Background

4-6 cts/min. during scans from weak incoherently scattering samples

[actively being reduced to (we hope) < 1 ct/min].

Range of λ , Q, ω

Present incident wavelength λ_i = 2.32 Å (PG002); = 1.62 Å Al(111), 1.40 Å Al(002), 0.99 Å Al(220); and 0.84 Å Al(113). From fixed

 $2\theta_{\rm M} = 40.5^{\circ}$.

Future $\lambda_{\hat{i}} = 0.8 \text{ Å} \rightarrow 3.8 \text{ Å}$

Momentum transfer 0.5 \mathring{A}^{-1} to 12 \mathring{A}^{-1}

Energy transfer up to ~ 60 meV (neutron energy loss)

Energy resolution typically $\sim 5\%$.

Specimen Height

10" or 71/2" from table.

Motors

Printed circuit D.C. motors (simultaneous positioning).

Digitisers

Moore-Reid contact type.

Control System

PDP8A

CAMAC Interface.

Software Programs

Real space or reciprocal space scan input parameters.

Scan mode: linear step scan: up to 64 points per scan

up to 84 scans.

Input: DECwriter or teletype keyboard, or paper tape, or buffered on

FLOPPY DISC.

User Programs: LINK, DAIS, DAID, SIRS, LIM, LINT, PRIN, MANU.

[It is hoped to add automatic temperature control in the near future].

Data Output

DECwriter or FLOPPY DISC

Harwell 6000 Series Units for visual display.

Ancillary Equipment

Variable temperature cryostats 4.5 K - 293 K.

Furnace 293 K to 1300 K.

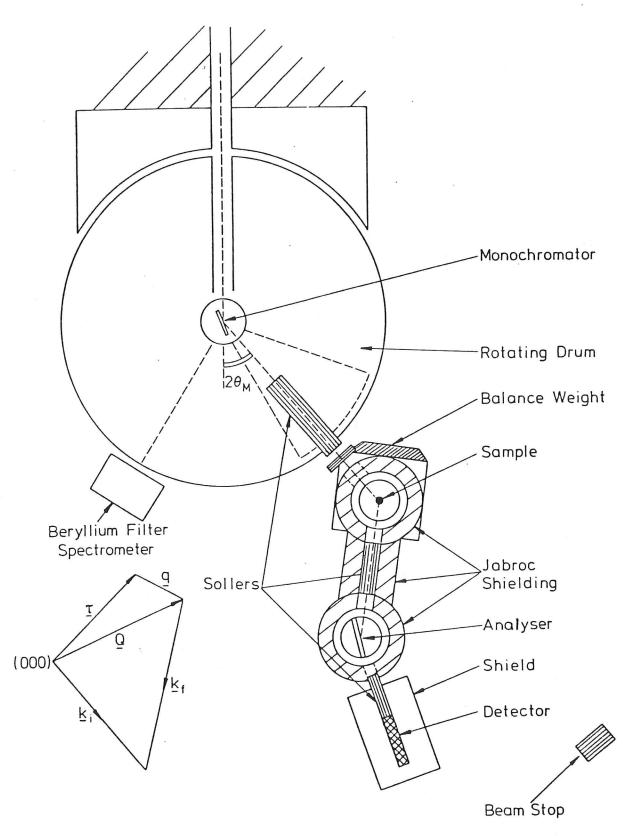
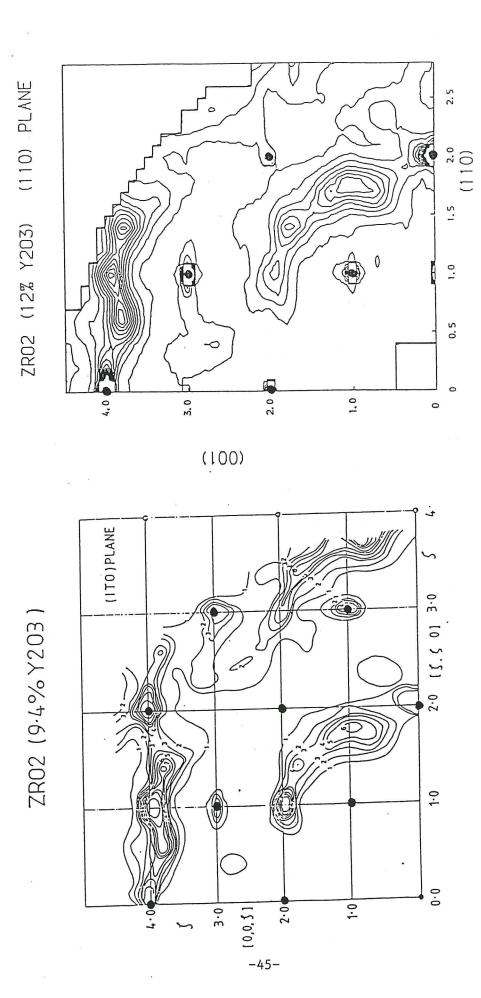


Figure 18.
Schematic drawing of DIDO triple axis (10H facility)



Figure 19.

DIDO 3-axis Spectrometer showing arrangement of detector shield, analyser table and sample table. A variable temperature cryostat is shown mounted on the sample table



Contour diagrams of the diffuse elastic intensity (in arbitrary units) at room temperature in the $(1\overline{1}0)$ plane of ZrO_2 (x mol% Y_2O_3) for x = 9.4, 12. obtained using the DIDO 3-axis spectrometer. Figure 20.

PLUTO Triple-Axis Spectrometer

Uses

Scans of $\hbar\omega$ may be made by varying either E_i or E_f . The resolution and energy range can be altered quickly by changing the monochromator or analyser from a wide variety of available crystals, by changing the particular plane of the crystal used, or by changing the soller collimators. The parameters which can be changed automatically by the software programs include monochromator or analyser planes from an aligned crystal.

Description

The incident energy is continuously variable by automatic variation of the take-off angle from the monochromator, the shielding drum rotating around the monochromator with sectors being automatically raised to allow passage of the beam from the reactor. The monochromator may be changed in approximately 1½ hours, including alignment, by access via the shielding sectors. The detector is surrounded by a 24" diameter shield which is raised from a large steel base floor by three air pads during movement of the machine. A system of boral shielding rings surrounds the sample and analyser table to reduce background. The soller slit assemblies locate on fixed keyways for quick interchange. The instrument is controlled from a sound-proof air-conditioned room.

For further information see:

M.J. Cooper, J.W. Hall and M.T. Hutchings, J. Appl. Cryst. 9, 444 (1976).

J.M. Milne, AERE - R 8309 (1978).

PLUTO Triple-Axis Instrument Details

Angular Ranges (All computerised)

Monochromator take-off angle

20° to 113°

Specimen - analyser angle

-109° to +109°

Analyser - detector angle

-95° to +95°

M, S, A crystal setting angles

 -320° to $+320^{\circ}$

Mechanical and software limits may be used to define range used.

Collimation

In-pile

0.8° or 1.7°

Monochromator - sample

0.22°, 0.33°, 0.5°, 1° and 2° (standard)

Sample - analyser

0.33°, 0.67°, 1° and 2° (standard); 0.5°, 0.67° and 0.83° (short)

Analyser - detector

0.33°, 0.67°, 1° and 2°.

NB. The sollers in the last three positions may be arranged to define vertical collimation.

Max. Beam Size

 $50~\text{mm}\times50~\text{mm}$

Detector

 $2^{"}$ diameter end window BF_3 , or He^3 , counter

Monitor

Low efficiency fission chamber.

Monochromators

(R = reflection, T = transmission)

Aluminium:-

planes $\perp [1\overline{1}0]T$.

Germanium:-

planes $\perp [1\overline{1}0]T$.

Pyrolytic Graphite:-

(002)R, (004)R etc. 0.4° or 0.8° mosaic (Curving about a horizontal axis will be

available shortly)

Copper:-

(111)R, (220)T

Beryllium:-

(002)R, (110)T

Analysers

As above plus

Zinc:-

(002)R, (110)T

Filters

 $2'' \times 2'' \times 2''$ Graphite, mosaic 3.5 ± 1.5° (before or after sample).

A cooled beryllium filter may be placed before or after

the sample.

NB. Use after sample necessitates use of short soller.

Absorbers/Masks

Polythene absorbers 1/8" to 2" thick.

Cadmium masks 1/2" to 11/2", horizontal and vertical aperture.

Flux at Monitor

 4×10^6 n cm⁻² s⁻¹ from Aluminium (111), $\lambda = 1.2 \text{ Å}$

 1.4×10^7 n cm⁻² s⁻¹ from Pyrolytic Graphite (002), $\lambda = 1.2 \text{ Å}$

Background

0.3-1.5 cts/min. during a typical scan from a low incoherently scattering

sample.

Range of λ , Q, ω

Incident wavelength: 0.8 to 5 Å

Momentum transfer: 0.5 to 12 Å^{-1}

Energy transfer up to ~90 meV extreme limit,

 \sim 60 meV usual limit (neutron energy loss)

Energy resolution typically $\sim 5\%$.

Specimen Height

10" or 7% above tables

150 kg maximum load a table.

Motors

(Simultaneous positioning)

Ironless armature D.C. motor.

Digitisers

Moore-Reid contact type driven by anti-backlash gearing from the shaft.

Control System

Computer controlled or manual operation.

PDP8

CAMAC interface

Software Programs

Real space or reciprocal space scan input parameters.

Scan mode:-

linear step scan: up to 64 points per scan, up to 84 scans.

Input:-

DECwriter or teletype keyboard, or paper tape, or buffered

on DECtape.

User Programs:- LINK, DAIS, DAID, SIRS, BEFS, LIM, LINT, PRIN, MANU.

Data Output

DECwriter and DECtape.

Automatic time, temperature print out.

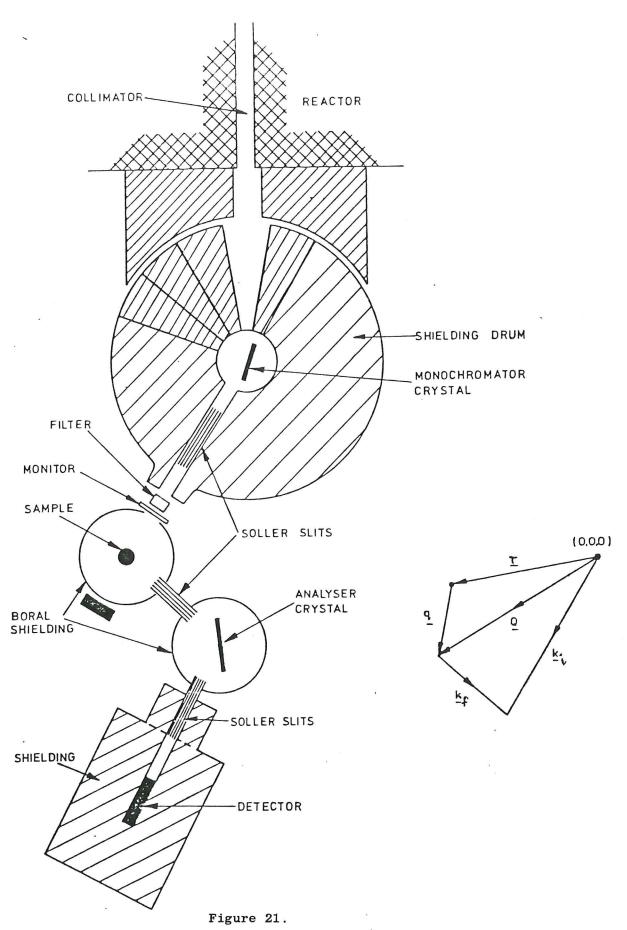
Harwell 6000 Series units for visual display.

Ancillary Equipment

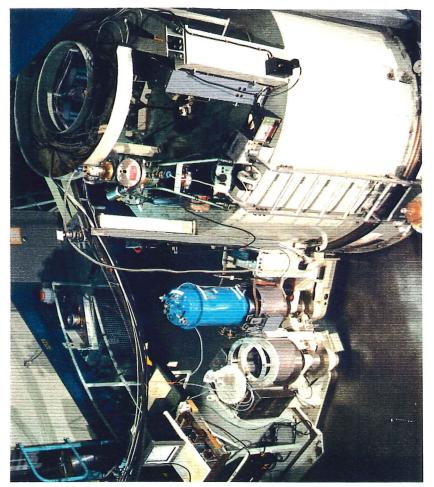
Variable temperature cryostat: 4.5 K to 293 K.

Furnace 293 K to 1300 K.

Superconducting magnets to 6.0 Tesla.



Schematic layout of PLUTO triple axis spectrometer



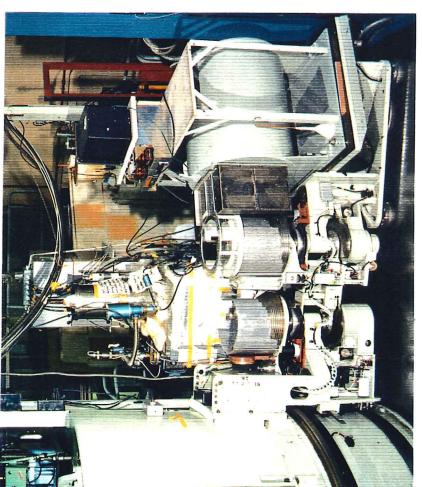


Figure 23.

General view of the PLUTO 3-axis Spectrometer with a cryostat mounted on the sample table

The PLUTO 3-axis Spectrometer showing the arrangement of Sample and Analyser tables

Figure 22.

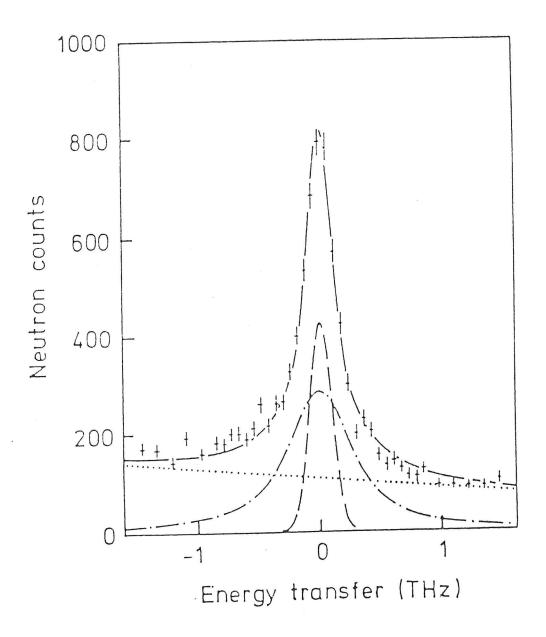


Figure 24.

Energy scan of quasielastic diffuse intensity due to vacancy mobility at the (1.7, 1.7, 1.0) point at 2700K from a single crystal of 18 mole% $\rm Y_2O_3/ZrO_2$; obtained using the PLUTO 3-axis spectrometer. Solid State Ionics 28.30 488 (1988)

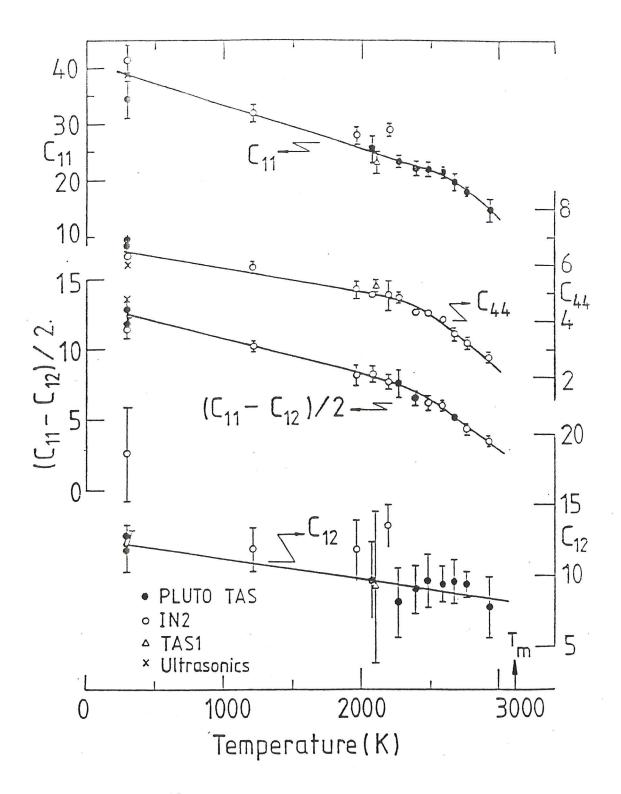


Figure 25.

Elastic constants of ${\rm UO}_2$ at temperatures up to 3000K obtained from the slope of the acoustic phonon branches measured using the PLUTO 3-axis spectrometer.

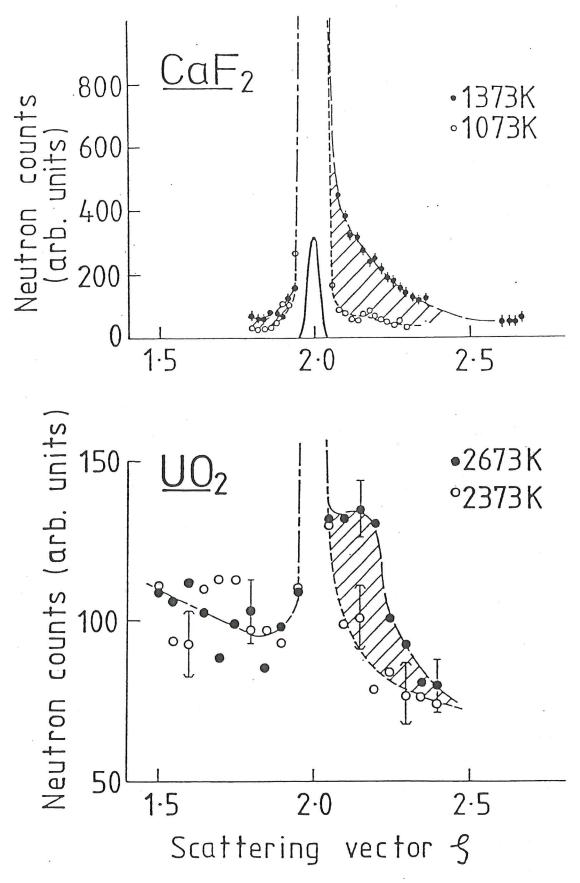


Figure 26.

The first observation of diffuse scattering from $\rm UO_2$ at very high temperature due to thermally induced oxygen lattice disorder. Obtained using the PLUTO 3-axis spectrometer. Phys Rev Letters $\underline{52}$, 1238 (1984).

Uses

The MARX spectrometer has two principal modes of operation which combine the speed of data collection of a time-of-flight spectrometer with the positional versatility of a triple-axis. In the first of these the use is in the investigation of quasielastic scattering (inelastic scattering centred on zero energy transfer) to determine diffusion rates and relaxation times. The scans in (Q,ω) space are similar to but not so restricted as those made by time-of-flight spectrometers, and can be arranged to give almost constant |Q|. The best resolution attainable is $\sim 130 - 150 \,\mu\text{V}$ and scattering at wavevectors out to 2.3 Å⁻¹ can be examined. The resolution may be broadened and wavevector range increased by simple changes. The second mode of operation is to use the spectrometer to investigate excitations in materials in a similar way to a triple-axis spectrometer. Because of the large range of Q and ω examined at one setting of the machine, the data collection rate is higher, and a scan of only five or so points should enable a phonon to be accurately located along a desired direction of Q. The sample background is measured simultaneously and can therefore be accurately subtracted.

The Position Sensitive Detector (P.S.D.) may be used to examine diffraction from a sample, if the analyser is removed. The spectrometer may also be converted to a conventional triple-axis machine with uses listed under the PLUTO or DIDO triple-axis.

Description

The MARX spectrometer is similar in construction to a triple-axis spectrometer but the use of a large dimension analyser crystal, no soller collimation between sample and detector, and a long position-sensitive detector, enables neutrons with a range of final wavevector and energies to be detected simultaneously. Two such rays from the sample are shown in the figure, and the locus of wavevector transfer \underline{Q} lies on the perpendicular bisector of the negative analyser tau vector.

The incident neutron energy is constant, but its value is manually variable by changing the takeoff angle $2\theta_{\rm M}$ from the monochromator, or by changing the monochromator crystal which is readily
accessible via an insertable "coffin". It is intended to automate the monochromator ω -angle in the
near future. The sample table, analyser and detector move on air bearings over a resin base floor. The
distance between the sample and analyser can be varied manually to change the resolution of the instrument. Surrounding the sample and analyser is a system of shielding which automatically adjusts with
the movement of the machine. [An improved shield which allows greater movement and the use of larger
cryostats will replace the present system in the near future.] The spectrometer can be converted to a
conventional triple-axis by the use of an end-on He³ counter, additional shielding in front of the P.S.D.,
and the addition of soller collimators before and after the analyser.

Recently the spectrometer has been used in a Double Back Scattering Diffraction (DBSD) mode, to observe diffraction at very high resolution.

MARX Instrument Details

Angular Ranges and Distances

Monochromator take-off angle -32° - 84.5°; manually variable in 1.0° steps

Specimen – analyser angle -100° to $+100^{\circ}$ dependent on configuration

Analyser – detector angle -100° to $+100^{\circ}$

M, S, A crystal setting angles -320° to $+320^{\circ}$

Specimen - analyser distance 470 - 850 mm manually variable

Analyser – detector distance 780 mm

Mechanical and software limits may be used to define angular range used.

Collimation

In-pile

Monochromator - sample

MARX
Sample - analyser

Analyser - detector

Open

Open

One

Open

Open

Open

Open

Open

Open

(NB. S-A 0.33° or 0.66° etc. and A-D 1.0°, 2.0° etc. in CONVENTIONAL 3-AXIS MODE).

Max. Beam Size

 $50 \text{ mm} \times 50 \text{ mm}$

Detector

50 mm diameter \times 500 mm active length

4 atmos. He³ position-sensitive.

Analogue to digital conversion of signal.

Typically at 4 Å and 6 channels/cm, each channel subtends 3.5' at S

and corresponds to $10 \mu V$ in energy.

Monitor

Low efficiency fission chamber.

Monochromators

(R = Reflection; T = Transmission).

Pyrolytic Graphite: (002)R, (004)R etc.

Aluminium:

Planes \perp to $[1\overline{1}0]T$.

(NB. Monochromators must have high transmission to avoid affecting the long-wavelength diffractometer beam).

Analysers

 $14'' \times 2'' \times \frac{1}{6}''$ Pyrolytic Graphite (002)R, (004)R etc. - MARX MODE

3.5" \times 2" \times $\frac{1}{16}$ " Pyrolytic Graphite (002)R - CONVENTIONAL 3-AXIS MODE

Other crystals are available

if required.

Filters

Cooled 150 mm beryllium filter before sample.

Pyrolytic Graphite ($1\frac{3}{4}$ " × 2" × 2"), 3.5 ± 1.5° mosaic, before sample.

Absorbers/Masks

Polythene absorbers of various thicknesses available.

Cadmium masks define beam size between 1/2" - 2", vertically and

horizontally.

Flux at Specimen

 2.8×10^5 n cm⁻² s⁻¹ at ~ 4 Å. (Be-filtered beam).

Background

~25 cts/min - no sample, summed over all channels P.S.D., MARX-MODE.

1.5 cts/min - 3-axis counter in CONVENTIONAL 3-axis MODE.

Range of λ_i, Q, ω

MARX Quasielastic: Typically at λ_i = 4.1 Å, Resolution \sim 150 μ V, Momentum transfer \sim 0.3 Å to 2.3 Å⁻¹

Inelastic:

Energy transfer 0.3 to ~ 60 meV (neutron energy loss)

Momentum transfer 0.5 Å^{-1} to 7 Å^{-1}

Energy resolution $\sim 3\%$.

CONVENTIONAL 3-Axis mode – see PLUTO 3-axis for comparable figures.

Specimen Height

25 cm or 18.6 cm above table.

Motors

Printed circuit D.C. motors (simultaneous positioning).

Digitisers

Moore-Reid Contact Digitisers.

Control System

PDP8E computer

CAMAC interface.

Software Programs

Real space or reciprocal space scan input parameters.

Scan mode (MARX) up to 2 points per scan.

up to 84 scans.

(3-AXIS) up to 64 points per scan.

Input:

Keyboard or teletype, paper tape or buffered on DEC tape.

User Programs: MARX, DAIS, DAID, DAIT, SIRS, LIM, LINT, PRIN, MANU.

Data Output

High speed printer or teletype.

DEC tape.

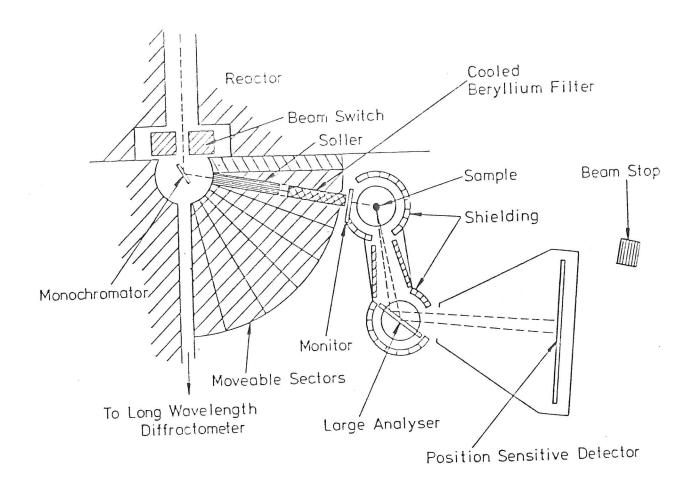
VDU for visual display of P.S.D.

Harwell 6000 Series Units for visual display of total counts.

Ancillary Equipment

Cryostats 4.5 K - 293 K

Furnaces 293 K - 1300 K



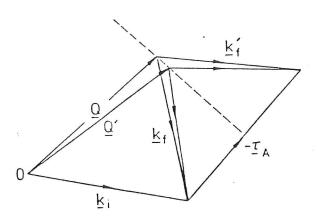


Figure 27.

Schematic diagram of PLUTO MARX spectrometer

Figure 29.

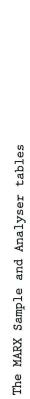
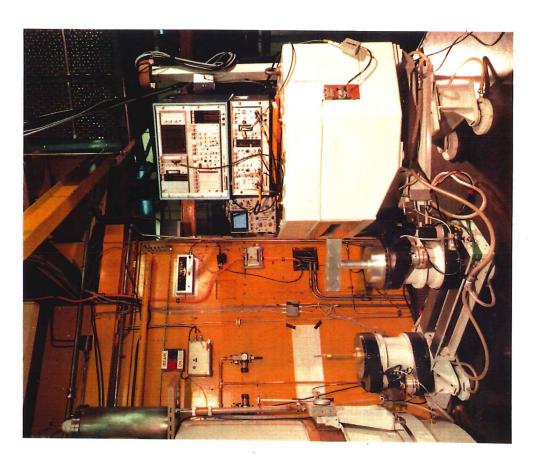
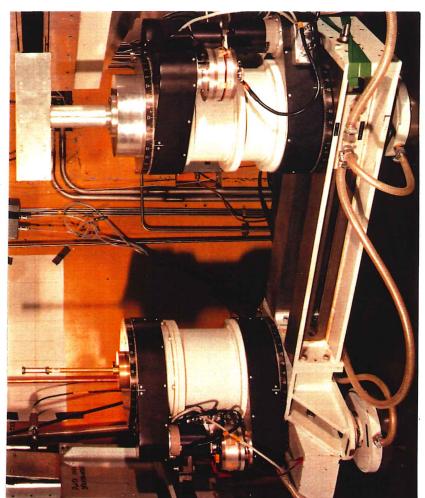


Figure 28.





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