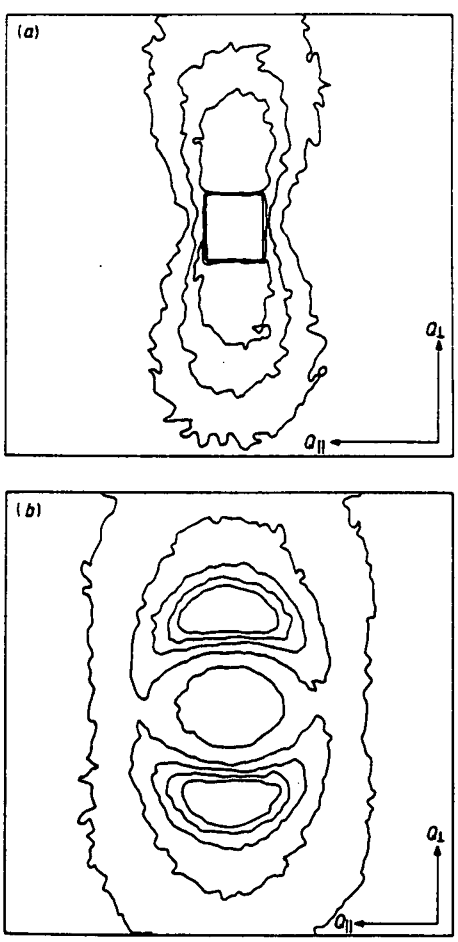

Figure 5. (*a*) Intensity contour plot for 3% $C_{16}E_6$ at 34°C, (*b*) intensity (in the $Q_\perp Q_\parallel$ directions) against momentum transfer Q for 3% $C_{16}E_6$ at 34°C.

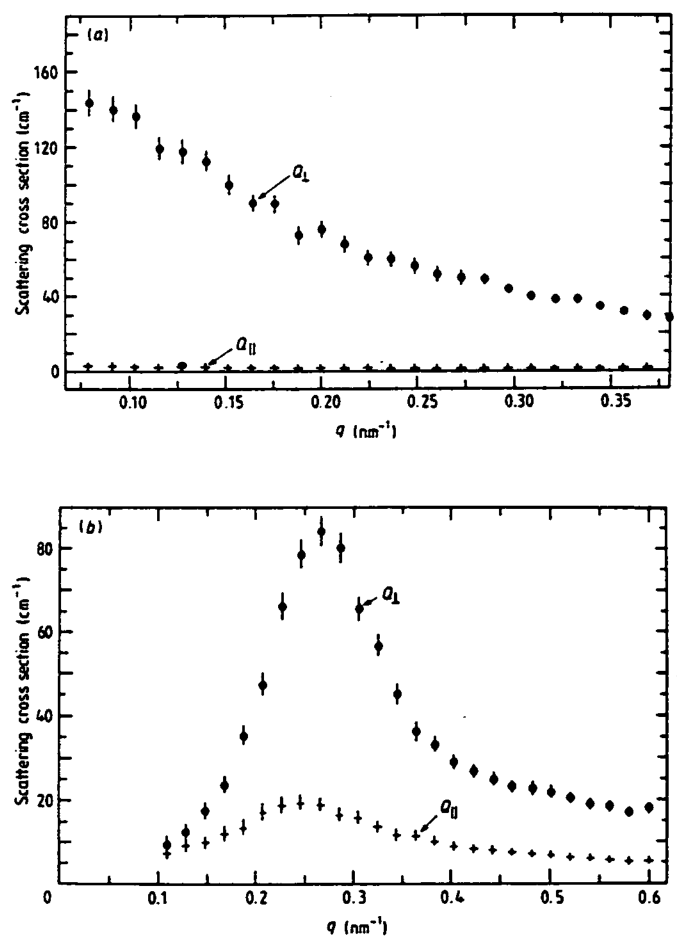

Figure 6. (*a*) Intensity contour plot for 3% $C_{16}E_6$ + 0.3% C_{16}TAB/D_2O at 40°C, (*b*) intensity (in the $Q_\perp Q_\parallel$ directions) against momentum transfer Q for 3% $C_{16}E_6$ and 3% C_{16}TAB/D_2O at 40°C.

$2l = 20$ nm (having constrained $2a = 6$ nm), and corresponding to an axial ratio of 3.3 : 1. It is evident from these simple observations that the addition of a small amount of cationic surfactant has dramatically changed the geometry of the micelle.

Elevation of temperature in the absence of shear results in very small changes in the rotationally averaged intensity profiles. However, under shear a dramatic change in the orientational coupling of the micelles can be observed.

4. Summary

We have described a Couette shear flow cell specifically designed for small-angle neutron scattering experiments on colloidal dispersions. Data from recent applications of the Couette shear flow cell have been presented and the range of applications has been discussed.

Acknowledgment

We wish to acknowledge contributions from J B Hayter during the prototype stages of the project.

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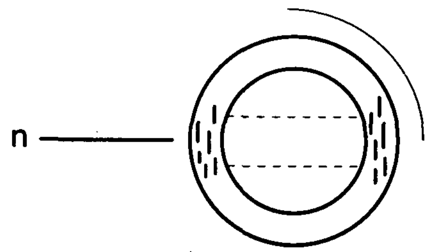
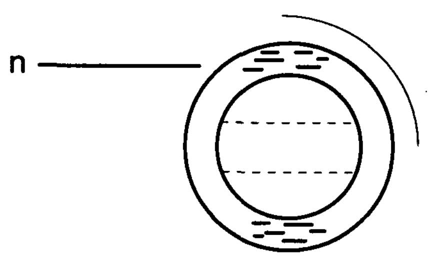
Modes of Operation

It is possible to conduct three types of experiment with the Couette Shear Flow Cell:

(a) Experiments where the neutron beam passes through the centre of the cell *(ie, perpendicular to the flow-vorticity plane)*



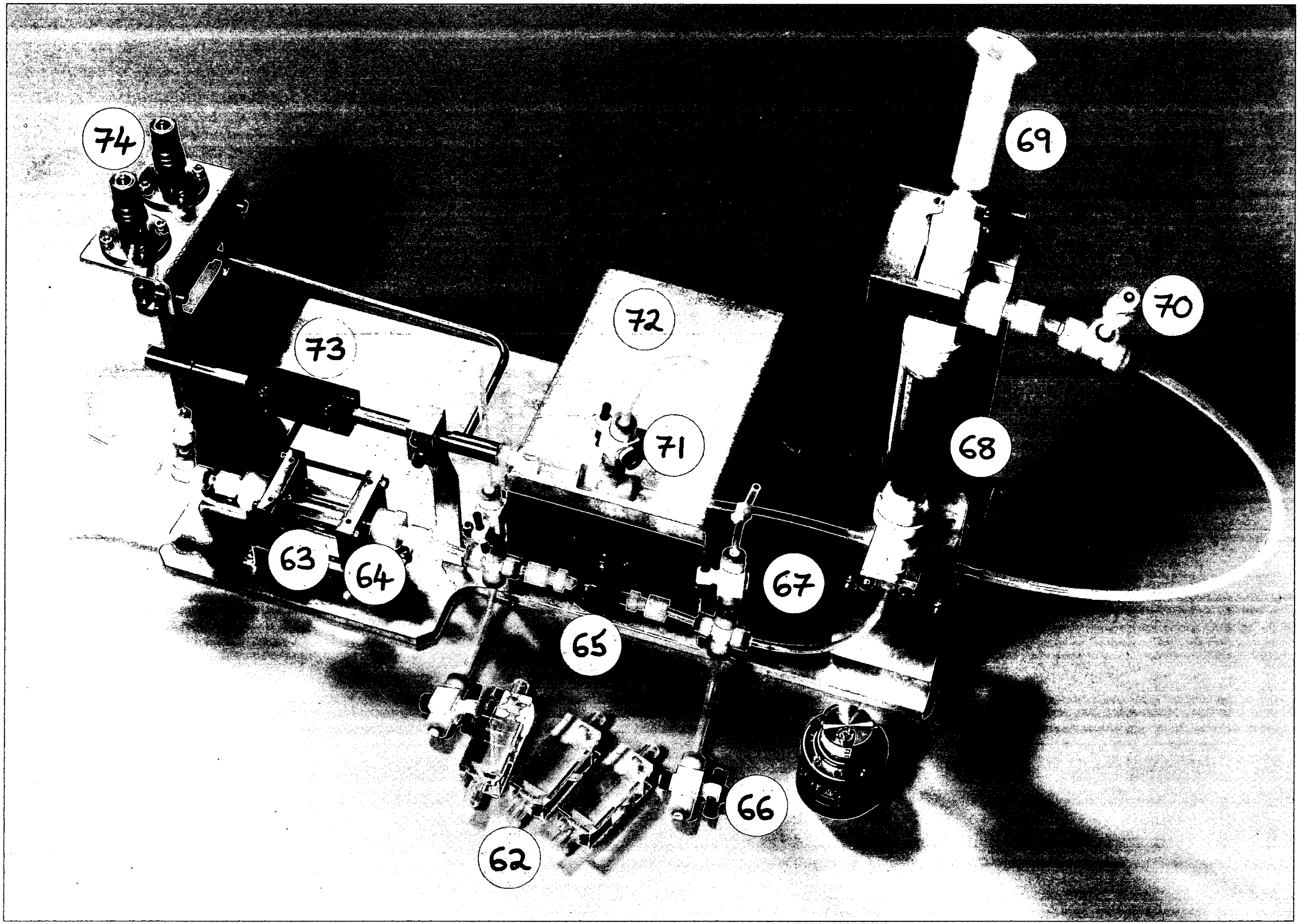
(b) Experiments where the neutron beam passes through one side of the cell *(ie, parallel to the flow-vorticity plane)*



(c) Experiments where the cell oscillates

The apparatus necessary for conducting oscillatory shear experiments was developed by a user group and is not resident at ISIS.

If you think that you have a suitable experiment for such measurements please contact SMK in the first instance.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Poiseuille Shear Flow Cell - I

[Negative 94RC6132]

This is a shear flow cell of a closed, recirculating, design in which the fluid is pumped through a narrow channel between two parallel plates in order to generate the conditions for Poiseuille shear flow. The actual flow cell is described overleaf. The pathlength through the sample is 1 mm and the volume of sample required is ~80 cm^3 with the flowmeter in place, or ~35 cm^3 with the flowmeter removed. A combination of a circulating fluid heat exchanger and electrical cartridge heaters give this apparatus an operational temperature range of 10 - 125°C. There are two K-type temperature sensors. The range of shear rate accessible with this apparatus is sample dependent but is approximately 100 - 5000 s^{-1}. Zero-shear measurements are possible at room temperature. At the time of writing there is no provision for computer control of the shear rate. For further information please consult the technical paper starting on Page 3-56. The apparatus was designed and built at ISIS in conjunction with Prof Julia Higgins and her research group at Imperial College, London. The apparatus is available for use by other users.

3.62 Flow Cells (removed)
- also see Pages 3-54 and 3-55.

3.63 Flow Cell

3.64 Flow Cell Compression Mount
- note the 4 holes for cartridge heaters.

3.65 Gear Pump

3.66 Drain Cock (1 of 2)

3.67 Filling Port (1 of 2)

3.68 Flowmeter
- is of the "ball-in-tube" type.
- two flowmeters are available, for low and high flow rates. Either flowmeter may be used with aqueous or non-aqueous fluids.

3.69 Fluid Reservoir
- also serves as an expansion volume at high temperatures.

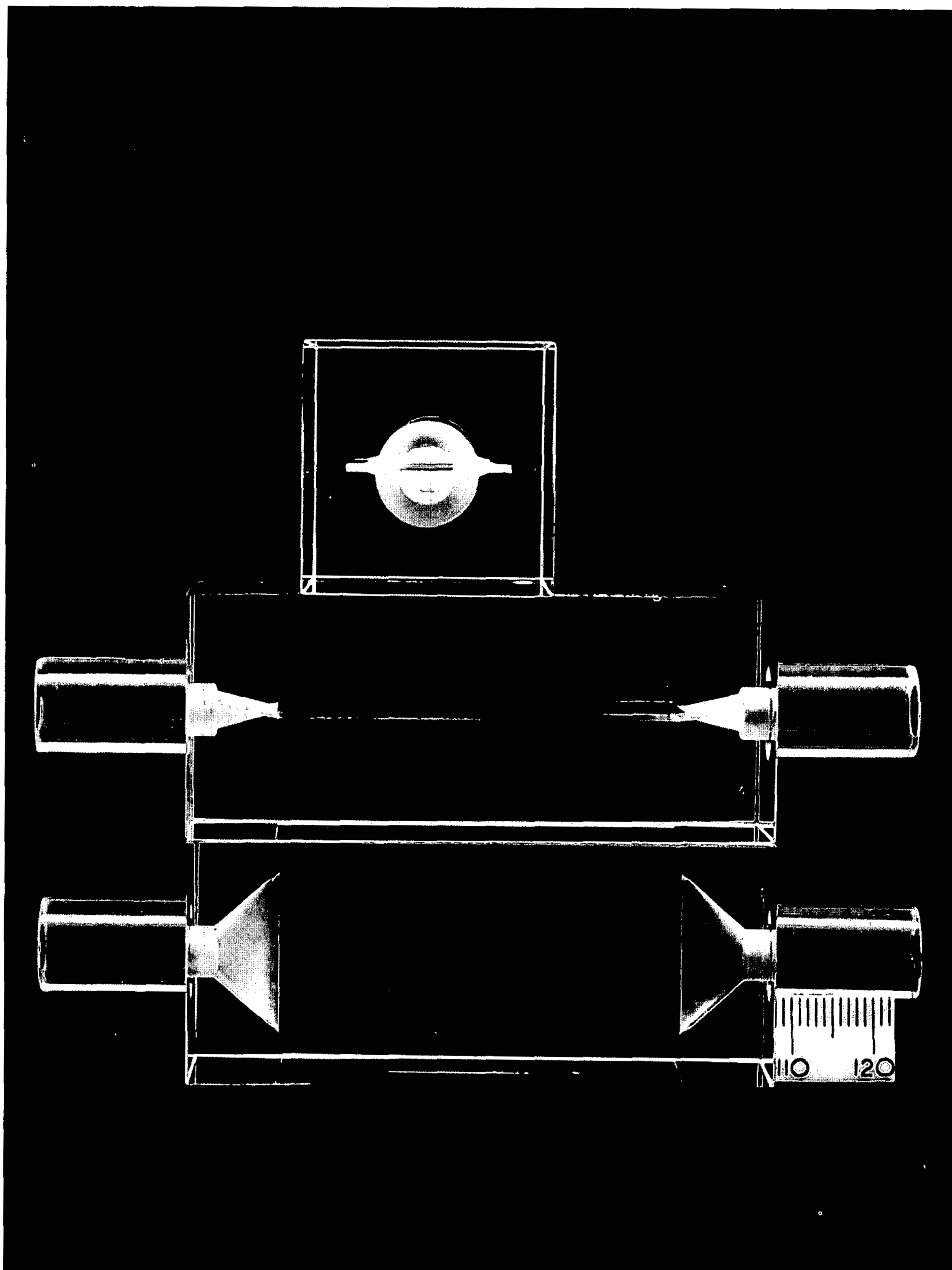
3.70 Thermocouple Mount (1 of 2)
- takes a K-type thermocouple which may be connected to the *Eurotherm Temperature Controller* (see Section 2.16 and Pages 3-3 to 3-6).

3.71 Vent Port

3.72 Pump Motor Splashguard

3.73 Cartridge Heater Mount
- takes an additional 4 cartridge heaters.

3.74 Circulating Fluid Bath Connections
- quick release couplings.
- also see Sections 2.11, 3.41 and 3.47.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Poiseuille Shear Flow Cell - II

[Negative 94FC4821]

The figure opposite shows three different views of the flow cell that forms the heart of the Poiseuille shear flow apparatus.

The principal feature of the cell is a narrow channel, 1 mm wide, between two smooth, parallel, walls. The channel 50 mm long and 20 mm high.

The cell is fashioned from the same type of synthetic quartz as standard sample cuvettes (see Pages 3-12 and 3-13). This combines low neutron absorbtion with good chemical resistance and thermal properties. With the apparatus in its normal configuration the neutron beam has the same view of the cell as that shown in the lower of the three views. If necessary, it is possible to rotate the cell through 90° (ie, as in the middle view), though the sample pathlength then becomes 20 mm.

A Poiseuille geometry shear flow apparatus for small angle scattering experiments

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A new apparatus to investigate the structure of fluids under Poiseuille shear flow using small-angle neutron or light scattering techniques is described. Important features of the design include low sample volumes, area average shear rates of up to 5000 s^{-1}, accurate temperature control over the range of 10 - 125 °C and the ability to study the development of flow-induced alignment phenomena. SANS data are presented for two example systems; the well-characterised surfactant system $C_{16}E_6/D_2O$, used to contrast the effects of Poiseuille and Couette flow, and the block copolymer Synperonic P-85/D_2O system.

I. Introduction

A. Background

The long-range correlations and degree of molecular order in a flowing fluid can be rather different from those present in a fluid at rest. This simple fact has enormous significance in the industrial arena where the behaviour and properties of a fluid under flow, its rheology, can affect or even dictate the conditions of transportation or processing of a product.

The combination of shear flow and SANS or SAXS has been widely used for the study of colloidal systems such as dispersions of cylindrical or rod-like surfactant micelles.[1] This is because by aligning the micelles in a shear field the orientational averaging effect normally conferred on the static scattering pattern by, amongst other things, the Brownian motion of the micelles, is removed. Analysis of the resulting anisotropic scattering pattern can then provide estimates of the micellar contour length, cross-sectional radius, flexibility (persistence length) and polydispersity, and also allows studies of hindered rotation. Other workers have investigated the behaviour of concentrated dispersions of polymer latex particles[2], clays[3] and polymer solutions[4] under shear. There has also been growing interest in shear-induced structural changes in systems such as liquid crystalline surfactant phases and ordered diblock copolymer melts.[5] Very recently, shear flow has been combined with the technique of Neutron Reflectometry, enabling thin flowing films and boundary layer effects to be investigated, and with an X-ray Surface Forces apparatus.[6]

Although many workers have utilised shear flow apparatus of the Couette and Searle (concentric cylinder[7]) or twin disc[8] design, far fewer workers have utilised Poiseuille shear flow.[2,5,9] In the main this seems to be due to the larger sample volume necessary, though there are other practical and theoretical considerations which space prevents us from discussing here. Such apparatus does, however, have applications in the study of industrial systems.[10]

B. Poiseuille Flow

Poiseuille flow is the laminar flow (Re < 2000) of a fluid in a pipe under a constant pressure gradient. Irrespective of the fluid type, in Poiseuille flow the velocity at the pipe wall is essentially zero, increasing to a maximum at the centre of the pipe. The shear rates in the pipe mirror this. If Newtonian, the fluid adopts a parabolic velocity profile. Geometrically similar profiles are generated when a fluid passes between two static parallel plates and this modified geometry lends itself more readily to experimental investigations.

To encompass the range of shear rates present between the plates, the area-average shear rate[11]

$$\dot{\gamma}_{AV} = \frac{2Q}{wh^2}\frac{(m+2)}{(m+1)} \tag{1}$$

has been used throughout this paper, where Q is the volumetric flow rate, w is the width of the plates, h is the separation of the plates and m is the dimensionless shear rate exponent in the power law equation

$$\mu = \mu_0 \dot{\gamma}^{(\frac{1}{m}-1)} \tag{2}$$

relating the viscosity, μ, and consistency, μ_0, of the polymer. For Newtonian fluids $m = 1$. For non-Newtonian fluids m must be determined from rheological measurements.

For a fluid flowing between two parallel plates, the distance after which laminar flow is fully developed, the entry length L_e, is given by[12]

$$\frac{2L_e}{h} = \frac{c\rho Q}{w\mu} = c\,\mathrm{Re} \tag{3}$$

where c is a dimensionless experimental constant[12,13] of the order 0.03 - 0.1 and ρ is the density of the fluid.

Equation 3 highlights an advantage of Poiseuille shear flow, namely that it permits the study of the evolution of flow-induced phenomena in a complementary, and arguably more practical (from the perspective of a scattering measurement), manner than is possible with Couette shear flow. In Poiseuille apparatus of the parallel plate geometry, the fluid is subject to a complex flow as it expands from the confines of the inlet pipe. This serves to scramble or randomise any structure in the fluid. However further along the channel the flow becomes better defined and alignment more pronounced. This behaviour is illustrated in Section III.B.

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II. Description of Shear Flow Apparatus

A schematic of the Poiseuille flow cell apparatus is shown in figure 1. The apparatus is a closed recirculating system comprising a quartz flow cell (1) (Optiglass Ltd.), an electrically-driven, magnetically-coupled, three-gear pump (2) (Micropump® Series D, CP Instrument Company Ltd.) for pulseless flow, and a glass in-line flowmeter (3) (FL-214 Rotameter, Omega Engineering Ltd.). These are all interconnected by PTFE fittings and lagged ¼ inch (6.35 mm) OD PTFE tubing. PTFE has been used for its chemical inertness and poor thermal conductivity. The pump gears are made from Ryton® (polyphenylene sulphide), an equally inert material, and are enclosed in a stainless steel housing. The layout of the components has been carefully chosen so that wherever possible the sample follows gentle curves as it circulates; this is designed to prevent complex or even turbulent flow occurring at 90° bends.

The fluid reservoir (4) allows air to be vented from the solution. The cell can be loaded via any one of six vents (10) using a syringe. The vents have been positioned in such a way to allow easy venting of the system; it being important to minimise the amount of air trapped in the apparatus. It is relatively easy to dismantle the apparatus for cleaning.

Temperature control is facilitated by three short sections of stainless steel tubing in the circuit. One of these passes through a heat exchanger (5) connected to a thermostatted circulating fluid bath, another is wrapped in heating tape (6) and the third is surrounded by four cartridge heaters in a brass block (7). These sections of tubing have been placed so that the solution is heated as evenly as possible as it circulates. Another four cartridge heaters are mounted in stainless steel blocks at either end of the quartz cell (8). These blocks also serve to act as a compression fitting to join the tubing to the quartz cell. For temperature measurement the apparatus is fitted with two K-type thermocouples (9). The position of the thermocouples in a small T-section may affect the flow pattern through the tubing, but the effect is not significant enough to disturb the region of interest in the quartz cell. One thermocouple is positioned before the entrance to the quartz cell and the other approximately half-way around the circuit. The measured temperature difference across the circuit is generally no more than ±2 °C; and can be improved to ±1 °C at higher flow rates. The operational temperature range of this apparatus is determined by the efficiency of the heat exchanger for temperatures below ambient and the specification of the PTFE fittings at higher temperatures. We have achieved a range of 10 to 125 °C. However, since circulation is important to achieving the desired temperature and preventing the formation of hot and cold spots, zero-shear measurements are only realistic at room temperature.

Inside the quartz flow cell, detailed in figure 2, the fluid passes through a rectangular channel 1 mm in pathlength and 20 × 50 mm in elevation. At the two ends of the channel the quartz has been carefully machined so that it tapers to the internal diameter of the tubing ($^5/_{32}$ inch, 3.96 mm) in such a way that back eddies or vortices, which would otherwise form at sharp corners, are essentially eliminated. This was checked visually with a coloured solution. The quartz cell has been designed so that, if required, it can be rotated 90° about the flow direction.

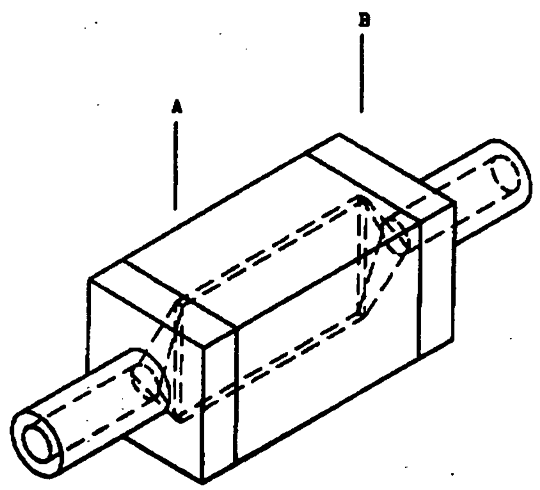


Figure 2. Schematic diagram of the quartz flow cell. Item (1) in Figure 1. The labels are explained in Section III.B.

For the shear rate determination it is important to know Q accurately, particularly if the viscosity of the sample changes with temperature. The volume of the apparatus is approximately 80 cm^3 with the flowmeter connected into the circuit and approximately 35 cm^3 without it. The pump is rated to 3000 rpm which is sufficient for a flow rate of approximately 30 cm^3s^{-1} (1.8 $l min^{-1}$) to be attained if the apparatus is filled with water. The range of area average shear rate that is accessible with this apparatus is therefore *circa* 100 to 5000 s^{-1}.

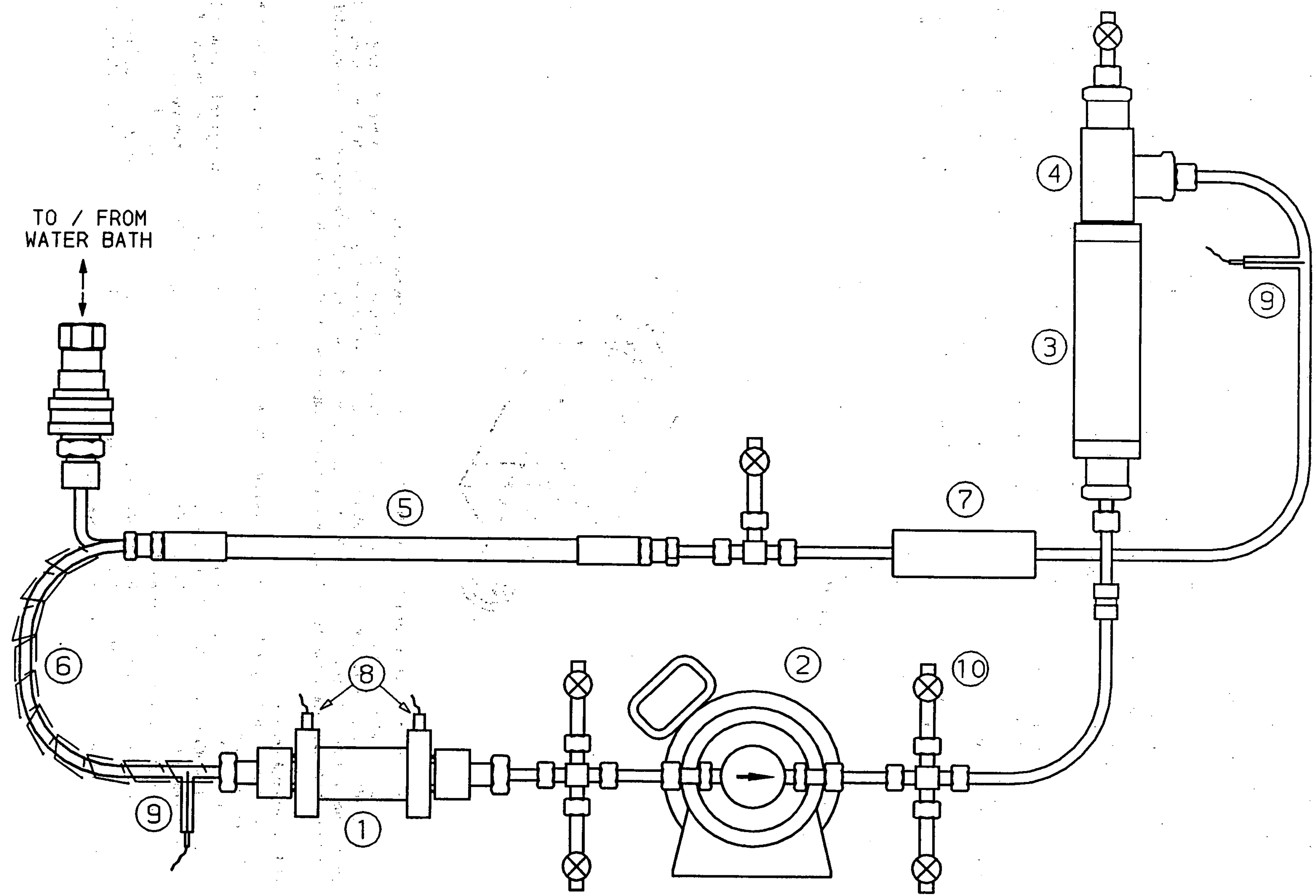
III. Experimental Results

The surfactant hexaethylene glycol mono-hexadecyl ether (also called 6 cetyl ether or $C_{16}E_6$) was obtained from Sigma Chemicals, U.K., and was used as supplied.

A sample of the low-molecular weight poly(oxyethylene)-poly(oxypropylene)-poly(oxyethylene) triblock copolymer Synperonic P-85 was obtained from ICI Surfactants, Middlesbrough, U.K. Since the commercial material has been reported to contain small amounts of the diblock and other impurities it was purified before use.[14] The polydispersity (M_w/M_n) of the copolymer after purification was ≤ 1.3.

Deuterium oxide, isotopic purity 99.9+ atom% D, was obtained from Fluorochem Ltd., U.K., and was used as supplied.

SANS measurements were performed on the LOQ diffractometer[15] at the ISIS Spallation Neutron Source, Oxfordshire, U.K.[16] This is a fixed-geometry, time-of-flight, instrument equipped with a 64 × 64 cm position-sensitive detector. Neutrons with wavelengths between 0.2 and 1.0 nm are combined to provide a simultaneous q-range of approximately 0.06 to 2.2 nm^{-1} where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$$q = \left(\frac{4\pi}{\lambda}\right)\sin\left(\frac{\theta}{2}\right) \tag{4}$$

is the modulus of the scattering vector, the resultant between the incident and scattered wavevectors, whilst λ is the neutron wavelength and θ is the scattering angle. The effective wavelength resolution was $\Delta\lambda/\lambda \approx 5$ %. The incident neutron beam was collimated to 10 mm diameter at the sample.

All scattering data were normalised for the sample transmission and incident wavelength distribution, background corrected using a flow cell filled with D_2O (this also removes the inherent instrumental background from vacuum windows, etc) and corrected for the linearity and efficiency of the detector response using the instrument-specific software package.[17] The data were converted to absolute units by scaling them to the scattering from a well-characterised partially-deuterated polystyrene blend calibration sample.[18]

A. Comparison of Poiseuille and Couette flow

For this comparison we present SANS data from a 1.1 $\%^w/_w$ solution of $C_{16}E_6$ in D_2O at 30 °C. This is a well characterised micellar system.[19] In a zero shear environment the surfactant self-assembles into cylindrical micelles approximately 6 nm in radius and some 300 nm in length, but when subjected to a shear flow field the long axis of the micelles have been shown to align about the flow direction. This generates an anisotropic scattering pattern which is perpendicular to the anisotropy of the particles.

SANS data were collected from this system in both the Poiseuille flow apparatus and in the Couette flow apparatus of Cummins et al.[7] In the Couette apparatus a uniform shear rate of 5000 s^{-1} was maintained, whilst in the Poiseuille apparatus the measurements were made at an area average shear rate of 5000 s^{-1}.

Figure 3. Corrected intensity contour plots comparing the effect of two different types of shear flow on the same system. (a) Poiseuille shear flow (anisotropy ratio 2.3), (b) Couette shear flow (anisotropy ratio 7.7). 1.1% w/w $C_{16}E_6$ in D_2O at a shear rate of 5000 s^{-1} and temperature of 30 °C. The horizontal and vertical axes correspond to the components of the scattering vector, q, parallel and perpendicular to the direction of flow respectively. Both axes cover the range $-0.8 \le q$ (nm^{-1}) $\le +0.8$ and the contour levels are the same in each plot.

As can be seen from the corrected intensity contour plots in figures 3(a) and 3(b) both types of flow induce anisotropy in the scattering pattern. The scattering from the micelles under Poiseuille flow is however markedly less anisotropic than that generated by Couette flow. Calculation of the anisotropy ratios (the ratio of the scattering perpendicular and parallel to the flow direction averaged over a range of q) reveals that for this system the alignment achieved under Couette flow is approximately three times greater than that under Poiseuille flow. This is because in Poiseuille flow the rod-like micelles are simultaneously subjected to a range of shear rates. This is not the case in Couette flow. Consequently towards the centre of the Poiseuille flow cell, where the shear rate tends to zero, the conditions for alignment are not as favourable. Micelles in this central region of the cell are therefore subjected to a lower average shear rate than those at the cell walls. The resulting scattering pattern is thus a convolution of signals from micelles in differing degrees of alignment.

B. Evolution of alignment

For these measurements a 1.0 $\%^w/_w$ solution of Synperonic P-85 in D_2O was used. Above the critical micellisation temperature, about 30 °C, the block copolymer molecules aggregate into roughly spherical micelles. Above approximately 63 °C these elongate into rod-like micelles[20]. Like the $C_{16}E_6$ micelles discussed above, these rod-like copolymer micelles also align in a shear field.

For this system the entrance length, L_e (see equation 3) is calculated to be 25 mm; that is, 25 mm downstream of position A in figure 2 the system is in fully developed laminar flow. Thus, by moving the position of the centre of the neutron beam between A and B it is possible to study the developing anisotropy in the scattering (developing alignment).

Figure 4. Corrected intensity contour plots comparing the effect of Poiseuille shear flow at two different positions along the flow cell on the same system. (a) 10 mm from entrance (anisotropy ratio 1.6), (b) 35 mm from entrance (anisotropy ratio 2.7). 1.0% w/w Synperonic P-85 in D_2O at a temperature of 76 °C. The horizontal and vertical axes correspond to the components of the scattering vector, *q*, parallel and perpendicular to the direction of flow respectively. Both axes cover the range $-0.6 \le q\ (nm^{-1}) \le +0.6$ and the contour levels are the same in each plot.

Measurements were made with the neutron beam initially 10 mm downstream from A, within the region of developing flow, and in the fully developed region 35 mm downstream. Evidence of the developing alignment of the micelles comes from the distinct change in anisotropy ratio; from 1.6 in the developing region to 2.7 when the flow is fully developed (figures 4(a) and 4(b)). This is despite the fact that the relatively large diameter of the incident beam must result in a significant degree of orientational averaging (particularly in the developing region) and the effects of polydispersity in molecular weight (which result in a broader distribution of rod lengths than in the $C_{16}E_6$ system above). We propose to investigate these effects in more detail in a future experiment using narrow slit collimation. This type of study is difficult to accomplish in a Couette shear cell except when it is operated in an oscillatory mode, however this can in itself introduce other experimental difficulties.

C. Effect of shear and temperature on P-85 in solution

The Synperonic family of copolymers is used in many different industrial formulations, and so their behaviour under likely processing conditions is of more than just academic interest. Another reason for our interest in the P-85/D_2O system is that the transition from spherical to rod-like micelles was only discovered comparatively recently[21] and all (light, X-ray and neutron) scattering data on this system at low concentrations has, to date, been obtained in the absence of shear. Undoubtedly one of the reasons for this is that much of the available Couette and Searle shear flow apparatus for use with X-rays and neutrons is limited to a maximum temperature of around 50 °C by the materials or methods of construction.

With this in mind we have conducted a detailed SANS study of this system between the sphere-to-rod transition temperature and the cloud point (~82 °C). Our preliminary findings will be reported in detail elsewhere[20] but we show here some representative data that illustrate how the system responds to changes in temperature and shear.

Figure 5(a) shows the SANS from a 1 % w/w solution of P-85 in D_2O in the Poiseuille shear flow apparatus at a constant average shear rate of 600 s^{-1} and at a temperature of 61 °C; that is, just below the sphere-to-rod transition temperature. The scattering is comparatively weak and isotropic, showing that there is no alignment of the micelles. At this temperature the micelles are, broadly speaking, spherical. In Figure 5(b) the temperature has been raised to 74 °C; approximately 10 °C above the transition temperature. The scattering pattern is now elongated in the direction perpendicular to the fluid flow as would be expected from micelles aligning with the flow direction. Initial analysis of the data indicates that at this temperature the micelles have a cross-sectional radius of about 5 nm and a length of 60 - 70 nm.

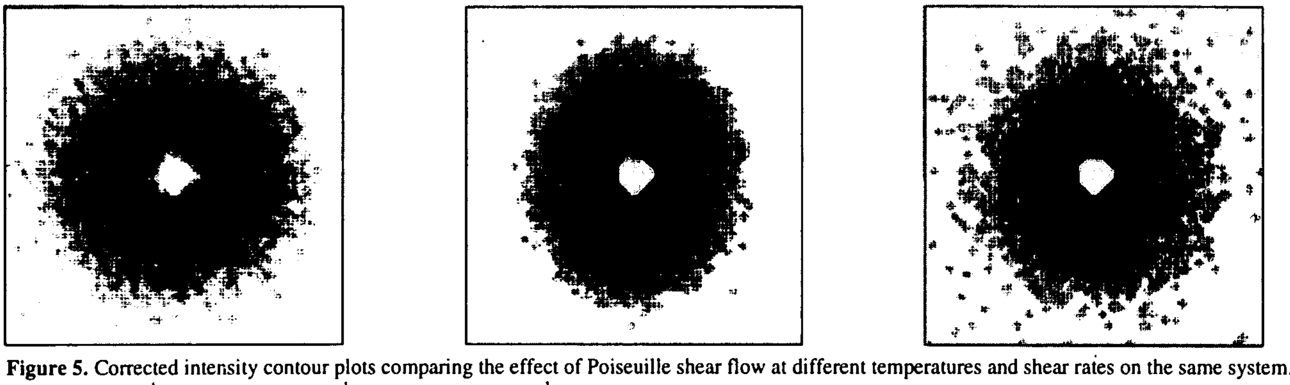

Figure 5. Corrected intensity contour plots comparing the effect of Poiseuille shear flow at different temperatures and shear rates on the same system. (a) 61 °C and 600 s^{-1}, (b) 74 °C and 600 s^{-1}, (c) 75 °C and 1550 s^{-1}. 1.0% w/w Synperonic P-85 in D_2O. The horizontal and vertical axes correspond to the components of the scattering vector, *q*, parallel and perpendicular to the direction of flow respectively. Both axes cover the range $-0.6 \le q\ (nm^{-1}) \le +0.6$ and the contour levels are the same in each plot.

Figure 5(c) shows the scattering at 75 °C and 1550 s^{-1} (which is still within the laminar flow regime). By 2500 s^{-1} the scattering pattern is almost as isotropic as it is under zero-shear conditions, although at this shear rate the Reynolds number is between the upper limit for laminar flow and the lower limit for turbulent flow.

Acknowledgements

The authors would like to thank the Engineering and Physical Sciences Research Council and ISIS for providing the materials and neutron beam time which have made this work possible. We would also like to express our gratitude to Richard Heenan, Jeff Penfold and Ed Staples for their support and encouragement, and to all those members of the ISIS Project Engineering, Sample Environment and Operations Groups who have assisted us in one way or another over the years. VMC would like to thank Shell Research for the award of a postgraduate studentship. We acknowledge ICI Surfactants for donating the Synperonic P-85 and thank Clive Washington for purifying it.

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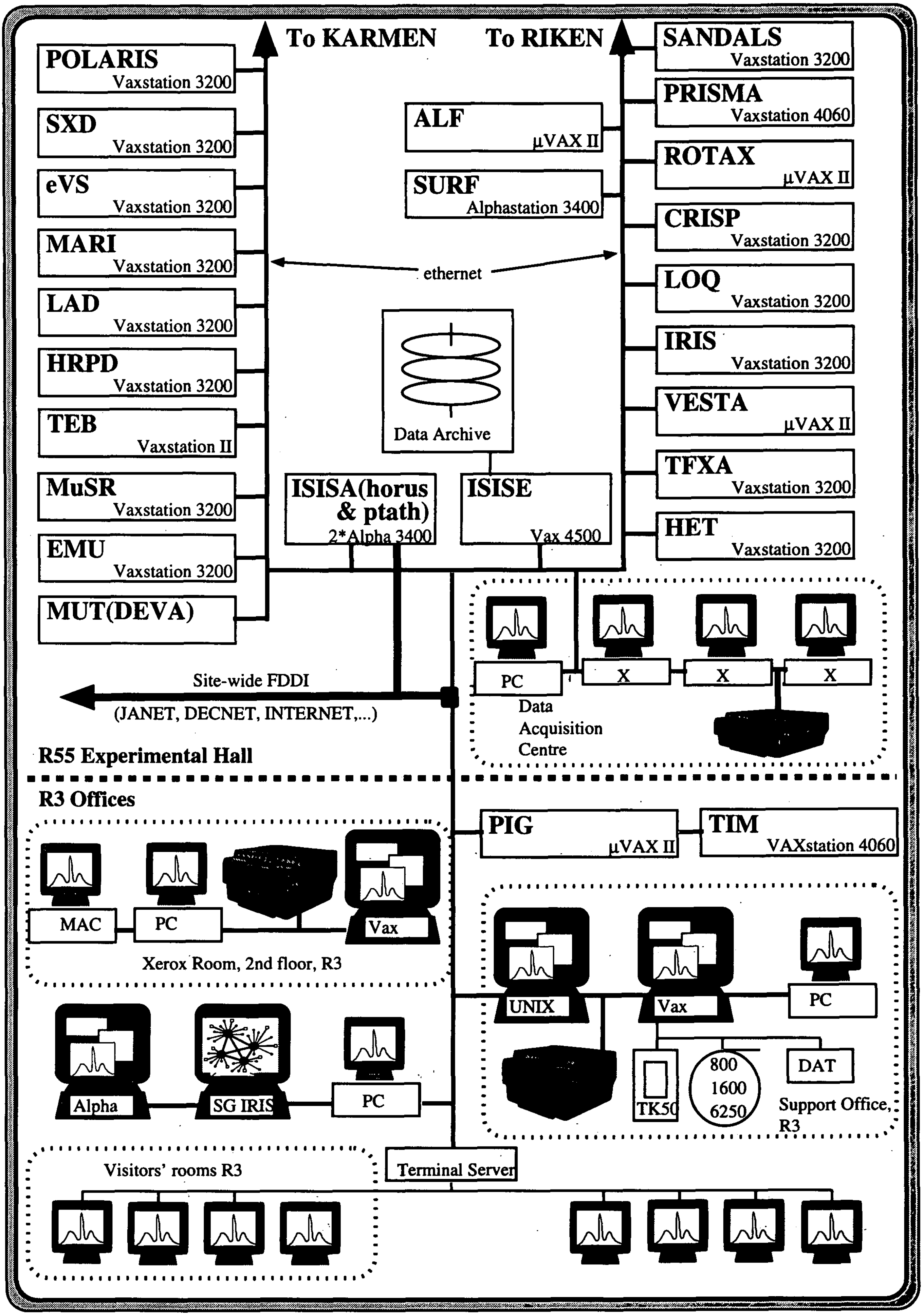
Notes

Section 4

Computing & Software

Contents



The PUNCH Network 95

The ISIS Computer System

The diagram on the previous page shows a schematic representation of the PUNCH (Pulsed Neutron Computer Hierarchy) Network at ISIS. The central (or "hub") computer, and the machine that most users will interact with, is called ISISE. This is a DEC VAX 4000 Model 500 with some 192 Mb of main memory, 10 Gb of magnetic disk storage and 40 Gb of optical disk storage. The ISIS instrument computers are either DEC VAX or DEC Alpha workstations. The LOQ instrument computer (see Section 2.23) is a MicroVAX 3200 with 24 Mb of main memory and a 1.6 Gb hard disk. At the time of writing, ISISE and all of the principal instrument computers run the DEC OpenVMS operating system. The PUNCH Network supports TCP/IP protocols, thereby enabling remote terminal (Telnet) logins and file transfers (FTP), and is also part of the JANET (Joint Academic Network) in the UK.

Some useful IP addresses are:

ISISE	**130.246.53.5**	**ISISE.RL.AC.UK**
Hub Work 4	130.246.53.4	HUBWK4.ND.RL.AC.UK
OpenGENIE server	130.246.53.8	SUTEKH.ND.RL.AC.UK

Please refrain from making remote network connections to LOQ when ISIS is running - it makes life hard for the users running experiments! SUTEKH is an OSF/1 (Unix) system solely for running the new GENIE ("Genie 3") data display program. At the time of writing OpenGENIE is at an "alpha-release" stage. In addition, it can only be used to view "raw" (as distinct from "reduced") LOQ data. It is therefore made available for use on a "teach yourself" basis only.

ISIS Computer Support

The ISIS Computer Group operate a weekly support rota during normal working hours. There is no out-of-hours support. The "supporter" can be telephoned internally on x3029 or from outside on 0585-286687 (this is a mobile phone). Non-urgent queries can be emailed to the address SUPPORT @ ISISE.RL.AC.UK.

The Support Office is Room 1.37 in Building R3. This is the same building, but the floor above, the University Liaison Secretariat (ULS). It contains a full set of manuals for reference use. Copies of the *PUNCH Mini-Guide* can also be obtained from here.

Problems with COLETTE, the LOQ data reduction program, should be addressed to the LOQ Instrument Scientists.

PUNCH Accounts

Before you can log onto an ISIS computer you will need a "user account". These are issued to the principal applicant on a proposal (ie, one account per research group) upon completion and return of an account request form (see opposite). The form should be handed to the "supporter" on duty. The account remains active for 12 months, after which time it may be renewed (with the consent of an instrument scientist) or archived and deleted.

PuNCH VMScluster Account Request

USER DETAILS

SURNAME/FAMILY NAME

OTHER NAMES/PERSONAL NAMES

HOME ESTABLISHMENT

ADDRESS FOR CORRESPONDENCE

HOME ESTABLISHMENT E-MAIL ADDRESS

ACCOUNT DETAILS

DATE ACCOUNT REQUESTED

LENGTH OF VISIT

INSTRUCTIONS TO BE USED

☐ CRISP	☐ LAD	☐ ROTAX
☐ EMU	☐ LOQ	☐ SANDALS
☐ eVS	☐ MARI	☐ SURF
☐ HET	☐ MuSR	☐ SXD
☐ HRPD	☐ POLARIS	☐ TEB
☐ IRIS	☐ PRISMA	☐ TFXA

☐ Other (Please Name)..

USE TO BE MADE OF ACCOUNT

CONTINUED OVERLEAF ⇨

INSTRUMENT SCIENTIST AUTHORIZATION

I request that an account be created for the purpose stated above. Signature of Instrument Scientist Date

NOTIFICATION DETAILS

Who should be notified when this account is created? Name: E-Mail: Telephone:

NEW USER UNDERTAKING

I undertake to use this account only for the purpose stated above and not to allow an unauthorised person to make use of this account. I understand that I am responsible for the actions of any user to whom my password is disclosed. I consent to the information on this form being made available on the PuNCH VMScluster to other users of the system for informational purposes only. Signature of User Date

GUIDANCE FOR COMPLETION

1. Complete all sections
2. Hand in the completed form to the SUPPORT OFFICE, R3 Room 1-27
3. Account creation takes 30-40 minutes from receipt of the completed form.
4. Do not request a User Account if you have previously had an account. Contact SUPPORT and ask for your old account to be restored.

SUPPORT can be contacted on extension 3029 during working hours.

FOR OFFICIAL USE ONLY

USER ID:	UIC:	QUOTA:
SUPPORTER:	DATE:	RESTORED ACCOUNT?

The World-Wide Web

There are an extensive set of web pages on the *ISIS WWW Server* under the umbrella URL:

http://www.isis.rl.ac.uk/index.html

The information available includes a general overview of ISIS and neutron scattering, examples of science conducted at ISIS, the ISIS accelerator performance, how to apply for ISIS beamtime, who to contact, and lots more.

There are also another set of web pages devoted to LOQ. The *LOQ Home Page* is reproduced overleaf and is located at the URL:

http://ndnt01.nd.rl.ac.uk/instr/loq/loq.htm

Welcome To The LOQ Home Page !

LOQ is the only Small-Angle Neutron Scattering (SANS) instrument at ISIS, and one of the most popular ISIS instruments.

LOQ may be used to investigate the shape and size of large molecules, small particles or porous materials with dimensions in the range 1 - 100 nm. Length scales of up to 400 nm can be probed in highly anisotropic systems. This instrument should therefore be of interest to anyone involved in the study of colloids, polymers, biomolecules, alloys, composites or porous systems.

The principle benefit of conducting SANS experiments at ISIS is that a "white" incident beam is combined with time-of-flight detection techniques to give LOQ a very large dynamic range in scattering vector, Q, all of which is accessible in a single measurement without any need to reconfigure the instrument.

Typically, the duration of a LOQ experiment is 24 or 48 hours. Longer awards of beam time are possible in exceptional cases.

Stop Press ! A high-angle detector test module was successfully commissioned at the end of the last cycle. For further details, click here.

Check out the following:

- Technical information about LOQ
- LOQ-related manuals, handouts and programs
- LOQ data - getting it restored, appropriate mask files, etc
- Current status of LOQ - updates automatically, but click *Refresh* !
- Some examples of work conducted on LOQ
- References to published work performed on LOQ
- The LOQ Gallery - a selection of visualised data
- Who to contact about LOQ
- How to apply for beam time at ISIS
- ISIS cycle dates for 1996/97 & LOQ Schedule

- Konrad Ibel's World Directory of SANS Instruments
- Clickable periodic table of Neutron Scattering Lengths & Cross-Sections

- The ACA Small Angle Special Interest Group Home Page
- The IUCr Small Angle Commission Home Page
- List of Neutron Scattering WWW Servers

Last updated: 5-Mar-1996 by Steve King SMK@ISISE.RL.AC.UK

COLETTE

What is COLETTE?
COLETTE is the name of the LOQ data reduction program. The acronym COLETTE is a contraction of “correct LETI”, the Laboratoire d'Electronique et de Technologie de l'Informatique (or LETI) being the manufacturers of the original LOQ “area” detector.

Why COLETTE and not GENIE?
The standard ISIS data display and manipulation program, GENIE, was not designed to handle two-dimensional datasets, an essential operation in the processing of SANS data.

COLETTE runs on top of, and thereby incorporates, GENIE. Data “workspaces” are shared between COLETTE and GENIE. This means that a COLETTE user can “shell” into GENIE and use all of its functionality and then return to their COLETTE session without any loss of data. However, for reasons of speed and ease of use COLETTE effectively functions independently of GENIE, it has its own command set and its own data input and output routines.

System Requirements
COLETTE is designed to run on the DEC VAX family of computers under Digital's VMS (Version 5.x or higher) or OpenVMS (Version 6.1 or higher) operating systems. COLETTE also requires DEC GKS (Graphical Kernel System) Version 4.2 or higher. (Note: DEC GKS Version 5.0 is incompatible with GENIE. The next compatible version is GKS 5.2). At the time of writing COLETTE does not run on DEC Alpha computers (even those running OpenVMS).

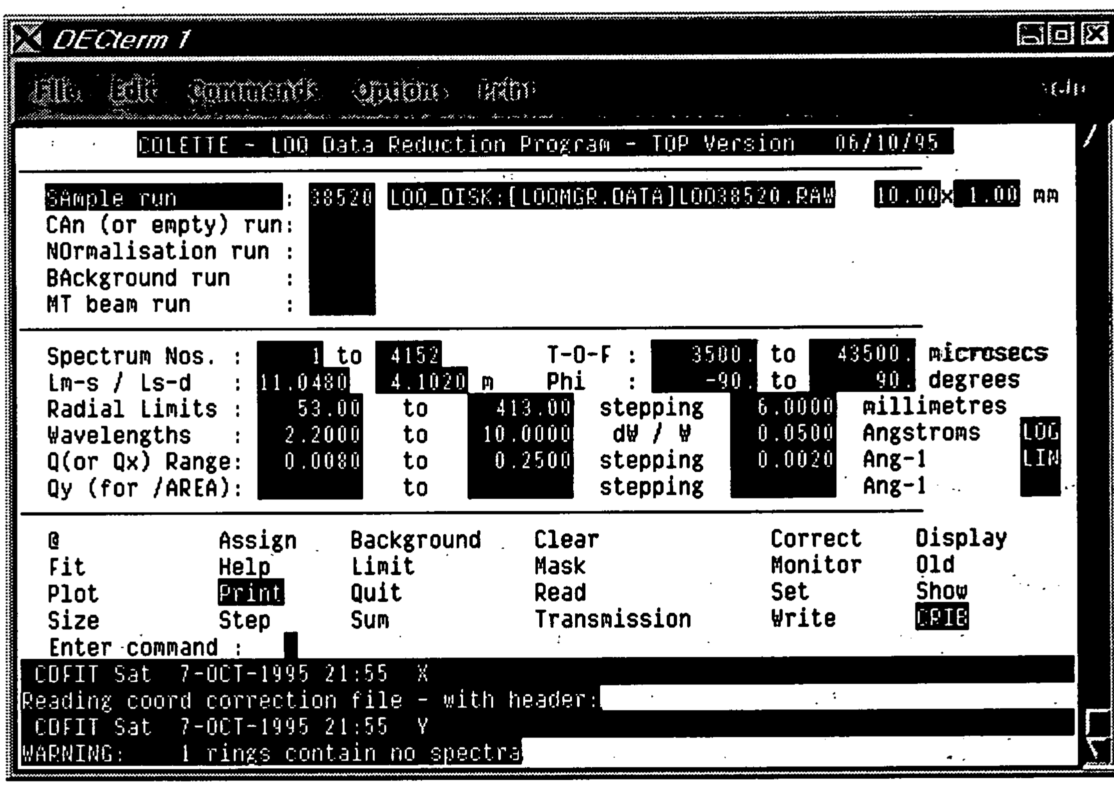
The disk space (a VAX disk blocks = 0.5 kB) and memory requirements (1 page = 0.5 kB) of the COLETTE executable image is at present:

COLETTE / GENIE Version 2.5(2) / DEC GKS Version 4.2	1900 blocks	35000 pages

COLETTE will be distributed free to *bone fide* educational establishments.

Using COLETTE

(A Simple Guide)



In the following document all of the commands to be typed are in **bold** type. Unless otherwise stated there is a <RETURN> at the end of each line. Most commands, their qualifiers and parameters can be abbreviated to just the first 3 letters. For additional information type HELP [command]. Should it become corrupted, the screen display may be "refreshed" by simply typing Control-W.

1) Run the program...

COL or **COL TEST** (ask your local contact or read the login notices)

COLETTE will respond with...

GKS$CONID = "DECW$DISPLAY"

CLASS = "WINDOWS"
OUTPUT = "POSTSCRIPT"
VERSION = "TOP"

...or something similar. GENIE will announce itself followed by various information notices about COLETTE and environmental setups. Finally the GENIE ">>" prompt will appear.

Please ignore any DECGKS error messages about incorrect Title & Border sizes; they're misleading!

NOTE:
Users who wish to import graphs produced by COLETTE into Microsoft WORD®, who wish to run COLETTE using PC KERMIT as a terminal emulator, or who wish to use a Regis graphics terminal should not invoke COLETTE in this way. Please see Appendix 5 instead.

2) Call COLETTE from GENIE...

>> **COL** or >> **LOQ** but not COL TEST

3) Assign the sample run...

ASSIGN/SAMPLE 1234

COLETTE will look for the file LOQ01234.RAW by default. Specify any other file descriptor; e.g. ASS/SA 1234.ADD

If you are using the Workstation in the LOQ cabin it is possible to look at the run currently in progress. To do this you must take a "snapshot" of the data in the DAE (Data Aquisition Electronics) and copy it to an area of the computers memory called the CRPT (Current Run Parameter Table). COLETTE must still be told where to look for the data however, and there are two ways of doing this, either by

ASS/SA CRPT

or

ASS/SA 0

The legend CRPT will be displayed instead of a filename. To actually read the data from the DAE into the CRPT type

JUMP "UPDATE

The screen will go blank for a few seconds and then ask you to press <RETURN>.

4) {Optional}
Assign the sample can run. This will be a run containing scattering to be subtracted from the sample, such as a solvent filled sample cell for example...

ASS/CA 1235

COLETTE will look for the file LOQ01235.RAW

5) Invoke the mask...

ASS/SA

@MASK

This executes MASK.COM. The instrument scientists frequently check the behaviour of the detector and have written this command file to read in the detector-to-spectrum mapping tables and to apply the necessary detector and time masks. It should normally only need to be called each time you enter COLETTE.

A number of information messages will appear in the bottom four lines of the screen.

If you are a regular LOQ user with your own account on the HUB computer, please ensure that you copy a recent MASK.COM to your directory before you analyse any new data! Alternatively, the command @USER:MASK will execute the current mask file on the LOQ FEM. The last LETI detector compatible mask was MASK912.COM (that is, @MASKS:MASK912). Finally, if you are running LOQ at 50Hz you will need to use a special mask file, please ask your local contact.

... and then decide which detector bank's data you wish to process. By default, when you first start COLETTE it will process data collected on the main detector (low-Q regime). If you want to look at the high-angle bank (high-Q regime) then type...

@HIGHDET

If you then want to look at the main detector again, type...

@MAINDET

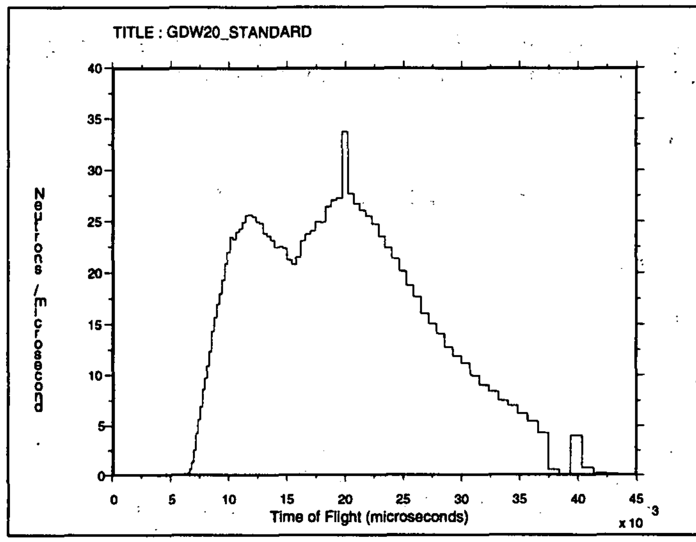
6) {Optional}
View the raw data as an intensity contour plot of the main detector. This display is most useful when studying anisotropic systems...

DISPLAY CONTOUR

A sub-menu will appear:
(a) To return to the main screen, type **0**.
(b) To normalise the contour levels, type **1** followed by the counts per unit area for contour level 1.
(c) To force the drawing of contour lines, type **2**
(d) To zoom on the centre, type **25**.

7) {Optional}
View the neutron time-of-flight spectrum as recorded by the main detector...

DISPLAY TIME

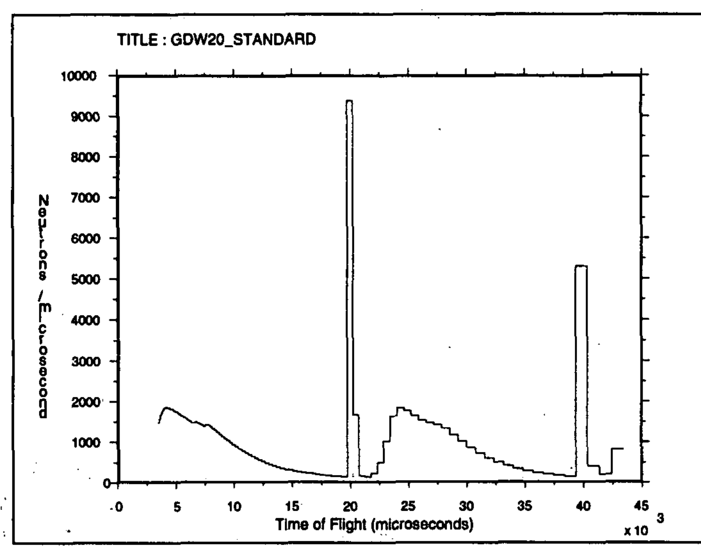


This may identify certain time channels which need to be masked out. The "prompt" pulse at 20ms must always be excluded. Again, this need only be done at the beginning of the experiment and will be done for you by the instrument scientists in MASK.COM.

LOQ also has two low-efficiency incident beam monitors. The neutron time-of-flight spectra that these record are stored in spectrum #1 and spectrum #2 and may be viewed by typing...

D/H S1

(where the /H qualifier signifies that the data is to be plotted in histogram format)

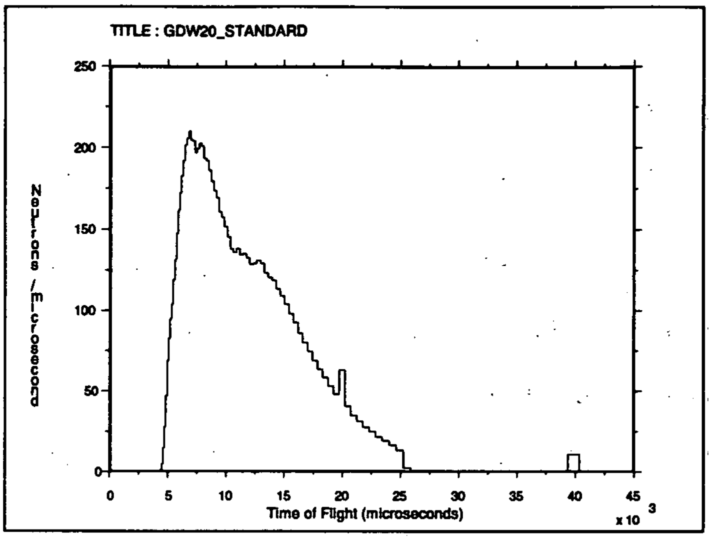


S1 contains the "raw" TOF spectrum, and is unaffected by your choice of wavelength or collimation. Because LOQ is normally a 25Hz instrument you should see two ISIS pulses. If not, ask an instrument scientist to check that the chopper is running and that LOQ is synchronised to the SMP (Secondary Master Pulse).

or...

D/H S2

S2 contains the TOF spectrum at the sample position and is used to normalise the small angle scattering from the sample as a function of wavelength. It should bear some resemblance to the spectrum obtained by DISPLAY TIME although the absolute scales will be different.



8) Calculate the transmission of the sample (also see the note under Step 10)...

TRANSMISSION/SAMPLE/MEASURE

COLETTE will respond with

"Enter transmission run number for sample run >> " **1236**

"Reading LOQ_DISK:[LOQMGR.DATA]LOQ01236.RAW"

"Enter direct beam run number >> " **1237.ADD**

"Reading LOQ_DISK:[LOQMGR.DATA]LOQ01237.ADD"

"Transmission contained in Workspace 21"

Write the transmission for the sample out as an ASCII datafile...

WRITE W21 TEMP:1236.TRAN or **WRITE W21 USER$DISK:[ABC01]1236.TRAN**

NOTE 1:
If you have also assigned a can and it is anything other than an empty beam run (ie, a scattering run through an empty sample position) then you must also calculate the transmission of the can...

TRANSMISSION/CAN/MEASURE

COLETTE will respond with

"Enter transmission run number for can run >> " **1238.RAW**

"Reading LOQ_DISK:[LOQMGR.DATA]LOQ01238.RAW"

"Enter direct beam run number >> " **1237.ADD**

"Reading LOQ_DISK:[LOQMGR.DATA]LOQ01237.ADD"
"Transmission contained in Workspace 22"

Write the transmission for the can out as an ASCII datafile...

WRITE W22 TEMP:1238.TRAN or **WRITE W22 USER$DISK:[ABC01]1238.TRAN**

NOTE 2:
If you forget to calculate the transmission then COLETTE will use whatever was calculated the last time you issued the above commands. If you forget what is in any workspace you can type SHOW W21, etc. See Appendix 3.

To clear a transmission use...

TRANSMISSION/CLEAR

This will set the transmission of the assigned run at all wavelengths currently in use to 1·0.

NOTE 3:
The TRANSMISSION/MEASURE command can be used in the format...

TRANSMISSION/MEASURE {sample transmission run #} {direct beam run #}

The {} brackets are not typed however!

If you omit to enter the direct beam run then COLETTE will use the last one entered.

NOTE 4:
Linearly-fitted transmissions of the LOQ polymer standards are stored within COLETTE and may be used in place of experimentally-measured transmissions for these samples. To use these stored transmissions, type...

TRANSMISSION/STANDARD (for the "old" LOQ standard GDW20)

or

TRANSMISSION/BLEND (for the "new" LOQ standard TK48/49)
TRANSMISSION/COPOLYMER (for the background for the "new" LOQ standard)

9) Display the transmission of the sample...

DISPLAY/MARKER W21 or better **D/M W21 2 10 0 1**

The /MARKER qualifier tells COLETTE to display the individual datapoints. For a moderately weak scatterer the transmission should be a straight line. In reality it probably also decreases at higher wavelengths. You can check how statistically significant any deviations are by overplotting the error bars.

<RETURN> (if using a TeKtronix-type terminal)

PLOT/ERROR

COLETTE will automatically assume that you want it to use Workspace 21 again in this instance.

10) {Optional}
Fit the transmission to a straight line. In some instances this will improve the quality of subsequent subtractions.

FIT W21 2.2 6.5
This command has an implicit /LINEAR qualifier. It is recommended that you only fit transmissions over the range 2.2 - 6.5Å. A quadratic fit is also possible using the command FIT/QUADRATIC.

Note that in this example the data in Workspace 21 is overwritten with data interpolated from the fit. To store the fit separately, say in Workspace 1, use...

FIT W21 2.2 6.5 W1

NOTE:
Some MASK.COM files include the command FIT/TRANSMISSION. If you are using such a file, all transmissions that you calculate will automatically be fitted. In such cases the "actual" transmission is placed in W25 (regardless of whether you have calculated the transmission of a sample run or of a sample can run) and W21, W22, etc., only contain the fitted transmission. A message to this effect is always displayed at the bottom of the screen. To turn this feature off use FIT/TRANSMISSION/CLEAR.
Check the fit by overplotting on the original data...

PLOT/LINE W21 or **PLOT/LINE W1** or **PLOT/LINE W25** (respectively)

<RETURN> (if using a TeKtronix-type terminal)

11) {Optional}
If you have assigned a can run then repeat steps 9) & 10) with Workspace W22.

12) Check that the beam size is correct. This is the first value after the run designation at the top right of the screen. Normally LOQ is run with an 8mm diameter beam at the sample. However, if you are not using standard 1mmx10mmx30mm quartz cells then your local contact may have altered the beam size. Unless the correct beam width was set in the instruments CHANGE utility it will be necessary to alter it in COLETTE using, for example, ...

SIZE/WIDTH=11 or **SIZE/SAMPLE/WIDTH=11**

... to set it to 11 mm. If it is necessary to change the beam size for the sample can aswell then you can use...

SIZE/CAN/WIDTH=11

Any such change of collimation is normally recorded in the instruments log book alongside the remark SNOUT or A_{snout}.

IMPORTANT !
When running your samples try to ensure that the incident collimation is the same for both the sample run and the sample can run. As you can see, COLETTE will still be able to handle the data if they are not, but the resulting differences in beam divergence, views of the incident beam and scattering around the beamstop will give rise to unquantifiable effects at low-Q. You have been warned!

13) Check that the sample thickness is correct. This is the second value after the run designation at the top right of the screen. If it is not...

SIZE/THICK=2.0 or **SIZE/SAMPLE/THICK=2.0**

... will change it to 2.0 mm, for example. If it is necessary to change the thickness of the sample can aswell then you can use...

SIZE/CAN/THICK=2.0

The sample thickness should be set every time you assign a new run, unless you entered the correct thickness into the .RAW file by specifying the thickness in PILOT.COM or by using the CHANGE utility (see the "PUNCH User Guide"). The default value for the thickness of a run is 1.0mm.

14) Check that the flight paths are correct. These are the values alongside the legend "Lm-s / Ls-d" and are the moderator-to-sample and sample-to-detector distances respectively. If these values are wrong then your Q-scale will be wrong!

These lengths are normally defined for you by the instrument scientists using the CHANGE or MENU utilities or by using the MODIFY command (see the "PUNCH User Guide") but you can easily check whether the values displayed are correct:

Lm-s
The back face (nearest your samples) of the Aperture #2 housing (the big rusty upright block in the sample pit) is 10.744 m from the moderator.

Ls-d
This is 15.150 - Lm-s.

If it is necessary to change these values then type...

SET LENGTHS

COLETTE will respond with

"NOTE: M-S = 11.0540 m S-D = 4.4190 m M-M = 10.4310 m"

"Mod-Sam length ==> " **10.96**

"Sam-Det ==> " **4.513**

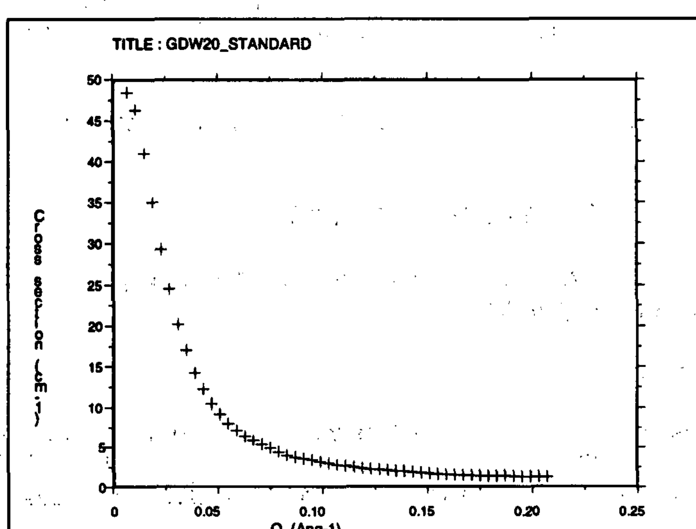
"NOTE: M-S = 10.9600 m S-D = 4.5130 m M-M = 10.4310 m"

If you are using the LOQ sample changer with LOQ's own quartz cell racks then Lm-s = 11.048 and Ls-d = 4.102 m. A table of other values is normally displayed in the LOQ Cabin.

15) Reduce the data (but please read Appendix 6, Appendix 7 and Appendix 8 first)...

CORRECT/SA W1

This would process the sample data (by rebinning into lambda, dividing by the monitor count, scaling by the direct beam for detector efficiency, scaling by the transmission and sample volume, weighting the masked data by zero and finally rebinning the data into Q weighting by solid angle) and put the data as a function of Q into Workspace 1.



A more preferable (where possible) reduction would be to subtract the sample can data from the sample data. To do this use...

CORRECT/SC W1

Write the corrected data out as an ASCII datafile of cross-section versus scattering vector...

WRITE W1 TEMP:1234.IQ or **WRITE W1 USER$DISK:[ABC01]1234.IQ**

or

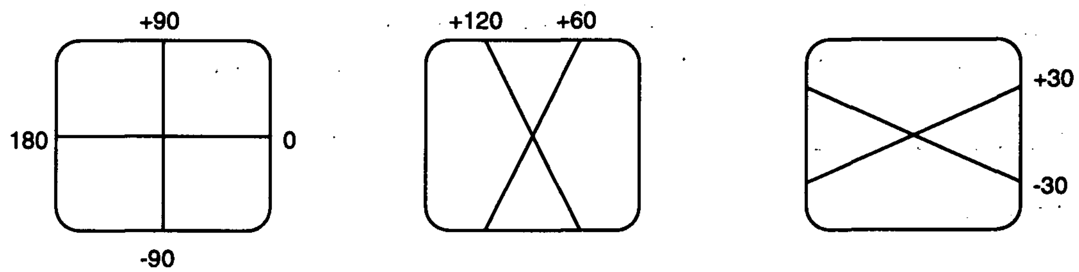
WRITE/TITLE="My Plot" W1 TEMP:1234.IQ

NOTE 1:
Data reduction by the above method involves a radial average and so the result is just one-dimensional (cross-section versus Q). COLETTE is however capable of making a two dimensional reduction; i.e., cross-section as a function of both Q parallel and Q perpendicular to the direction of an applied tensor (e.g., shear flow, magnetic field, etc.). For details of how to perform such a reduction, see section 19.

NOTE 2:
It is possible to only reduce data in selected sectors of the detector. This is done by altering the range of azimuthal angles used by typing...

LIMIT/PHI {minimum angle} {maximum angle}

For a full radial average (the default) the minimum angle should be set to -90 and the maximum angle to +90. Other sectors may be chosen with reference to the diagram below:



The LIMIT/PHI command only takes effect when another CORRECT is issued.

NOTE 3:
It is possible to calculate a "fully normalised" scattering cross-section, see Section 20.

16) Display the corrected cross-section...

DISPLAY/MARKER W1

<RETURN> (if using a TeKtronix-type terminal)

...and overplot the error bars...

PLOT/ERROR W1

<RETURN> (if using a TeKtronix-type terminal)

Other qualifiers available include...

/YLIN - for a linear Y axis
/XLIN - for a linear X axis
/XYLIN - for linear X and Y axes
/YLOG - for a logarithmic Y axis
/XLOG - for a logarithmic X axis
/XYLOG - for logarithmic X and Y axes

/SPECIAL - for other specialised representations (e.g., Guiner, Porod, Kratky). You will be prompted to select the representation you require and prompted to enter a background to be subtracted from the data. This can either be a single value (if in doubt enter 0) or a workspace as Wn. If you give a workspace it must have identical Q bins to the data workspace.

NOTE:
Different types of marker can be selected with the command...
ALTER MARKER n

<RETURN>

where -13 ≤ n ≤ -1 (-1="●") and 1 ≤ n ≤ 5 (1="•", 2="+", 3="✱", 4="o" and 5="×").

17) {Optional}
To generate a hard copy of the display on a Laser Printer, type...

PRINT

This tells COLETTE to write the graphics screen to a file called DEC_POSTSCRIPT.DAT in the directory from which you are running COLETTE. You will then be asked to select a printer. Choose...

0 - if you are in R3 (the ISIS administration building)
2 - if you are in the DAC (the Data Assessment Centre in the experimental hall)
4 - if you are in the LOQ cabin (the printer is in the CRISP cabin behind LOQ)
9 - if you are in the LOQ cabin (the printer is in the HET cabin opposite LOQ)

NOTE 1:
The PRINT command can be used in the form...

PRINT/ON=n/COPIES=m

...where n is the laser printer number (shown above) and m is the number of copies - between 1 and 9 - required. The default value of m is 1.

18) {Optional}
To read COLETTE format ASCII datafiles of, say, corrected data or transmissions back into COLETTE workspaces, type...

OLD W1 TEMP:1234.IQ or **OLD W21 USER$DISK:[ABC01]1236.TRAN**

NOTE:
If a transmission file is read back into W21, W22, W23 or W24 then COLETTE will not use that data in a CORRECT unless you first issue...

TRANSMISSION/WORKSPACE

...in place of the TRANSMISSION/MEASURE command.

19) (Optional)
Perform a two-dimensional reduction of the data. It is first necessary to establish Q limits in the directions parallel and perpendicular to the applied tensor, what COLETTE refers to as the Q_X and Q_Y directions using the commands...

LIMIT/QX 0 {maximum value of Q_X}
LIMIT/QY 0 {maximum value of Q_Y}

...and then to determine the number of Q bins (points) in either direction with...

STEP/QX/LIN {interval}
STEP/QY/LIN {interval}

Due to the way in which data is stored inside COLETTE there is a limit to the number of Q bins which can be handled. As a rough guide

(maximum value of Q) / (interval) ≤ 100

NOTE:
The use of logarithmic (/LOG) Q bins in 2D reductions is not recommended. Only use them in normal 1D reductions (i.e., STEP/Q/LOG). See Section 15.

If necessary, reset the azimuthal limits...

LIMIT/PHI -90 90

The data may then be corrected as before using...

CORRECT/SA W61 or **CORRECT/SC W61**

...but, the result must be written to a 2D workspace (see Appendix 3), such as W61.

NOTE:
CORRECT/SA W61 and CORRECT/SC W61 are identical with CORRECT/AREA/SA W61 and CORRECT/AREA/SC W61 respectively. Providing that a 2D workspace is given as an argument the /AREA qualifier can be omitted.

To display the result of a 2D reduction, use...

DISPLAY W61

...as the data can only be displayed as line contours. The /E, /H or /M qualifiers will have no effect on the display of 2D workspaces.

To write the data out as an ASCII datafile of cross-section versus Q_X and Q_Y, use...

WRITE W61 TEMP:1234.LQA or **WRITE W61 USER$DISK:[ABC01]1234.QXY**

...or, to write the data out in a shorter binary format, use...

WRITE/BIN W61 TEMP:1234.LQB

NOTE:
WRITE W61 is identical with WRITE/AREA W61. Providing that a 2D workspace is given as an argument the /AREA qualifier can be omitted.

20) (Where appropriate)
It is possible to calculate a "fully normalised" scattering cross-section (as is often done on reactor-based SANS instruments) where the scattering from the sample is divided by that from a normalisation sample. However, this procedure should be used with caution on LOQ because it is very difficult to find a normalisation sample with a well-defined cross-section over all of the LOQ wavelength range. This limitation applies to the two most common normalisation samples, vanadium and water.

The only purpose of the normalisation is to eliminate the detector efficiency from the scattering law. On a time-of-flight SANS instrument, like LOQ, it is preferable to determine the detector efficiency directly. When you use the CORRECT command in COLETTE the program automatically reads in a file containing the efficiency of the LOQ main detector unless you issue CORRECT/SCNB or CORRECT/FULL (the two qualifiers are equivalent (see below).

To make a full normalisation, first assign a normalisation run (eg, a vanadium run)..

ASSIGN/NORMALISATION 1239

...followed by a background run (ie, whatever must be subtracted from the normalisation run. The background run may be considered to be a can run for the normalisation)...

ASSIGN/BACKGROUND 1240

...then calculate their respective transmissions...

TRANSMISSION/NORMALISATION/MEASURE 1241.RAW 1237.ADD

FIT W23 2.2 6.5

TRANSMISSION/BACKGROUND/MEASURE 1242.RAW 1237.ADD

FIT W24 2.2 6.5

Assuming that the width and thickness of both the normalisation and background runs are correctly set (see Sections 12 & 13 if they are not), a fully normalised reduction may then be made using...

CORRECT/SCNB W2 or **CORRECT/FULL W2**

Should a full two-dimensional normalisation be required then the above CORRECT commands should be replaced by (but also see Section 19), say, ...

CORRECT/SC W62

CORRECT/NB W63

AREA/ARITHMETIC

...followed by, for example, ...

64 0.0 1.0 62 63

...to do W64 = 0.0 ´ W64 + 1.0 ´ W62/W63

Notes

APPENDIX 1

Analysing Old Data

(a) DATA COLLECTED WITH THE LETI DETECTOR AND OLD BEAMSTOP (ONE WITH HOLES!)
This is data collected prior to ISIS Cycle 1 1990 - Run Numbers < 4493

(1) Assign an empty beam run (**ASS/MT {empty beam run number}**)
(2) Use an appropriate mask file (**@MASKS:MASK89**)
(3) Use **TRANSMISSION/CALCULATE** (instead of TRANSMISSION/MEASURE)

(b) DATA COLLECTED WITH THE LETI DETECTOR AND NEW BEAMSTOP
This is data collected prior to ISIS Cycle 3 1991 - 4493 < Run Number < 9607

(1) Use an appropriate mask file (**@MASKS:MASK912**)
(2) Use the correct detector efficiency file:-
MONITOR/DIRECT=LOQ$DISK[LOQMGR]BEAM_FUDGED.RKH

NOTE 1:
If you are changing detector types during the same COLETTE session you must clear all detector and time masks before reading a new mask file using...

MASK/CLEAR
MASK/TIME/CLEAR

NOTE 2:
Old masks and masks generated for special purposes are stored in the directory LOQ$DUA0:[LOQMGR.MASKS] which has been equivalenced to the logical name MASKS: To find a mask file for a particular period type...

DIR/DATE MASKS:

... at a DCL prompt ($ or LOQ>). Do not under any circumstances delete, rename or edit any of the files in this directory. If you need help please contact an instrument scientist.

APPENDIX 2

Logging On At ISIS

An increasing number of terminals at ISIS now connect to the PUNCH computer system via an EtherNet protocol called LAT (Local Area Transport). This includes terminals in the DAC and LOQ. These so-called "LAT terminals" will be routed through one of two types of terminal server; a Lantronix Server (which displays a Vista> prompt) or a DECserver (which displays a Local> prompt).

- **If logged out of a Lantronix**
  [press return]
  Vista VCP-1000
  Enter Username> mickey_mouse
  Vista>

- **If logged out of a DECserver**
  [press return twice]
  DECserver 90TL ...etc
  Enter Username> fred_flintstone
  Local>

- **To log onto the HUB (central computer)**
  C ISISE <RETURN>

- **To log onto the LOQ FEM**
  C LOQ <RETURN>

- **To view the Protons Per Pulse display**
  C PPP <RETURN>
  (There are only a limited number of PPP connections so please do not "hog" them and always disconnect when you have finished. Thank you.)

- **To make an additional alternate connection** (3 alternate connections are allowed)
  <BREAK>
  C [new nodename]

- **To clear a connection**
  <BREAK>
  DISCO

- **To resume alternate connection number 1**
  <BREAK>
  RES 1

A plea! When you have finished your experiment and are analysing data in the DAC or back in your home institution, please DO NOT logon to the LOQ FEM either as LOQ or under your own username. Use the much more powerful ISIS HUB computer called ISISE instead. Otherwise you only slow LOQ down for the current users (and for yourselves)! Thank you.

APPENDIX 3

COLETTE Workspaces

COLETTE has 67 workspaces for data. Most can be used without any restriction, although some are reserved for specific purposes.

W1	Normal (1D) workspace	
.	.	
.	.	
W14	Normal (1D) workspace	
W15	Graphics buffer	RESERVED
W16	Monitor spectrum versus wavelength for sample run	RESERVED
W17	Monitor spectrum versus wavelength for can run	RESERVED
W18	Monitor spectrum versus wavelength for normalisation run	RESERVED
W19	Monitor spectrum versus wavelength for background run	RESERVED
W20	Flat-scatterer cross-section versus wavelength (on test)	RESERVED
W21	Transmission versus wavelength for sample run	RESERVED
W22	Transmission versus wavelength for can run	RESERVED
W23	Transmission versus wavelength for normalisation run	RESERVED
W24	Transmission versus wavelength for background run	RESERVED
W25	Fitted transmission versus wavelength for assigned run	RESERVED
W26	Main detector efficiency versus wavelength	RESERVED
W27	High-angle detector efficiency versus wavelength	RESERVED
W28	Normal (1D) workspace	
.	.	
.	.	
W60	Normal (1D) workspace	
W61	2D workspace for use with CORRECT/AREA only	
W62	2D workspace for use with CORRECT/AREA only	
W63	2D workspace for use with CORRECT/AREA only	
W64	2D workspace for use with CORRECT/AREA only	
W65	Used by CORRECT/AREA (counts buffer)	RESERVED
W66	Used by CORRECT/AREA (solid angle buffer)	RESERVED
W67	2D graphics buffer	RESERVED

APPENDIX 4

Absolute Intensities (also see Page 1-8)

COLETTE reduces data in such a way that the scattered intensity (or, more formally, the differential scattering cross-section) you see displayed on the Y-axis has units of cm^{-1}; ie, absolute dimensions. The data <u>should</u> also be on an absolute scale. However, to correct for small instrumental non-linearities, geometrical factors, repeatability of sample postioning, etc, it is in your own interests to run a *LOQ Standard Sample.*

Each of the two *LOQ Standard Samples* is a solid blend of perdeuterated polystyrene dispersed in protonated polystyrene matrix. The "new" (TK48/49) standard has a special copolymer background, the "old" (GDW20) standard does not.

The correction procedure requires that you make 2 runs (for say a minimum of 1M Counts each) at each instrument geometry that you use:

1. a scattering run on the Standard Sample
2. a scattering run on an empty sample position (ie, an EMPTY BEAM run) or on an empty cell (if the GDW20 Standard is placed in a cell; ie, between windows), or a scattering run on the copolymer background for TK48/49 (in a cell if necessary).

Then reduce the data as follows...

ASS/SA {standard sample scattering run}
SIZE/THICK=w (the thickness of the LOQ Standard Sample in mm)
SIZE/WIDTH=x (where x is beam diameter in mm at the sample)
TRANS/STANDARD or TRANS/BLEND
ASS/CA {empty beam or cell run or copolymer background}
SIZE/THICK=v (if the GDW20 standard is being used, v=w)
SIZE/WIDTH=x

then either

TR/CLEAR
(for an empty beam run - resets the can transmission in W22 to unity all wavelengths)

or

TR/CAN/MEASURE {sample can transmission run} {direct beam run}
(for an empty cell run)

or

TR/COPOLYMER
(for the copolymer background if the TK48/49 standard is being used)

LIMIT/PHI -90 90
LIMIT/Q 0.008 0.25
STEP/Q/LIN 0.002
CO/SC Wn (where n is a workspace number)
WRITE Wn TEMP:STANDARD.Q

Now fit the I(Q) data to a Debye Gaussian coil model and determine I(Q=0) and the (z-average) radius of gyration, Rg. Both RKH's "FISH" program and SMK's "SANDRA" program will do this. To use SANDRA type...

SANDRA

... at a DCL prompt ($ or LOQ>) and then enter...

1
TEMP:STANDARD.Q
1
24
1
 # REM 0.1 0.3
 # SET

1	Intensity @ Q=0	**: 350**
2	Z-average Rg	**: 50**
3	Background	**: 1**

 # GO
 # N
Now check the quality of the fit...
 # P D
 # P O F
... and if you are satisfied with it then record the values of I(Q=0) and Rg.
 # KEEP
 # P R
 # KEEP
 # EX

<RETURN>
-1

To plot the fit and the residuals on laser printer n...

PLASERn SYS$SCRATCH:FRILLS.PS;-1
PLASERn SYS$SCRATCH:FRILLS.PS

The expected values of I(Q=0) and Rg for the *LOQ Standard Samples* are given on Page 1-8. So, now scale the observed scattering from your samples (in say Wm) by the ratio that the observed standard scattering must be scaled by in order to bring the I(Q=0) values into agreement...

QUIT

>> **Wm =Wm × {expected I(Q=0)} / {measured I(Q=0)}**
>> **COL**

APPENDIX 5

Special Versions of COLETTE or COLETTE Startups

(a) TO GENERATE MICROSOFT WORD® COMPATIBLE GRAPHICS FILES

Normally when you issue a PRINT command COLETTE creates a graphics file called DEC_POSTSCRIPT.DAT in your current directory and then queues it to the printer that you select. This is because the default hardcopy output language is PostScript. Although Microsoft Word can import PostScript files they tend to require a great deal of memory (for example, it would be stupid to try and import a COLETTE contour plot in PostScript - we've tried!). Converting the PostScript to GIF format and then converting the GIF format into Windows Bitmap format (BMP) does work quite well, however.

If you want to import COLETTE graphs into Microsoft Word run...

COL WORD

... instead of COL or COL TEST. This will cause COLETTE to load the Hewlett Packard graphics driver for hardcopy operations and to create files called DEC_HP7475.DAT. COLETTE will still queue the file to print but the operation will fail. These files can be transferred to a PC and imported into Word using *INSERT PICTURE.*

(b) TO RUN COLETTE THROUGH A PC USING KERMIT AS A TERMINAL EMULATOR

Instead of running COLETTE using COL or COL TEST try **COL KERMIT** or type the following at a DCL prompt ($ or LOQ>)...

SET COMMAND LOQ$DISK:[LOQMGR.COLETTE.XCOLETTE]XCOLETTE

... and then invoke COLETTE using...

NEWCOLETTE/DEV=KERMIT/SCALE=1.0 (equivalent to COL)

... or...

TESTCOLETTE/DEV=KERMIT/SCALE=1.0 (equivalent to COL TEST)

(c) TO RUN COLETTE ON VAX 4000 WORKSTATIONS

Some newer VAX workstations have a more recent version of the DECGKS graphics library and so some display aspects of COL and COL TEST may not work properly (eg, cursor commands, etc). Instead, run...

COL SURF

(d) RUNNING COLETTE OVER DECNET OR TELNET IN A DECWINDOWS ENVIRONMENT

In order to redirect the graphical output to your workstation screen you will need to type...

SET DISPLAY/CREATE/NODE={your node name; eg, SURF}

... or...

SET DISPLAY/CREATE/NODE=aaa.bbb.cc.d/TRANSPORT=TCPIP

... where aaa.bbb.cc.d is your InterNet IP address (eg, ISISE is 130.246.84.3)

(e) TO RUN COLETTE WITH A REGIS GRAPHICS EMULATOR

Some commercial PC terminal emulation software uses REGIS escape codes when generating graphical displays. If you are using such software then invoke COLETTE using...

COL VT

APPENDIX 6

Detector Coordinate Corrections

When a neutron arrives at the main detector its time of arrival and position on the detector are noted by the electronics. But, because of the way in which all neutron detectors work, the position that is recorded is not the actual physical position at which the neutron arrived. In general the recorded position is out by a few mm. This discrepancy increases as you move out from the centre of the detector. This behaviour is known as "detector non-linearity".

From October 1994, COLETTE now corrects for detector non-linearity by default, using a procedure devised by RKH. A more detailed description of both the problem and the correction procedure may be found in a handout available from RKH. Also see Appendix 8.

To see if the detector coordinate correction is active, type...

SHOW TRANS or **SHOW MONITOR**

...and look at the second page of output (press <RETURN>) for..

"X coordinate correction is in use"
"Y coordinate correction is in use"

To deactivate the detector coordinate correction, type...

SET/XCOR/OFF or **SET/XCOR/CLEAR**
and
SET/YCOR/OFF or **SET/YCOR/CLEAR**

APPENDIX 7

Pixel by Pixel Detector Efficiency Corrections

Because of the way in which all neutron area detectors are built and operated, the efficiency with which a neutron is detected varies from position to position (pixel to pixel).

From October 1994, COLETTE now corrects for the detector efficiency on a pixel by pixel basis by default, using a procedure devised by RKH. A more detailed description of both the problem and the correction procedure may be found in a handout available from RKH.

To see if the detector coordinate correction is active, type...

SHOW TRANS or **SHOW MONITOR**

...and look at the second page of output (press <RETURN>) for..

"Cell by cell normalisation is ON"

To deactivate the pixel by pixel detector efficiency correction, type...

MON/FLAT/OFF or **MON/FLAT/CLEAR**

NOTE:
This efficiency correction is completely separate from the "normal" wavelength-dependent detector efficiency correction which is always used, irrespective of whether this pixel by pixel efficiency correction is activated or not.

APPENDIX 8

Semi-automatic Determination of the Beam Centre Coordinates

The apparent coordinates of the transmitted (or "through") beam on the main detector are periodically checked by the instrument scientists and entered in the mask file. If necessary, these coordinates may be changed with the command...

SET CENTRE [X value in mm] [Y value in mm] 0 0

Determining the beam centre coordinates is relatively straightforward but somewhat involved. To make things easier the procedure has now been semi-automated. The following description assumes that you have assigned a sample run and a can run, read in an appropriate mask file and calculated the relevant transmissions. Then type...

FIT/MID/FILE=CENTRE64SC or **FIT/MID/FILE=CENTRE64SA**

WARNING! *This command runs very slowly on LOQ.*
It will also overwrite W31, W32, W33 and W34.

...and watch what happens! If you are using a Tektronix-type terminal it will be necessary for you to press return after each new plot is displayed. A more detailed description of this command can be found in a handout available from RKH.

SYSTEM COMPATIBILITY

Depending on the type of "Windows"-based terminal environment you are using (be it a PC running eXceed or some type of workstation) to run COLETTE, it is possible that you may experience problems in obtaining a GKS graphics window.

The compatibility charts on the following pages indicate known problem areas, and known fixes.

Notes

COLETTE (GENIE) COMPATIBILITY CHARTS

(When using Workstations or PC's with Windows-type Terminal Emulators)

1. When Redirecting Screen Output to a PC (running eXcursion(4) or eXceed(5))

From an ISIS	System Running GKS 4.2		System Running GKS 5.3		System Running GKS 6.0	
Windows Driver	**Motif**	**DecWindows**	**Motif**	**DecWindows**	**Motif**	**DecWindows**
Using GENIE 2.5(2)	✔	✔			✘(6)	✔
Using GENIE 2.5(6)	✔	✔			✘(6)	✔

From an ISIS	System Running GKS 4.2		System Running GKS 5.3		System Running GKS 6.0	
	Via an IP Connection from an AXP under OSF/1					
Windows Driver	**Motif**	**DecWindows**	**Motif**	**DecWindows**	**Motif**	**DecWindows**
Using GENIE 2.5(2)	✔ (1)	✔ (2)			✔ (3)	✔ (3)
Using GENIE 2.5(6)	✔ (1)	✔ (2)			✔	✔

1. But generates X_Font errors on startup and doesn't annotate graphs. Also ASCII screen escape codes are not interpreted correctly
2. But GKS icon has to be opened manually and cursor function cannot be used
3. But graphs are not displayed until you click in the GKS window
4. Windows™ 3.1 or Windows95™ requires at least eXcursion 1.1
5. Windows™ 3.1 requires eXceed 3.3.2 Windows for Workgroups™ 3.11 requires eXceed 3.3.2 or 4.0
6. Except with eXceed 4.0

2. When Redirecting Screen Output to a VAX (under OpenVMS)

From an ISIS	System Running GKS 4.2		System Running GKS 5.3		System Running GKS 6.0	
Windows Driver	Motif	DecWindows	Motif	DecWindows	Motif	DecWindows
Using GENIE 2.5(2)	✔					
Using GENIE 2.5(6)						

3. When Redirecting Screen Output to an AXP (under OpenVMS)

From an ISIS	System Running GKS 4.2		System Running GKS 5.3		System Running GKS 6.0	
Windows Driver	Motif	DecWindows	Motif	DecWindows	Motif	DecWindows
Using GENIE 2.5(2)	✘	✔			✔	✔
Using GENIE 2.5(6)	✘	✔			✔	✔

4. When Redirecting Screen Output to an AXP (under OSF/1) (4)

From an ISIS	System Running GKS 4.2		System Running GKS 5.3		System Running GKS 6.0	
Windows Driver	Motif	DecWindows	Motif	DecWindows	Motif	DecWindows
Using GENIE 2.5(2)	✘ (1)	✔ (2)			✔ (3)	✔ (3)
Using GENIE 2.5(6)	✘	✔ (2)			✔	✔

1. Fatal floating divide by zero error
2. But cursor function cannot be used
3. But graphs are not displayed until you click in the GKS window
4. Try issuing “mc decw$clock” first. If nothing happens, type “xhost + remote_node_name” to overcome the X security.

GKS Versions
To determine which version of GKS the ISIS system you are using is running, type

SHOW LOG GKS$VERSION

Notes for COLETTE Users:
GENIE 2.5(2) is invoked by typing COL (or COL DEBUG or COL TEST)

GENIE 2.5(6) is invoked by typing COL NEW or COL SURF

The command procedure which is run whenever a "COL" command is issued will normally automatically invoke COLETTE with an appropriate graphics screen device driver (referred to as the Windows Driver above). This is not possible in the following cases:

1. After a "SET HOST" login
2. After a "TELNET" login
3. When the terminal is routed through a terminal server

In these cases the user is required to select an appropriate device from a list of alternatives.

Silicon Graphics Workstations
Version 6 of the X11 Driver, normally shipped with Version 5 of the Irix Operating System, is <u>incompatible</u> with the DECGKS currently running on ISIS VAX/VMS systems. Although this driver is compatible with the DECGKS on some ISIS Alphas (eg; PTATH) COLETTE, unlike GENIE 2.5(7), does not run on Alphas!

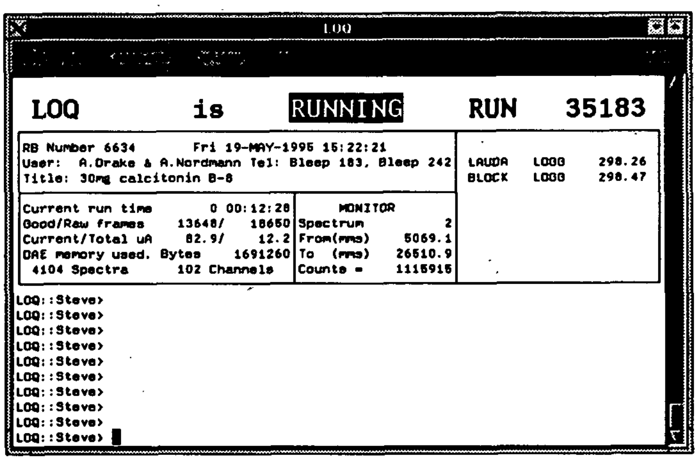
Version 5 of the X11 Driver, normally shipped with Version 4 of the Irix Operating System, <u>is</u> compatible with the DECGKS on ISIS VAX/VMS systems.

Unix Systems
To determine which version of the operating system a Unix machine is running, type

uname -r

A brief guide to the **CAMAC** *and what to do if something goes wrong.....*

1. Introduction
CAMAC (Computer Aided Measurement And Control) is the name of the system LOQ uses to control your sample environment from the instrument computer. In the picture of the LOQ "dashboard" display below, the CAMAC information area is the block on the right (under the run number).



Normally, the CAMAC information area of the dashboard will contain three columns:

- A Sample Environment Block Name (called a Seblock)
- A Seblock state identifier
- A value

2. The Seblock
There will be a Seblock for each piece of CAMAC-controlled equipment in use by LOQ, although not all of the Seblock's are always displayed. In general, the instrument scientists only display those Seblock's of direct use to the user, such as those associated with temperature sensors for example. Seblock's associated with, say, the LOQ chopper are not usually displayed unless you are running LOQ in an unusual manner. The normal set of LOQ Seblock's, and what they control, are:

- TEMP — Eurotherm temperature sensor (programmable for type and scale)
- TEMP1 — Eurotherm temperature sensor (programmable for type and scale)
- LAUDA — LAUDA Circulating Bath temperature (°C)
- BLOCK — LAUDA Circulating Bath <u>external</u> temperature sensor (°C)
- C_SPEED — Chopper rotational frequency (Hz)
- C_PHASE — Chopper phase delay (ms)
- C_INDEX — Chopper opening (degrees)
- TRANS — Transmission monitor & Aperture 2 controller

The Seblocks CHANGER, HAAKE and MAGNET are not true CAMAC Seblocks. For more information on these Seblocks, see the CAMEX document starting on Page 4-35.

3. The state identifier
This tells you what the CAMAC is doing with a particular Seblock and will be one of the following:

- LOGG CAMAC will periodically interrogate the device for the current value (logging)
- FALS CAMAC is not logging the device
- MOVE CAMAC is changing the value associated with the device <u>or</u> a new value is being read by the CAMAC (this should be a transient state!)
- TRUE A new value for the device has been correctly set (this is a transient state)

Note: Some Seblocks function quite normally without being in a LOGG state. On LOQ this applies to C_SPEED, C_PHASE, C_INDEX and TRANS.

4. The value
If the Seblock is in a LOGG state, this will be the value that CAMAC last read from the associated device and it have the units specified in Section 2 above. If the Seblock is in a FALS state, this will either be the last value read from, or the last setpoint issued to, the associated device.

5. CAMAC commands
These fall into two groups, those that set the device associated with a particular Seblock to a particular value (CSET commands), and those that read the current value of the device (CSHOW commands). For further information you should consult Chapter 5 ("Using an ISIS Instrument") of the black "PUNCH Manual".

In the following "LOQ>" is the computers prompt. Angle brackets (< >) indicate something which you must supply, but square brackets ([ ]) indicate something which is optional.

5.1 CSET commands

5.1.1 To ENABLE logging on a Seblock
The format of the command is:

```
LOQ>  CSET  <Seblock>/LOG[=t]
Eg;   CSET  TEMP/LOG=120
```

where t is in seconds. The default logging interval is 10 seconds. It is strongly recommended that you do not log any faster than at 5 second intervals. To change the logging interval it is first necessary to disable logging (see Section 5.1.2).

On LOQ it is only the temperature sensors which might need to be logged. Under no circumstances should log commands be issued on the Seblock's C SPEED, C PHASE or C INDEX - doing so will cause the CAMAC to "hang"!

5.1.2 To DISABLE logging on a Seblock
The format of the command is:

```
LOQ>  CSET  <Seblock>/NOLOG

Eg;   CSET  TEMP/NOLOG
```

5.1.3 To change the value of a device (and it's Seblock)
The format of the command is:

```
LOQ>  CSET  <Seblock>[/qualifier]  <new value or identifier>

Eg;   CSET  TEMP  298.0
      CSET  LAUDA/RANGE=2  25.0
      CSET  CHANGER  262.00
      CSET  CHANGER  J
```

5.1.4 To change the value of a Seblock (but not the device)
The format of the command is:

```
LOQ>  CSET  <Seblock>/VALUE=n

Eg;   CSET  CHANGER/VALUE=262.00
```

The /VALUE qualifier may be used to set the Seblock (and therefore CAMAC) to a value that has been achieved by manual manipulation.

5.1.5 To display/not display a Seblock in the CAMAC information area
The format of the command is:

LOQ> CSET <Seblock>/DISPLAY
LOQ> CSET <Seblock>/NODISPLAY

Eg; CSET TEMP1/DISPLAY

In the case of Seblock's TEMP and TEMP1, it is possible to display both the thermoelectric voltage (in mV) returning from the sensor in addition to the "converted" reading. To do this the format of the command is:

LOQ> CSET TEMP/CONVDISP
LOQ> CSET TEMP1/NOCONVDISP

5.1.6 Other CSET commands
Some CSET commands do not operate on a Seblock but rather they operate on the whole CAMAC instead. Consequently they should be used with great care! These commands may be divided into two groups:

LOQ> CSET CAMAC/RESET	Resets the CAMAC & Seblocks
LOQ> CSET CAMAC/INIT	Initialises the CAMAC & Seblocks
LOQ> CSET CAMAC/LOAD	Loads CAMAC software from FEM

and

LOQ> CSET CAMAC/STOP	Stops the CAMAC process on FEM
LOQ> CSET CAMAC/START	Starts a CAMAC process on FEM

You should never use the two commands in the second group because although the system will allow you to stop the CAMAC it will not allow you to restart it!

5.2 CSHOW commands
The format of the command is:

LOQ> CSHOW <Seblock>[/ENQUIRE][/FULL]

The /ENQUIRE qualifier forces the CAMAC to read the current setpoint of the associated device. If this qualifier is omitted then the value at the last logging interval (if the Seblock is logging) or the value last requested (if the Seblock cannot log) will be displayed. The /FULL qualifier gives more detailed information about the specified Seblock.

6. Identifying CAMAC problems (and how to rectify them)
The CAMAC is an aging system that does occasionally go wrong! A CAMAC fault cannot prevent data collection (since that is handled by a separate process) but it will affect the environment experienced by your sample (temperature setpoints may be lost, etc) and so being able to identify a CAMAC fault and being able to rectify it quickly is obviously advantageous!

If the message "No CAMAC data" is displayed in flashing reverse video in the CAMAC information area of the dashboard then the CAMAC has been stopped (by a CSET CAMAC/STOP, see Section 5.1.6) or it has crashed (this can happen after power dips or mains failures). If the CAMAC information area is empty then the CAMAC has been initialized (by a CSET CAMAC/INIT, see section 5.1.6. This command erases the area of memory used by the CAMAC process). Less serious, but harder to spot, is a "hung" CAMAC. This is most easily identified by one or both(!) of the following symptoms; a) the values returned by the temperature sensors have not changed for several logging intervals, or b) an enable logging command on a temperature Seblock gets stuck in a "MOVE" state.

6.1 Recovering from a hung CAMAC
Type the following command:

LOQ> CSET CAMAC/RESET

All of the Seblock's will go into a "FALS" state. It will then be necessary to enable logging on any temperature sensors in use and to re-issue any temperature setting commands (see Sections 5.1.1 and 5.1.3). Also see Section 7.

6.2 Recovering from a CAMAC Initialization
Type the following commands:

LOQ> @CAMAC$DIR:BUILD_CAMAC
LOQ> CSET CAMAC/RESET

After a minute or so, the Seblock's should re-appear in the CAMAC information area of the dashboard. It will be necessary to enable logging on any temperature sensors in use and to re-issue any temperature setting commands (see Sections 5.1.1 and 5.1.3). Also see Section 7.

6.3 Recovering from a CAMAC crash
First, go and "boot" the FALCON computer in the CAMAC crate, see Section 2.17 (this is the card on the right with two green and one red LED's labelled HYTEC 1050). The boot switch must be pulled out, flicked down and then released. Then type the following command:

LOQ> CSET CAMAC/LOAD

A block of text should appear telling you what is going to happen. If, instead, the message "Device is allocated to another user" appears then see Section 6.3.1. The load procedure will take about two or three minutes. Then issue:

LOQ> CSET CAMAC/RESET

After a minute or so, the Seblock's should re-appear in the CAMAC information area of the dashboard. It may be necessary for you to tell the CAMAC to display the Seblock's you were using or for you to remove other Seblock's from the display (see Section 5.1.5) but it will also be necessary to enable logging on any temperature sensors in use and to re-issue any temperature setting commands (see Sections 5.1.1 and 5.1.3). Also see Section 7.

6.3.1 "Device is allocated to another user"
This message means that the instrument computer is unable to establish communication with the FALCON computer. At this point it is best that you contact one of the instrument scientists. However, if they have gone to bed you could try re-booting the instrument computer (see Section 2.23). To do this, press in and release the button marked "RESTART". The LOQ workstation screen will go black and then messages will scroll up it for about ten minutes. A login window will then appear. Login with the username LOQ (the current password is usually on the whiteboard in the LOQ Cabin). After another few minutes three terminal windows will appear. Type STATUS ON in one of them. The message "No CAMAC Data" may continue to flash for several more minutes until the system is loaded. You should then proceed as outlined in Section 6.1.

7. Special note for users of the LAUDA Circulating Bath
A CAMAC reset command will set the bath setpoint to its default value of 25.0°C, so you must remember to re-issue a CSET LAUDA {temperature required}. In some instances it may be necessary to go and press the white reset button on the LAUDA R61 communications interface (on top of the bath itself) to re-establish communications. This latter situation can normally be identified by the fact that the LAUDA Seblock is stuck in "MOVE" whlist the rest of the CAMAC appears to function normally. Also ensure that the "PROG" button on the LAUDA is depressed (position 1) for computer control and that the thumbwheel temperature setting switches are set to 025.00.

Also see Pages 3-20 to 3-25.

CAMEX - CAMac EXtension

May 1996 (Revision 5)

Overview

CAMEX is the name for a collection of DCL command procedures and FORTRAN executable images which intercept and, where necessary, preprocess all CAMAC CSET and CSHOW commands on LOQ.

CAMEX looks at the Seblock (sample environment block) name in the command and decides whether that command should be processed by the CAMAC or by CAMEX. This flexible, and extendable, approach makes it possible to control equipment from the LOQ MicroVAX using "CAMAC-like" commands. This methodology means that the operation of CAMEX is essentially transparent from the operators perspective. It also provides continuity of command language and will facilitate any changeover of the equipment to CAMAC control at a future date.

Commands

The following CAMAC commands are implemented in CAMEX:

CSET CAMEX/START
CSET CAMEX/STOP

CSHOW CAMEX

CSET <Seblock> <Value>

CSET <Seblock> /RESET
/DISPLAY
/NODISPLAY
/HOME
/LOG[=<Setting>]
/NOLOG
/LOLIMIT=<Setting>
/HILIMIT=<Setting>
/RANGE=<Setting>
/VALUE=<Setting>

CSHOW <Seblock> /ENQUIRE
CSHOW <Seblock> /FULL

Some of these commands are implemented a little differently to their CAMAC counterparts and you are strongly advised to test them manually before using them in command procedures. In particular, the CAMEX CSHOW/ENQUIRE returns the current value of the Seblock and not its setpoint. Also, the CAMEX /LOLIMIT and /HILIMIT qualifiers specify "soft" limits and cannot exceed "hard" limits that have been coded into CAMEX (these have been set at the relevant physical limits of the equipment being controlled). Unlike CAMAC, CAMEX does not require that a /NOLOG command be issued before a /LOG=<Setting> command.

CAMEX And Log Files

Any CAMEX CSET <Seblock> <Value>, CSET/RESET, CSET/(NO)LOG or CSHOW/ENQUIRE command will result in a time-stamped entry being written to the current run log, INST_DATA:INST.LOG. This entry is similar, but not identical, to a normal CAMAC log entry:

Fri	30-SEP-1994	09:49:21	STOPD	CHANGER	TTA0:	999.9999	9.8500
Fri	30-SEP-1994	09:49:27	COMND	CHANGER	TTA0:	999.9999	999.9999
Fri	30-SEP-1994	09:50:32	COMND	HAAKE	TTA1:	18.9600	30.8900
Fri	30-SEP-1994	09:50:33	STOPD	CHANGER	TTA0:	999.9999	999.9999
Fri	30-SEP-1994	09:50:37	COMND	CHANGER	TTA0:	9.8500	9.8500
Fri	30-SEP-1994	09:50:38	STOPD	HAAKE	TTA1:	999.9999	999.9999
Fri	30-SEP-1994	09:51:41	COMND	CHANGER	TTA0:	9.8500	9.8500
Fri	30-SEP-1994	09:52:46	COMND	HAAKE	TTA1:	18.9700	30.8900

The CAMEX log entry shows the Seblock name, the logging status of the Seblock (COMND if logging is enabled, STOPD if logging is disabled or a command was issued), the MicroVAX terminal line communicating with the equipment and two real numbers. The first of these numbers is normally a "setpoint" whilst the other is normally a "current value", but there are exceptions:

Command	1st Value	2nd Value	Comment
CSET <Seblock> <Value>	Setpoint	Current	(Valid temperature or translator position)
	Requested	Current	(Invalid temperature requested)
	0.0	Current	(Translator "home" position requested)
	0.0	0.0	(If STOPD; Set zero requested or error) CSET
<Seblock> /RESET	0.0	999.9999	
CSET <Seblock> /(NO)LOG	999.9999	999.9999	
CSHOW <Seblock> /ENQUIRE	999.9999	Current	

Input Values
/LOG commands require an interval in seconds. Specify temperature commands in °C and position commands in mm or by a logical of the form <Seblock>_X; ie, CSET <Seblock> X. Such logical names are held in the normal CAMAC logical name table CAMAC_TABLE and are defined in SAMPLE_CHANGER.COM in the normal manner.

Equipment Controlled By CAMEX
At the time of writing three pieces of LOQ sample environment equipment are under CAMEX control. These are the large, floor-standing, Haake Circulating Fluid Bath (via the Haake IF 24-1 communications interface), the Ealing Electro-Optics 600mm Translation Stage (via the Ealing DPS - Digital Positioning System - Controller) and the Goudsmit Electromagnet (via the Danfysik System 8000 Controller).

As a result of the introduction of CAMEX the command verb HAAKE, previously used to control the Haake Bath, is withdrawn from use with immediate effect.

Miscellaneous Information
It is possible to "talk" to a CAMEX device directly in using the DCL command SET HOST/DTE TTAn: To use this method you will need to know the ASCII command set for the relevant device. These mnemonics will be found in the device instruction manual.

CSET CHANGER/HOME - This command causes the sample changer to seek its magnetic reference position (approximately at the midpoint of the stage) and to do so at $^1/_{20}$ of normal speed. After this command has executed issue a /RESET command followed by a /VALUE command to define the absolute value of the reference position. A command file of CAMEX commands to align the stage on its reference point will be found in LOQ$DUA0:[LOQ]SETUP_CHANGER.COM.

CSET CHANGER HOME - This command moves the sample changer to the position last defined by a CSET CHANGER/VALUE=<Setting> command.

Known "Features"
You might sometimes get messages like:

%CAMEX-I-TIMEOUT, Timeout waiting for response from controller.

This is an information message. It arises because a device controller does not handshake to the LOQ FEM properly and so the only foolproof means of ensuring that the controller is ready is to wait for it to timeout.

%CAMEX-E-NOASSGN, Unable to assign an I/O channel to device TTA0:
%CAMEX-F-ISINUSE, Interface in communication with another process at...

These messages mean that your CAMEX command cannot be sent to the device because another process issuing CAMEX commands is already in communication with the same device. In such instances, wait a few seconds and then try your command again. If this error persists then contact the author (SMK). If he is not available you could try (in sequence):

(a) stopping the CAMEX log process with CSET CAMEX/STOP,
(b) cycling the power on the device interfaces (as opposed to the devices themselves), or
(c) as a very last resort, reboot (not switch off!) the LOQ FEM.

Note that in cases (b) and (c) you will need to re-establish current device setpoints.

%CAMEX-E-UNRECOG, Unrecognised response from interface...
%CAMEX-E-UNRECOG, Unrecognised response from controller...

These messages mean that the program does not understand the characters it just received from the device it is connected to. Since a device only sends data back to the LOQ FEM when a specific request is made (ie, a CSHOW command is issued) or to signify completion of an operation, these messages present no cause for alarm.

Technical Information For LOQ Instrument Scientists, ISIS System Managers & Fans of VMS!

To obtain the Seblock names CAMEX translates the process logical names:

SAMPLE_CHANGER_SEBLOCK	(normally CHANGER)
WATER_BATH_SEBLOCK	(normally HAAKE)
MAGNET_SEBLOCK	(normally MAGNET)

To obtain the names of the MicroVAX terminal lines communicating with the equipment CAMEX translates the process logical names:

CHANGER_COMMS_CHANNEL	(normally TTA0:)
BATH_COMMS_CHANNEL	(normally TTA1:)
MAGNET_COMMS_CHANNEL	(normally TTA1:)

These terminal lines are configured by CAMAC_SET.COM during CAMAC initialisation and are thus unaffected by reboots or crashes of the LOQ MicroVAX.

The CAMAC CSET and CSHOW commands are intercepted by defining the following DCL foreign symbols:

```
CSET   :==     $CAMEX_DIR:INTERCEPT_CSET
CSHOW:==       $CAMEX_DIR:INTERCEPT_CSHOW
```

INTERCEPT_CSET and INTERCEPT_CSHOW then call a routine which extracts the remainder of the command line using LIB$GET_FOREIGN. If the Seblock is not one of those specified by the logical names above then the command is treated as a CAMAC command, otherwise it is parsed against the CLD file CAMEX_TABLE using CLI$DCL_PARSE and an appropriate routine called to action the command.

Communication with the equipment is in the form of ASCII strings using normal FORTRAN READ and WRITE statements in the case of the Haake Bath but SYS$QIOW routines in the case of the Ealing Translator and Goudsmit Electromagnet (to overcome handshake synchronisation problems and timeouts).

Logging is handled by CAMEX_LOG using the CAMAC WRITE_LOG subroutine. This runs as the detached process "Log Process 2" which is created automatically during CAMAC initialisation and will thus restart after a reboot or crash of the LOQ MicroVAX. Should this process crash for some reason it may be restarted by issuing the command CSET CAMEX/START. This command can only be executed by an account with LOQ_MANAGER process rights (eg, LOQMGR). As with its CAMAC counterpart, the command files which control this process reside in CAMAC$DIR. The error file associated with the process is also written to this directory. To stop the "Log Process 2" use CSET CAMEX/STOP.

CAMEX permits logging on the sample translator Seblock (eg; CHANGER). No ill-effects have been experienced - yet! Enabling logging on a CAMAC sample translator Seblock (eg; SAMPLE) is of course possible, though extremely unwise - it will hang the CAMAC system!

The CAMEX log process also provides a link to FREEDASH process which generates and updates the familiar instrument dashboard. This link is in the form of three logicals held in the logical name table CAMAC_TABLE. These logicals are:

CHANGER_DISPLAY
BATH_DISPLAY
MAGNET_DISPLAY

The form of these logicals is {Seblock}_{LOGG or FALS}_xxxxxx_{Y or N} where LOGG and FALS indicate whether logging of the Seblock is enabled or not, xxxxxx is a FORTRAN i6.6 representation of the value to be displayed (divide by 100 to get the actual value) and Y and N indicate whether or not the information is to be displayed by the dashboard. These logicals are translated and converted into a "dashboard format" by the routines in the module SMK_MOD which must be linked into FREEDASH.

If these logicals are not set, for example, if the system reboots whilst logging is disabled (FALS) then the dashboard will display "No CAMEX data" (assuming that a /DISPLAY command has been issued).

The date and time when the _DISPLAY logicals were last updated is held by two other logicals which are also in CAMAC_TABLE. These are:

CHANGER_UPDATED
BATH_UPDATED
MAGNET_UPDATED

The FREEDASH process also includes a variable called MAX_INTERVAL. This is currently set to 60 seconds. If the last update of a _DISPLAY logical was made any earlier than this then the dashboard will display "Logging failed" (assuming that a /DISPLAY command has been issued). CAMEX_LOG updates the _DISPLAY logicals on a regular basis irrespective of any logging interval specified by a /LOG command. This interval is determined by the variable DEADTIME in CAMEX_COMMON.INC and is currently also set at 60 seconds.

SMK_MOD also contains a routine called TRANSLATE_ENQUIRE_LOGICAL which translates the logical name CSHOW_LAST_VAL, held in the logical name table CAMAC_TABLE, which is created by the CAMAC CSHOW process. This logical is updated whenever a CAMAC CSHOW/ENQUIRE command is executed. CAMEX_LOG periodically spawns CSHOW TRANS/ENQUIRE commands so that FREEDASH can obtain and update the status of the transmission monitor and beam attenuator using TRANSLATE_ENQUIRE_LOGICAL. Although the displayed status of the monitor and attenuator are refreshed at the normal dashboard interval (typically every 5 seconds), the actual status can only change after polling by CAMEX_LOG (typically every minute as explained above). Hence, should "Log Process 2" crash these status indicators will be out-of-date.

Notes

Appendix 1

LOQ Risk Assessment

Risk Assessment for the LOQ Diffractometer
(Part of ISIS, Building R55 at RAL)

Introduction
The LOQ diffractometer uses cold neutrons diffracted at small angles to examine the structure of many types of materials. This document relates to the layout of the instrument as rebuilt in September 1995 with a shielded enclosure for a new scintillator detector at 0.5 m from the sample, in addition to the existing two dimensional ^3He gas detector at 4.3 m.

LOQ is operated by visiting experimental scientists from the UK and overseas, who are trained by the in-house CCLRC instrument scientists. A written system of work and user registration ensures that visiting scientists are aware of hazards and issued with radiation dosimeter badges (see ISIS/SI/20).

The primary hazard is the neutron beam which at the sample has a maximum dimension of 25 mm diameter (though is normally around 10 mm) and a cold neutron flux of no more than $\sim 3x10^6$ cm^{-2} s^{-1}. The maximum dose rate in the beam is estimated to be no more than ~20 mSv hr^{-1}. (Note that the annual dose limit for a body organ or extremity is 500 mSv, for adult males, except for the eye, see details in ISIS Safety Note 2).

All reasonably practical steps have been taken to prevent access to areas with radiation when beam is being delivered. All access gates to radiation areas and removable shielding above the chopper at the front of the beam line are interlocked.

Contingency plans allow the ISIS main control room (MCR) staff to deal with emergencies such as fire, radiation leak or other accidents.

Additional hazards are a 1.0 mW laser used for sample alignment and vacuum window hazards in the "sample pit" and "high angle detector bank" areas.

General hazards
Potential hazards, common to all ISIS beam lines, are the presence of high voltage cables; the use of sample environment equipment and a pillar crane; and the radiation, chemical and toxic hazards of samples themselves. These are considered in detail by the ISIS "local rule" safety instructions, which are introduced in ISIS "Local Rules", LR/22 , and further detailed in the Neutron Beam Operating Instructions - the ISIS/NBOI - series of documents. A 0.8 ton, 2 Tesla, Goudsmit electromagnet and power supply, for use on CRISP & LOQ, has its own safety assessment available from the instrument scientists.

All samples and any sample equipment placed in the neutron beam should be covered by a "Sample Record Sheet", or SRS, which gives a hazard assessment as required under COSHH, Control Of Substances Hazardous to Health, regulations. The SRS should have been supplied in advance by the experimentalists for assessment by ISIS safety personnel (ISIS SI/29). A copy of the SRS should be placed in the holder on the gate to the sample pit. In an emergency the MCR crew, or others, will need to ascertain from the SRS the nature of any hazards present. Procedures carried out in the ISIS sample preparation laboratories may require separate safety assessments. Equipment and materials temporarily left on benches, etc, should be appropriately labelled. External users are supplied with relevant safety information on their first visit to ISIS and are required to have watched an ISIS safety video.

Avoidance of High Radiation Exposure

The potential dose rates in the neutron beam on LOQ represent a modest radiological hazard.

Radiation induced in samples by the neutron beam is generally not a hazard on LOQ. Certain elements are however strongly activated and will be noted on the SRS. All samples should be monitored with the supplied portable monitoring equipment before removal from the sample pit or sample environment apparatus. ISIS LR/22 and several ISIS visitor safety notes, as well as the ISIS safety video deal with the handling, storage and logging of radioactive samples.

Steel, borated wax, and other shielding materials reduce radiation in accessible areas external to the beam line to less than 2 $\mu Sv\ hr^{-1}$ when the shutter is open. If surveys show higher radiation levels then barriers and warning notices are erected.

A standard ISIS beam shutter isolates the instrument from the neutron source. With the shutter closed the dose rate in the sample and high angle bank enclosures is less than that on top of the beam line.

Access to the sample pit and high angle bank area is controlled via an electro-mechanical interlock system, such that the access gate cannot be opened unless the main shutter is closed. The interlock system ensures that once access is gained, the shutter cannot be opened again until the access doors are closed and the interlocks remade.

Removal of shielding or structural changes to the beam line are controlled by a permit to work system, described below.

A "super mirror bender" device, located in the target station wall, deflects the cold neutrons used on LOQ through an angle of 24 mrad. This effectively removes all fast neutrons from the beam arriving at the sample position. The remaining slow neutron beam is completely adsorbed by a cadmium beam stop, 2 mm thick, in front of the main detector. (The, typically 30 cm, of borated wax shielding around the sample area and detector vacuum tank is primarily to keep out external radiation).

The ISIS MCR crew test interlocks at the beginning of each experimental period to verify correct operation of the system.

Interlock System

Access to the sample area is restricted by a fence with an access gate which is locked by a Fortress interlock bolt.

The master key which is required to release all other interlock keys is mechanically trapped in a 'green solenoid box'. It can only be removed when an electrical solenoid is energised after the main shutter is closed. On rotating the master key to remove it from the green box an electrical switch prevents the beam shutter from being opened. Any attempt to open the beam shutter before the interlocks are made, or any malfunction, causes the ISIS accelerator to trip off (removing the majority of the radiation). A radiation monitor in the chopper enclosure provides an additional interlock against failure of the primary system.

When the interlock chain is enabled prior to and when the shutter is open, blue lamps are visible in the sample area, high angle bank "cave" and fenced area. A red "Beam on" lamp adjacent to the access gate is also illuminated. Lamps indicating the interlock status are visible from the access gate. Emergency "beam-off" push-buttons are provided in the sample and high-angle bank enclosures. These will trip the accelerator should someone be trapped inside.

Permit-to-work

Removable shielding blocks above the chopper pit have their crane lifting points normally locked by keys held in the Main Control Room key-press. The shielding blocks are additionally connected to the beam line interlocks by a Fortress interlock key and bolt. They may only be removed after an authorised person (ISIS NBOI/9) has obtaining a "beamline permit-to-work" (see ISIS/SI/28) from the control room staff, who will first lock the beam line shutter in the closed position. The permit is only released upon the signature of whoever is responsible for raising it, that the shielding has been reinstated. If necessary a radiation survey will also be conducted.

Vacuum and other access hazards

The 3.6 m long vacuum tank varies in diameter from 150 mm at its 5 mm thick, quartz glass, entrance window to 900 mm before the main detector. A test blank of the glass window survived a 3 atm pressure differential, but is not considered to be within normal engineering safety margins. Due to the large amount of stored energy represented by a vacuum tank of this size, operators (experimentalists) must be protected from spontaneous or accidental breakage of the window:

(a) Sample area. This is the only area normally accessed by operators. A steel shutter, mounted externally on the shielding wall between the nose of the vacuum tank and the sample area, will protect the operators from breakage of the vacuum window. The presence of the shutter itself makes breakage of the window by other objects much less likely. The shutter is operated by a contact in the "green box" mentioned in the interlock section above, such that it will go down as soon as the master key has been removed from the box. The shutter is actuated by a guaranteed compressed air supply and a buffer air reservoir tank (which is a registered pressure vessel, subject to regular inspection).

The sample area is reached by a ladder, some 1.7 m in height, which is the only practical means of access. Operators are instructed to take care and not to attempt to carry anything whilst climbing up or down. Sample environment equipment is normally installed or removed using a pillar crane, which may only be operated by those with an appropriate crane drivers licence.

(b) High angle detector "cave" enclosure. This is a region around the nose cone of the vacuum tank, connected by a circa 600 mm square hole through a 300 mm thick shielding wall to the sample area. Access to the enclosure is through a hinged door in the shielding adjacent to CRISP. Once the high angle detector is installed, the "cave" will be barely large enough to contain a person. The object of access will be to replace detector components or cables, or to adjust temporary shielding. Warning notices will restrict entry to instrument scientists or detector group personnel familiar with the equipment. A second person should either be close by or checking frequently to avoid an injured person remaining undetected. All EHT supplies (~1 kV) to the detector photomultiplier tubes will be protected by current trips (as is standard on LOQ's LeCroy EHT supply).

The adjacent area between CRISP and LOQ, providing access to the "cave", has an interlocked door, requiring two Fortress keys to open. The first key is one of the normal radiation protection interlock keys. The second key is held by a solenoid, which will only be released when a pressure operated switch on the vacuum system is close to atmospheric pressure. Removing this key from the solenoid also breaks the starter circuit to the vacuum pump, preventing the tank from being re-evacuated whilst the interlocked door is open. The high-angle detector enclosure is thus not accessible unless the vacuum window hazard has been removed.

To close the interlocked door a "search button" must first be activated within the detector enclosure. Closing the shielding door will activate a micro-switch to start a "search buzzer" which gives a fixed time in which to "search" the area and close the exit door. A "panic off button" and blue "beam on" light are included within the detector enclosure. A yellow "vacuum on" light and red "beam on" light are displayed outside the interlocked door.

(c) Main detector. The shielding around the rear of the main detector sits on a trolley on rails, so that it may be rolled back to expose the detector. An electrically powered actuator moves this trolley. To operate this a standard radiation interlock key releases both a sliding bolt that locks the shielding closed and also an isolator key. To operate the actuator motor the isolator key must be placed in a control panel to provide power to non-latching switches which move the trolley either in or out. The actuator key should then be removed and placed in the pocket of someone working inside the shielding to prevent the trolley being moved whilst they are in front of it. Access is normally only required by instrument scientists for maintenance of the beam stop mechanism or to insert a calibration mask inside the vacuum tank, in front of the detector. (Note that the cadmium beam stop and/or the detector itself adsorb all radiation from the sample, the shielding here is primarily to adsorb external radiation, though it also forms a useful mechanical barrier to a possible radiation source, in place of a fence. The detector itself may only be rolled back from the vacuum tank flange by about 120 mm, so does not provide a significant access route to a radiation source).

The active area of the main detector is 650 mm square and has a 12.5 mm thick aluminium alloy window to contain 1.2 atm gas pressure. This was verified as adequate by RAL engineers on purchase of the detector in 1991. Though its vacuum flange bolts may be removed after rolling back the shielding trolley, the detector would only come away from the tank on admitting air due to the large force on such a diameter.

Alignment Laser
Within the sample area there is a 1.0 mW helium-neon laser for sample alignment. The installation is in accordance with current regulations for Class II lasers, and the appropriate warning notices are displayed.

Appendix 2

Further Information

Other Information

The following additional information is available...

... on LOQ (please contact the instrument scientists):

1. *"Development of the Small-Angle Diffractometer LOQ at the ISIS Pulsed Neutron Source"*, R K Heenan & S M King, Proceedings of the International Seminar on Structural Investigations at Pulsed Neutron Sources, Dubna, Russia, September 1992, Publication E3-93-65, JINR, Dubna, 1993, V L Aksenov, A M Balagurov & Yu V Taran (editors).

2. *"Small-Angle Neutron Scattering from Non-Crystalline Materials on Pulsed Neutron Sources"*, R K Heenan & S M King, Proceedings of the NCM5 Conference held in Sendai, Japan, August 1991, J. Non-Cryst. Solids, (1992), 150, 153.

3. *"The Effects of Inelastic Scattering on Small-Angle Diffraction Measurements"*, R K Heenan & A R Rennie.

4. *"Using COLETTE (A Simple Guide)"*, S M King, June 1996.

5. *"Recent Changes to COLETTE"*, R K Heenan, November 1994.

... on ISIS (please contact the ISIS University Liaison Secretariat):

6. *"ISIS User Guide"*, B C Boland & S Whapham, Report RAL-92-041, 1992.

7. *"Directory of UK Users"*.

8. *"ISIS Safety Video"*.

9. *"Changing a Sample on ISIS Instruments"*, J Chauhan, A V Belushkin & J Tomkinson, Report RAL-93-006, 1993.

10. *"Use of Cryogenic Liquids on ISIS Instruments"*, J Chauhan, A V Belushkin & J Tomkinson, Report RAL-92-041, 1992.

... on the ISIS Computer System (please contact ISIS Computer Support):

11. *"PuNCH Manual MiniGuide 1"*.

***ISIS Proposal Forms* and, for UK users, *Claims Forms* for travel and subsistence are available from the ISIS University Liaison Secretariat.**