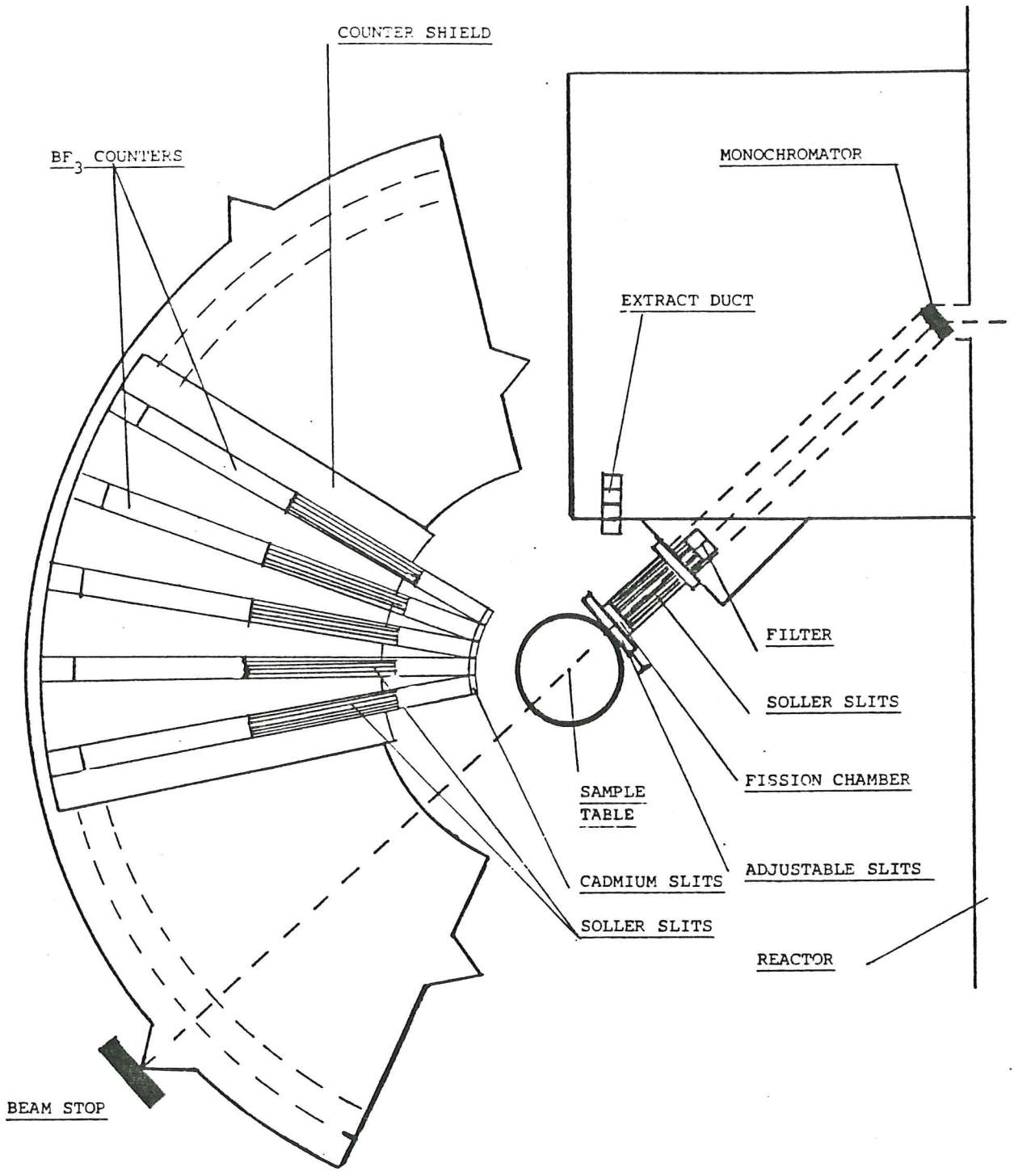

Figure 8.

Schematic layout of CURRAN diffractometer

Figure 9.

The CURRAN Powder Diffractometer with a variable temperature cryostat mounted on the sample table.

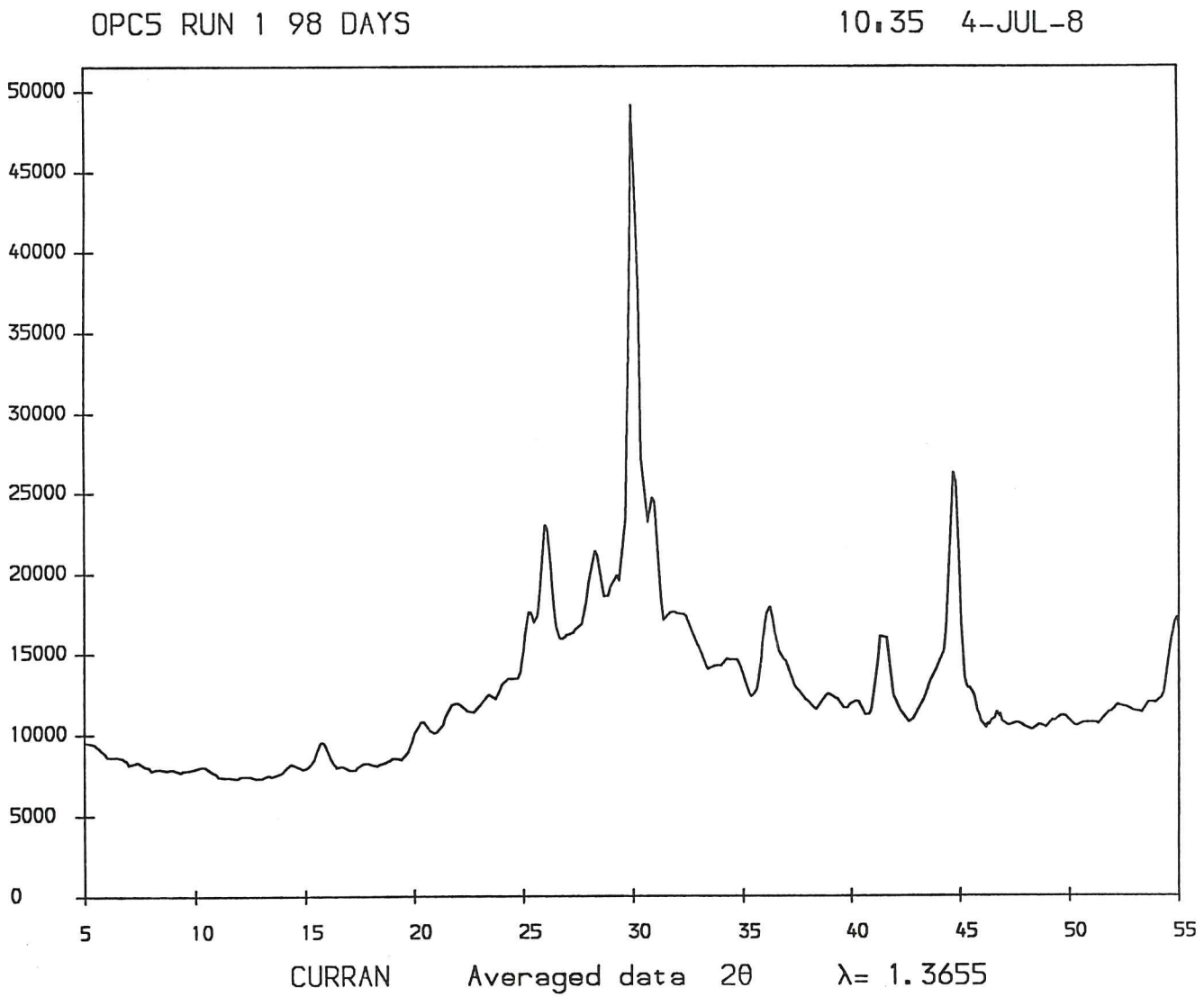

Figure 10.

A typical CURRAN diffraction pattern for deuterating cement. Not only can the disappearance of peaks from anhydrous crystalline phases be observed as deuteration progresses, but also the appearance of new peaks due to deuterated products. Here in this 98 day old cement, the most prominent peaks are due to $Ca(OD)_2$.

10H Powder Diffractometer

This diffractometer has good angular resolution ($\sim 0.5°$) for all values of 2θ and so is particularly suitable for structure studies of powders. It can also be used for structure work on liquids and amorphous solids over the range of momentum transfer Q = 0.8 to 11.4 $\mathring{A}^{-1}$.

The machine background is high due to the close proximity of other spectrometers and a sample giving medium or high intensity scattering is advised.

There is independent 2θ and ω movement to facilitate the use of flat or cylindrical samples and the whole range of Harwell's cryostats, furnaces and magnets can be accommodated.

The instrument is fully automated in operation, control and data acquisition is by an INTEL 8080 microprocessor.

10 HPD Instrument Details

Monochromator	Cu (4,2,2)
Take-off Angle	85°
Wavelength	1.00 Å
Maximum Beam Size	45×33 mm^2
Usual Sample Size	10 mm $\phi \times 40$ mm
Flux at Sample	2×10^6 ncm^{-2} s^{-1}
Resolution	0.6° at 2θ = 25°
	0.45° at 2θ = 55°
	0.55° at 2θ = 85°
	0.7° at 2θ = 110°
Angular Range	−20° to 130°
Minimum Useful Angle	8° (1)
Minimum Step Length	0.01°
Background (No Specimen)	50 cpm (2θ = 55° to 130°)
	65 cpm (2θ = 35°)
	100 cpm (2θ = 25°)
Detector System	3×50 mm He^3, 14° apart
	or 5×50 mm He^3, 7° apart (2)
Q-Range (Å^{-1})	0.8 to 11.4 Å^{-1} (1.0 Å)

(1) A new nose-piece for the counter shield is manufactured which should reduce this angle appreciably.

(2) Using the 5 detector array increases the background by ~30%.

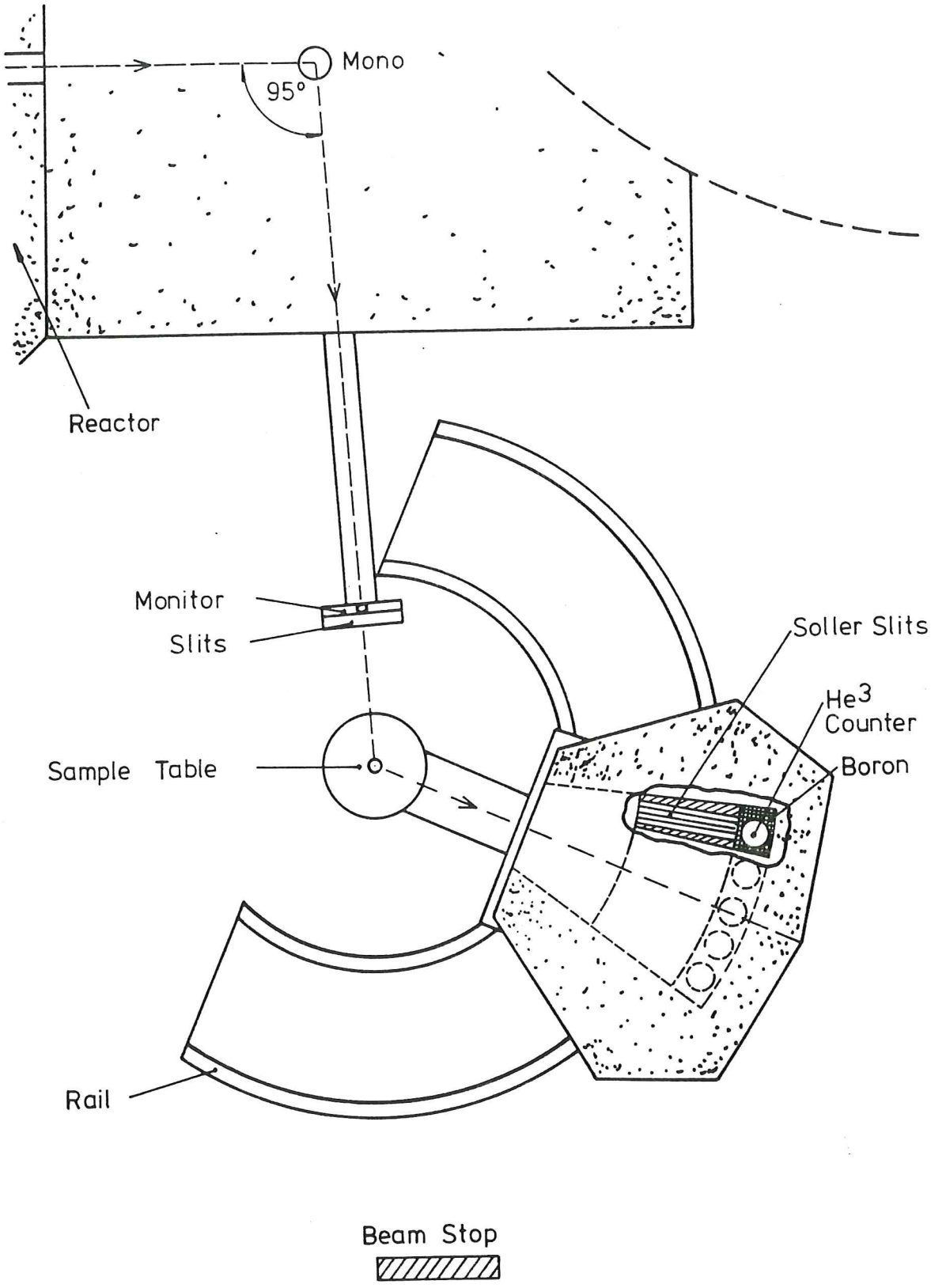

Figure 11. Schematic layout of 10H powder diffractometer HRPD

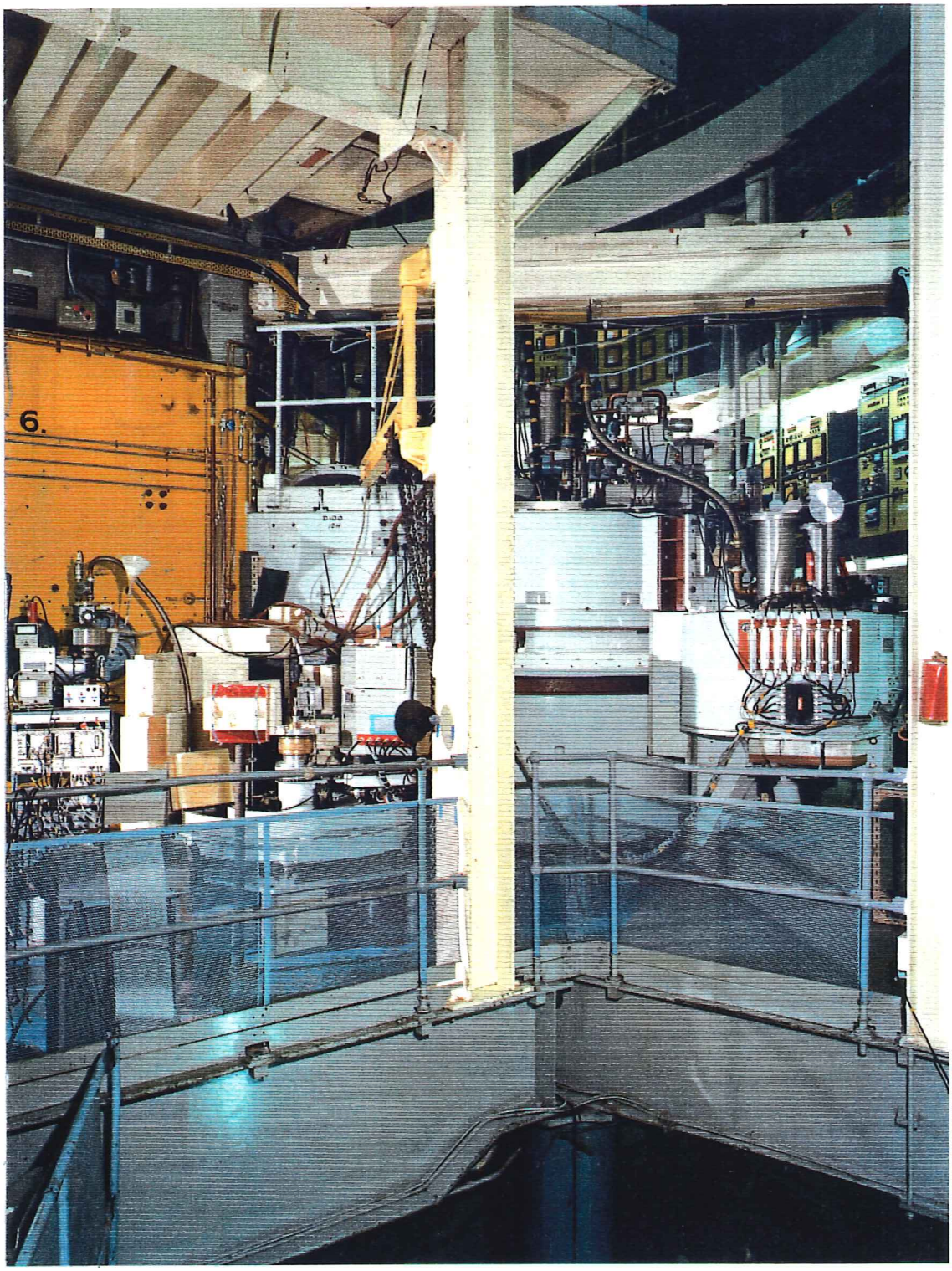

Figure 12.

General view of the High Resolution
Powder Diffractometer (HRPD).
The Beryllium Filter Spectrometer
is shown on the right of the picture.

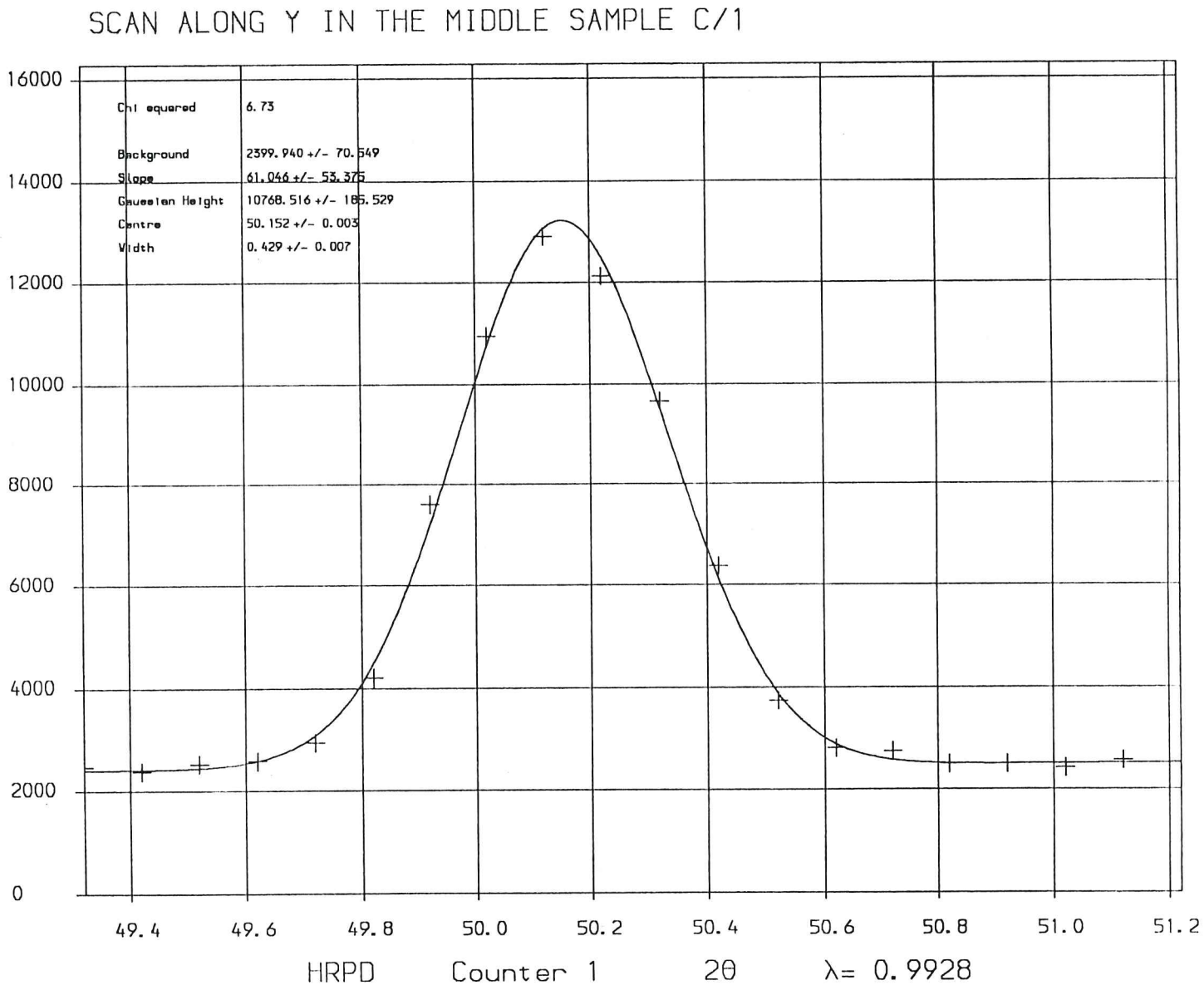

Figure 13.

The (211) diffraction peak from a 3mm x 3mm x 3mm measuring volume inside a welded steel sample. The continuous curve is a computed fit using the program PKFIT. Derived parameters are used together with reference values to determine the lattice strain from which stress values can be determined.

PANDA High Resolution Powder Diffractometer

This instrument is designed as a general purpose powder diffractometer. Its prime use is for powder profile refinement of structures over a wide range of temperatures. Because it has a variable take-off angle (TOA) the instrument can be used either in a high intensity mode with a low TOA or in a high resolution mode with a high TOA. Because of the demand for profile analysis it is generally used at the present in the latter mode.

Special features include:

1. Variable take-off angle (9-90°) giving good resolution up to 100°. The focussing angle can be changed to give the experimenter optimum resolution over a wide range of Q space.
2. Choice of squashed germanium, perfect germanium or copper monochromators. These combined with the variable TOA feature allow the user a very wide choice of wavelengths in the range .7 Å to 4 Å. These can be easily selected by either rotating the monochromator or changing the TOA.
3. An array of BF_3 detectors arranged on a 3×3 matrix. This allows the user to change the effective vertical collimation and hence improve counting statistics.

The instrument has been used for numerous investigations over the past ten years. Studies of phase changes have ranged from 10 K to 1900 K. Materials studied have included polymers such as polypropylene, PTFE and polyethylene; organics such as adamantane, acetic acid and chlorinated aromatics, complex inorganics such as zeolites and β-alumina, simple ceramics such as UO_2 and ThO_2 as well as metallic compounds and alloys. Defect structures and doped fluorite structures have also been extensively investigated. Recently, absorbed gases on surfaces have been examined. The instrument can be usefully used for any investigation involving Bragg diffraction. At Harwell, however, it is usually used where high resolution is required, at the expense of intensity.

Physical Description

The instrument is shown in plan view in the accompanying drawing. The TOA is changed by rotating the shielding turret together with the specimen arm. Three slots are available to vary $2\theta_m$ from 0° to 90°. The monochromator is shown in reflection. Normally the Copper monochromator is used in this sense whereas the germanium monochromator is used in transmission (rotated by 90°). Planes are selected by a rotation about an axis in the plane of the drawing. The angle θ_m is matched to suit $2\theta_m$ by rotating the monchromator plug (ω rotation). The sample table can be rotated continuously, or in steps either with or without 2θ. The printout of the nine BF_3 counters can be adjusted by a diode board. Various scans can be selected (ω-2θ, ω, or 2θ) by a hard wired unit using thumb wheel switches. This unit can also be used to set monitor count limits. Data output is via a computer and hard disc.

PANDA Instrument Details

Beam hole	7H4R PLUTO
Monochromators	Germanium, squashed germanium and copper.
Wave length range	.7 Å to 4.6 Å
Flux	3×10^5 n cm^{-2} s^{-1}
Resolution	.3° angular
	$\Delta d/d \sim 3 \times 10^{-3}$ ($2\theta_M = 90°$)
Angular range	$-60° < 2\theta < 110°$ (depends on TOA)
	$0° < \omega < 360°$
Beam size at sample	65 × 65 mm
Collimation	α_1 20' (fixed)
	α_2 variable (slits and sollers)
	α_3 13.4' and 30'
Detectors	9 × BF_3 at 70 cm Hg (5 cm diameter)
Background without sample	10 cpm
Filters	Pyrolytic graphite and beryllium
Ancillary equipment	Cryostats 1.5 K to 300 K.
	Various types, e.g. centre loading variable temperatures.
	Furnace (1600°C)

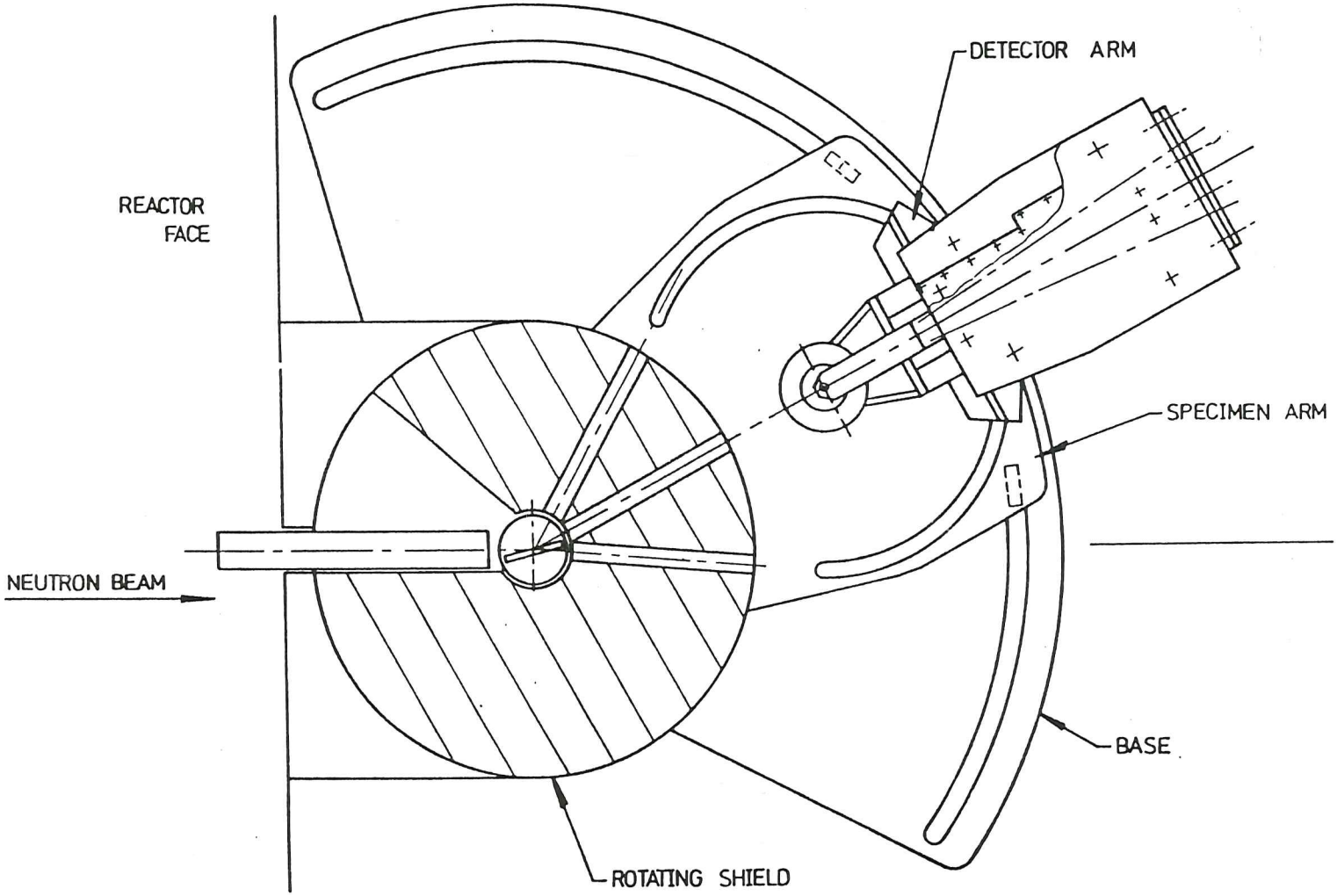

Figure 14.

Schematic layout of PANDA diffractometer

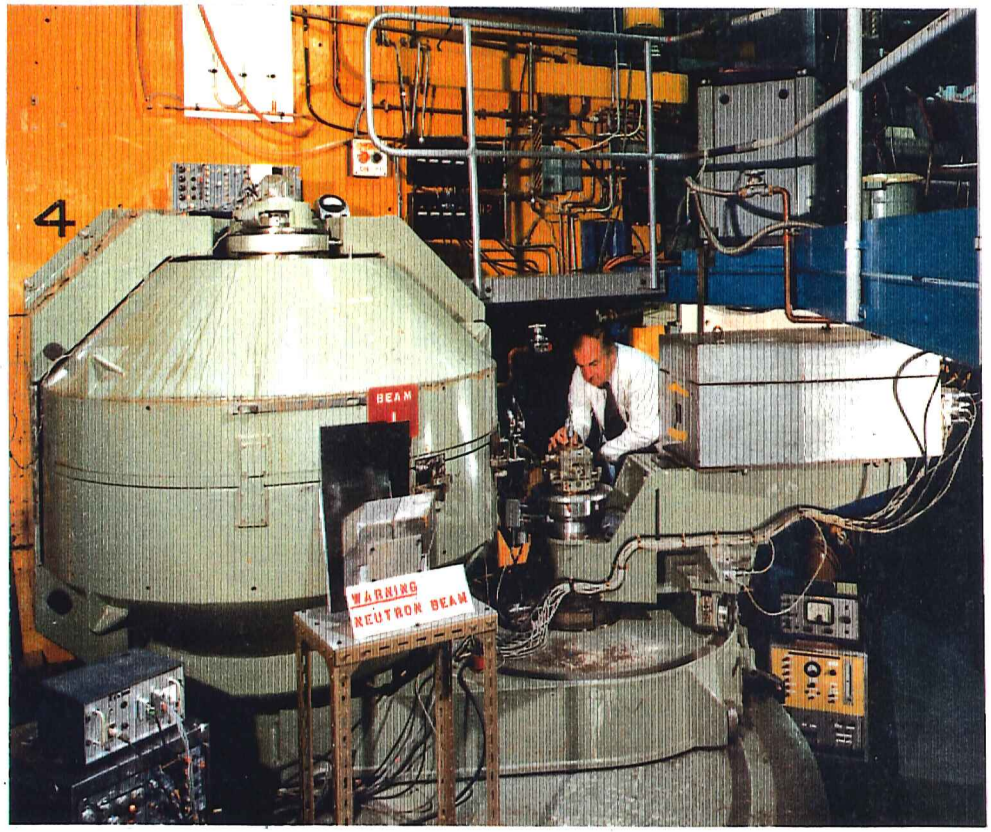

Figure 15.

The PANDA High Resolution Powder Diffractometer

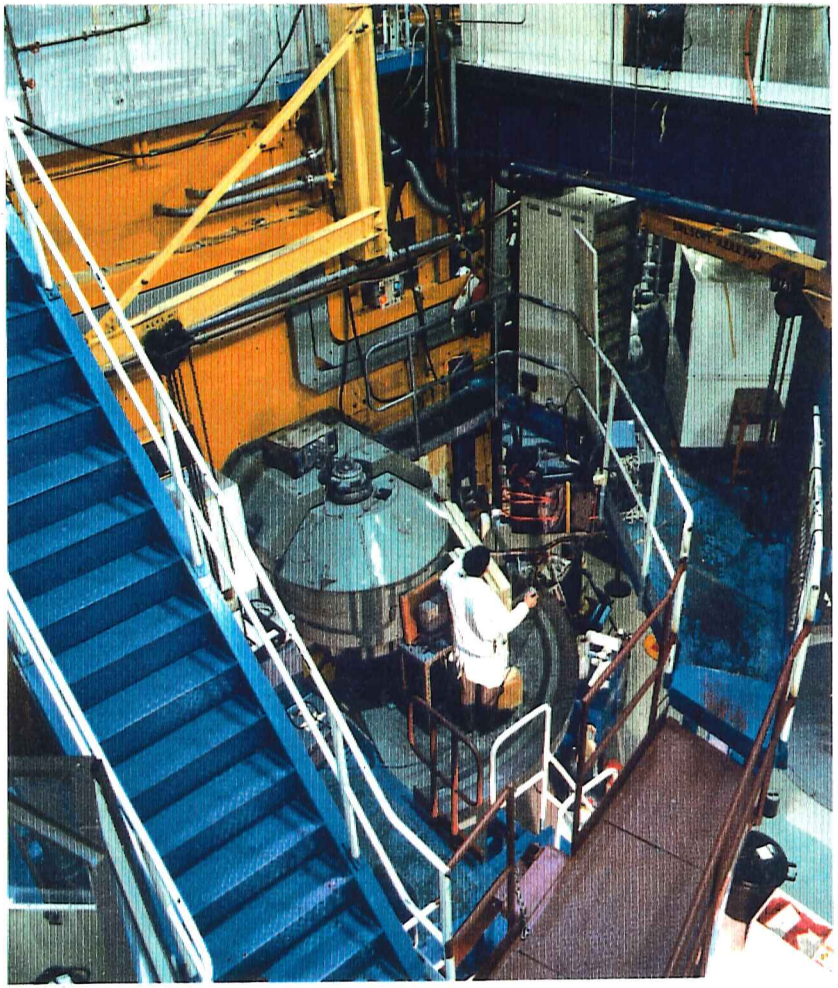

Figure 16.

General view of the PANDA High Resolution Powder Diffractometer

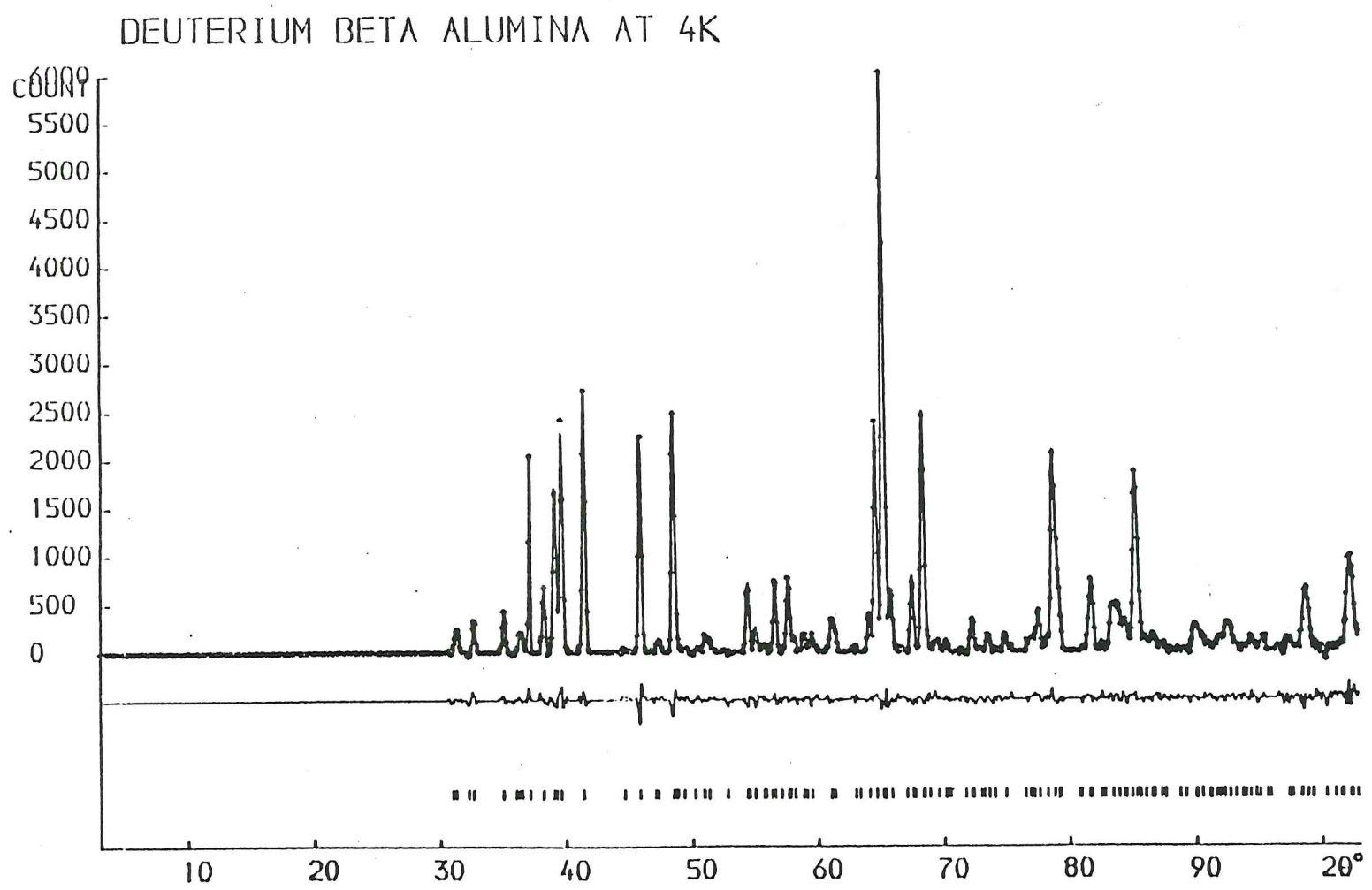

Fig. 17
Observed (dots) and calculated diffraction pattern for $DAl_{11}O_{17}$ at 4.6 K after the best refinement.
The difference profile and the positions of the reflections are also shown.

Triple Axis Spectrometers

Introduction

The two instruments are conventional medium energy range spectrometers with principal use in the measurement of $S(Q,\omega)$ at constant Q or constant $\hbar\omega$, giving for example the energy dispersion relation of excitations in solids: phonons, magnons, excitons, the form of critical scattering etc. Software programs in the controlling computers are very versatile and enable all relevant parameters to be changed automatically.

The instruments may be used as two-axis spectrometers with the advantage that automatic scans may be made in any direction in the reciprocal space of a single crystal sample. They may also be used as powder or single crystal diffractometers with elastic energy selection by the analyser.

DIDO Triple-Axis Spectrometer

Uses

Monochromator and analyser crystals can be readily changed, as can the soller slit collimation. Scans of (Q,ω) can be made by varying $\underline{Q}$, or $\hbar\omega$ via E_f. At present only one take-off angle from the monochromator is available ($2\theta_M = 40.5°$), but in the near future θ_M and $2\theta_M$ will be automatically variable via the software programs, with $2\theta_M$ varying between 30° and 70°, so that $\hbar\omega$ scans may be also made by varying E_i.

Description

The instrument shares its monochromator-drum shielding with the two other instruments of the 10H complex:– the Beryllium Filter spectrometer and the Powder Diffractometer. The main scattering arm from the sample is cantilevered from a rotating gun mounting, and the analyser axis and detector shield are in turn cantilevered from the arm. The sample table moves relative to the main arm on which it is mounted. A system of Jabroc shielding houses the S-A soller and contains curved boral plates which rotate around the analyser, Jabroc wedges shield the sample. The monochromator is readily accessed via an 'insertable coffin'. At present the whole S-A-D assembly is bolted to the reactor floor in one fixed position corresponding to $2\theta_M = 40.5°$, however in the near future it will be mounted on air pads which move on rails around the drum, and $2\theta_M$ will become automatically variable between 30° and 70°. The monochromator is similarly automated, so that scans with E_i varying can be made.

DIDO Triple-Axis Instrument Details

Angular Ranges

Monochromator take-off angle	40.5° (fixed); Shortly 30-70°.
Specimen – analyser angle*	-110° to +95°
Analyser – detector angle*	-95° to +95°
M, S, A crystal setting angles	M ± 160°; S & A ± 320°.

*Limited slightly in certain configurations by the position of the beryllium filter.

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

Collimation

In-pile –	open
Monochromator – sample –	0.33°, 0.75°, 1.5°, 3°
Sample – analyser –	0.34°, 0.5°, 0.76°, 1.0°
Analyser – detector –	1.0°

Max. Beam Size

40 mm wide × 50 mm high.

Detector

He^3 end window.

Monitor

Low efficiency fission chamber; 20 μgm U^{235} cm^{-2} 10 cm diameter.

Monochromators

(R = reflection, T = transmission)

Pyrolytic Graphite (002)R, (004)R etc. (0.4 or 0.8° mosaic)

Aluminium planes $\perp$ to $[1\bar{1}0]$ T.

Copper (111)R.

Other crystals are available if required.

Analysers

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

Germanium planes $\perp$ to $[1\bar{1}0]$T.

Copper (111)R.

Other crystals are available if required.

Filters

Pyrolytic Graphite (2" × 2" × 2"), 3.5° ± 1.5° mosaic.

Absorbers/Masks

Polythene absorbers – a variety of thicknesses.

Cadmium masks – variety to define beam size.

Flux at Specimen or Monitor

2×10^6 n cm^{-2} s^{-1} at 1.6 Å Al (111)T.