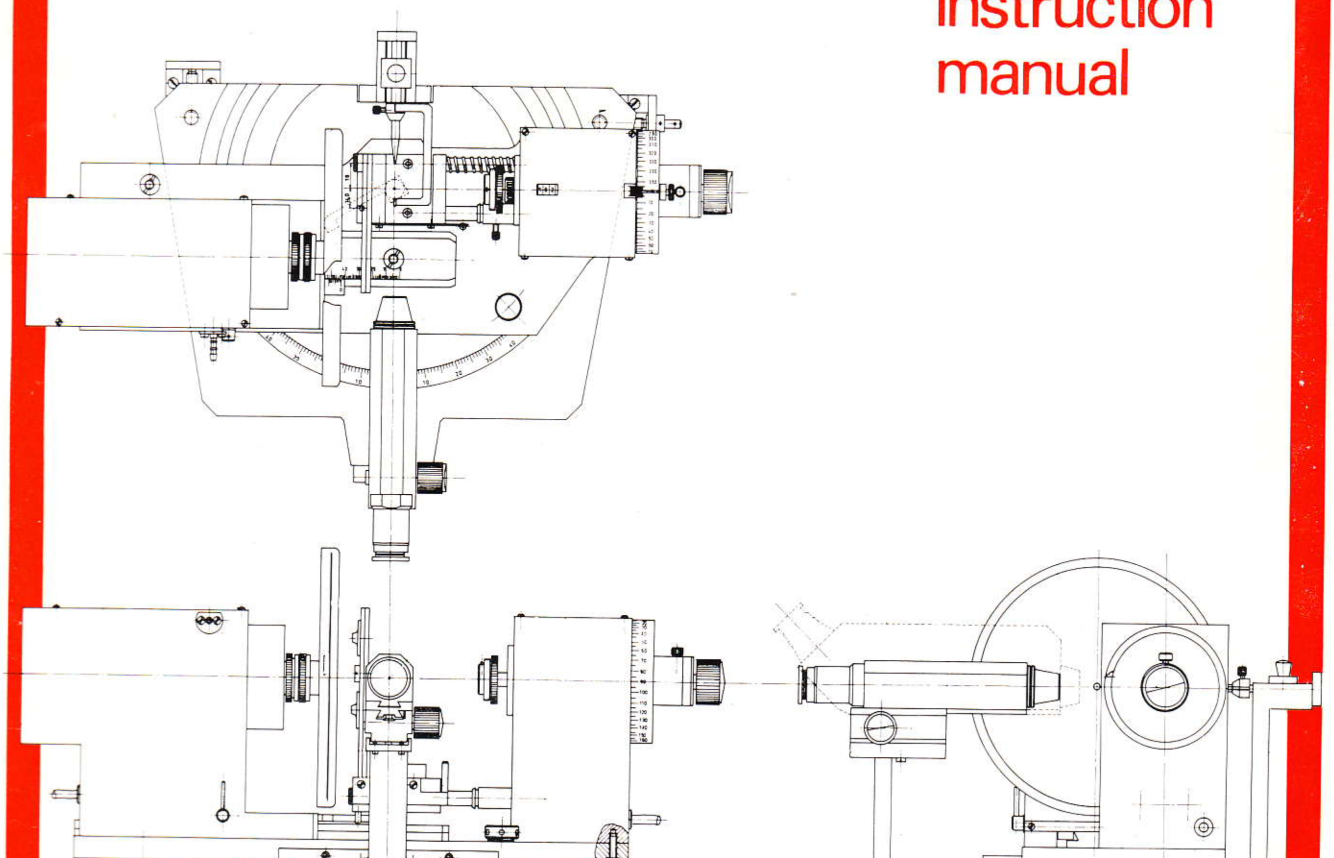
ENRAF
NONIUS
DELFT

RETIGRAPH

Instruction manual



ENRAF NONIUS DELFT

N.V. VERENIGDE INSTRUMENTENFABRIEKEN
ENRAF-NONIUS

P.O. BOX 483, TEL.015-56 92 30, TELEX 31558
RÖNTGENWEG 1, DELFT, HOLLAND

ENRAF NONIUS DELFT

DELFT — SOLINGEN — PARIS
LONDON — NEW-YORK — COPENHAGEN

RETIGRAPH

Important

Read the instructions before assembling and operating the instrument.

Contents

		Page
1.	Warning to users	2
2.	Assembly instructions	5
3.	List of parts with reference numbers	6
4.	Description of the units	23
4.1.1.	Base with collimator stand	23
4.1.2.	Inclination table	23
4.1.3.	Crystal driving unit	23
4.1.4.	Microscope support with focussing slide	23
4.1.5.	Microscope and autocollimator	23
4.2.1.	Collimators	24
4.2.2.	Goniometerhead	24
4.2.3.	Beamstops	24
4.2.4.	Screen holder	25
4.2.5.	Retigraph cassettes	25
4.2.6.	Laue/Polaroid stand	25
4.2.7.	Laue cassette	25
4.2.8.	Polaroid cassette	25
4.3.1.	Film driving unit	26
4.3.2.	Integrating mechanism	26
4.4.1.	Controller	27
4.4.2.	Crystal axis, film axis and integration	27
5.	Operating instructions	27
5.1.1.	Choice of the X-ray tube	27
5.1.2.	Mechanical check on the alignment of the instrument	28
5.1.3.	Aligning the Retigraph in front of the X-ray tube	28
5.2.1.	Centering of the crystal	29
5.2.2.	Alignment of the crystal (oscillation exposure on flat film way)	30
5.2.3.	Alignment of the crystal (Retigraph technique)	31
5.2.4.	Determination of the cell length along the rotation axis (flat film method)	33
5.2.5.	Determination of the cell length along the rotation axis (Retigraph technique)	33
5.2.6.	Determination of the axes lengths not coincident with the rotation axis and the angles between them	40
5.2.7.	Retigraph exposures (non-integrating/integrating)	40
6.	The reciprocal lattice and the Retigraph geometry	53
6.1.1.	The reciprocal lattice	53
6.1.2.	Construction of the Retigraph	56
6.1.3.	Recording techniques	57
6.1.3.1.	Recording the zero layer	57
6.1.3.2.	The equal cone technique	59
6.1.3.3.	Equi inclination technique	59
6.1.4.	Check on the correct position of the film	60
6.2.	Intensity factors	65
6.3.	Cooling technique	66
7.	Literature	75

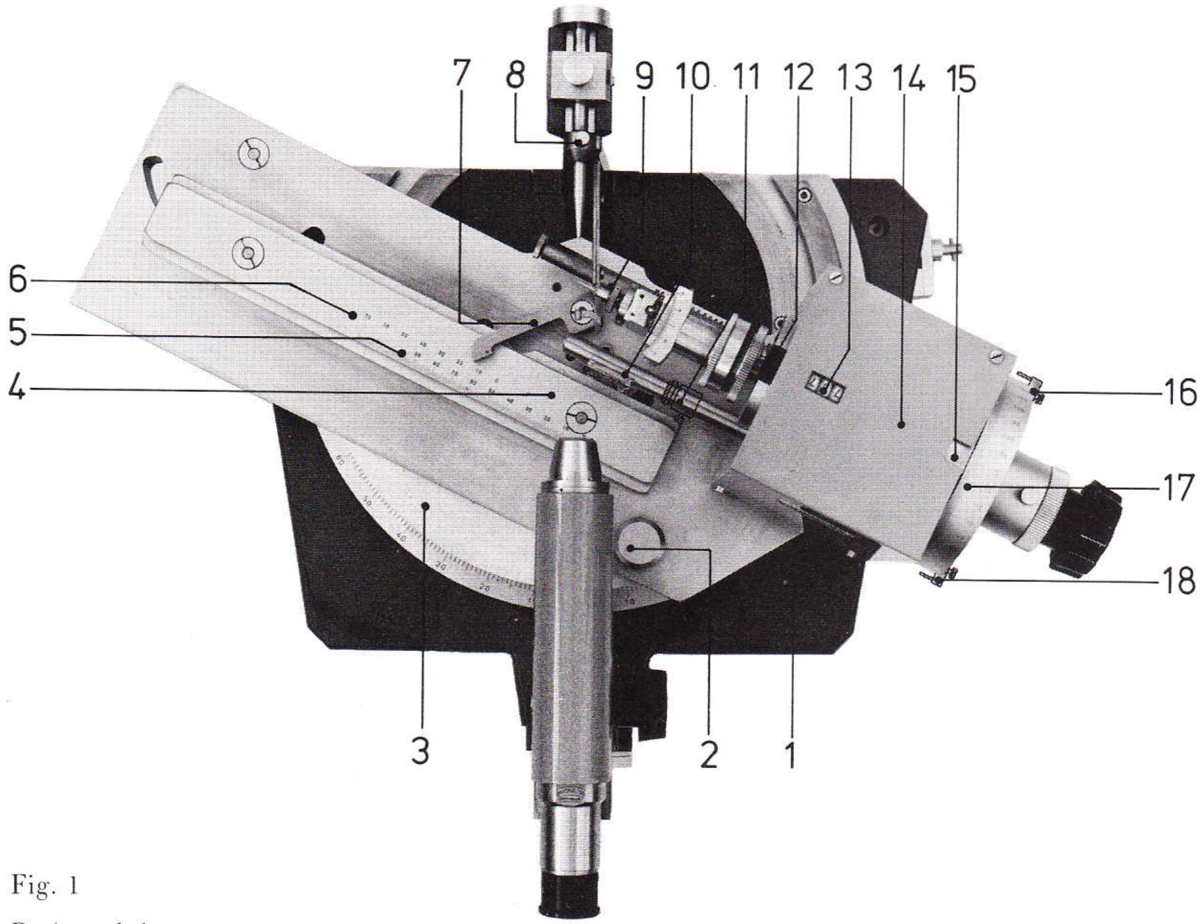


Fig. 1

Retigraph base

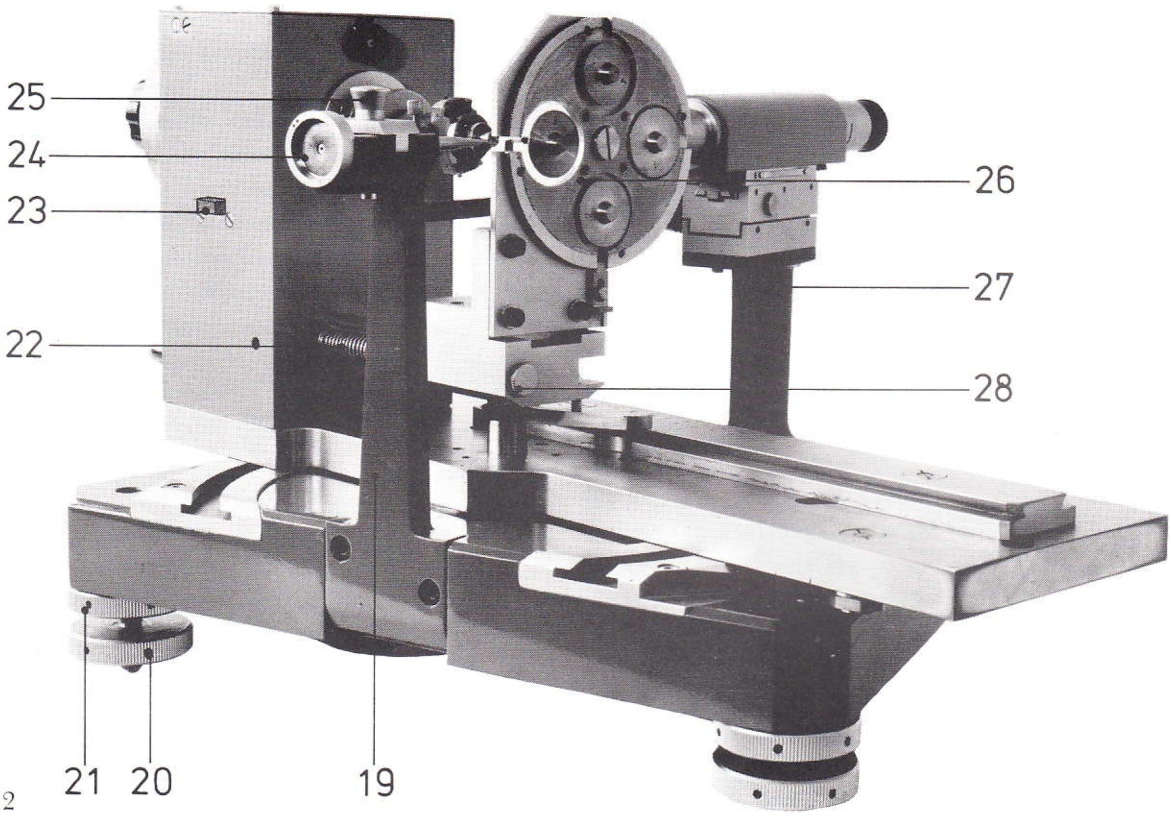


Fig. 2

Base with screen holder mounted

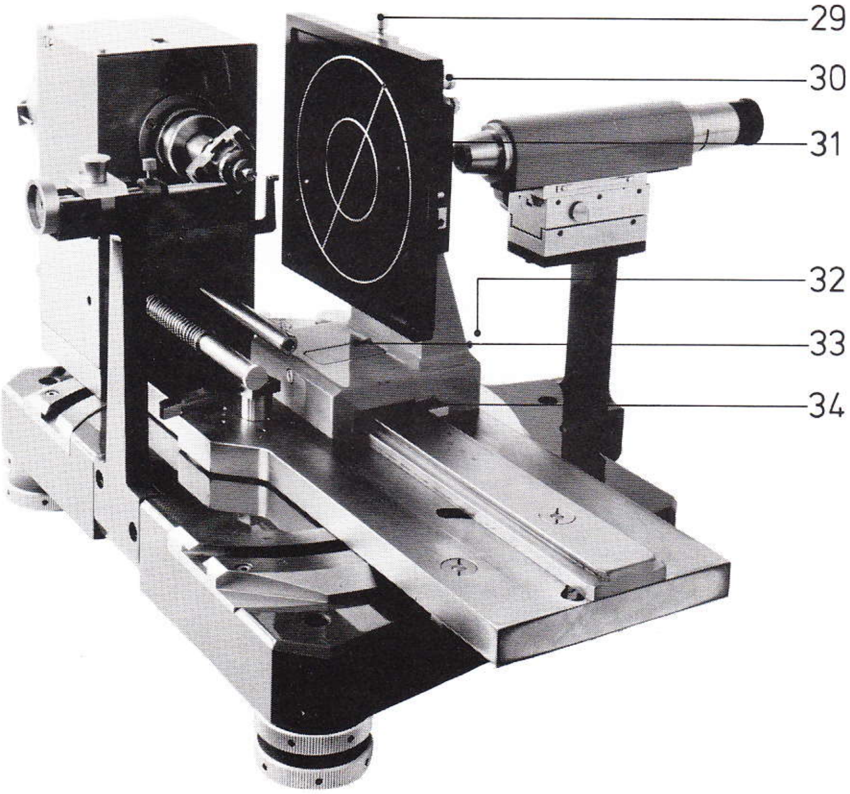


Fig. 3

Retigraph with Laue cassette

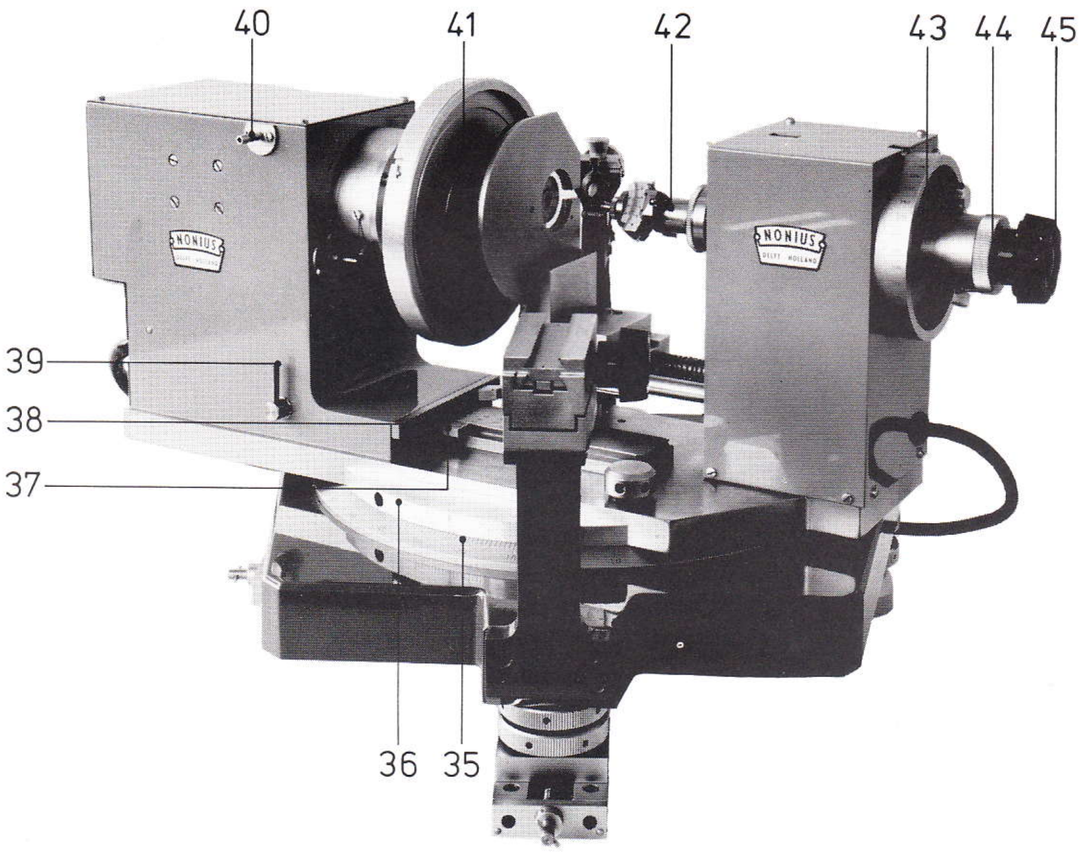


Fig. 4

Retigraph in inclination position

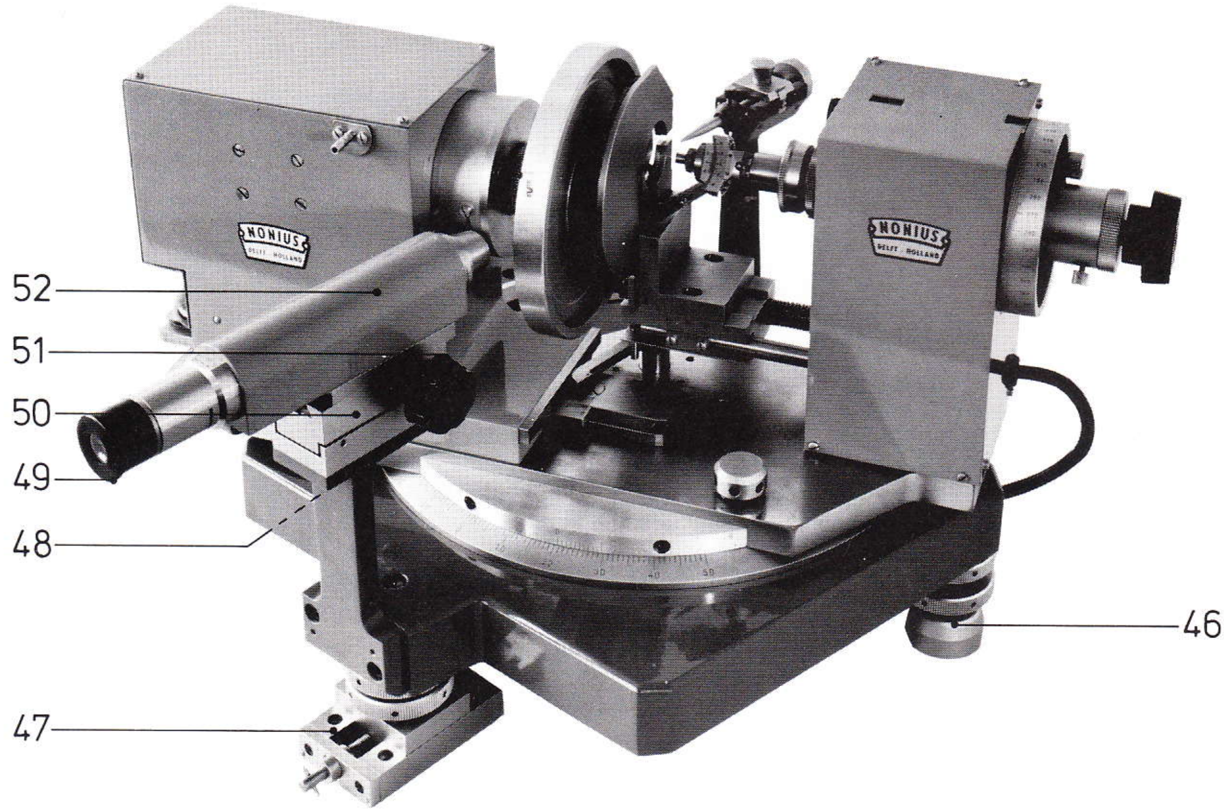


Fig. 5

Retigraph in anti-inclination position

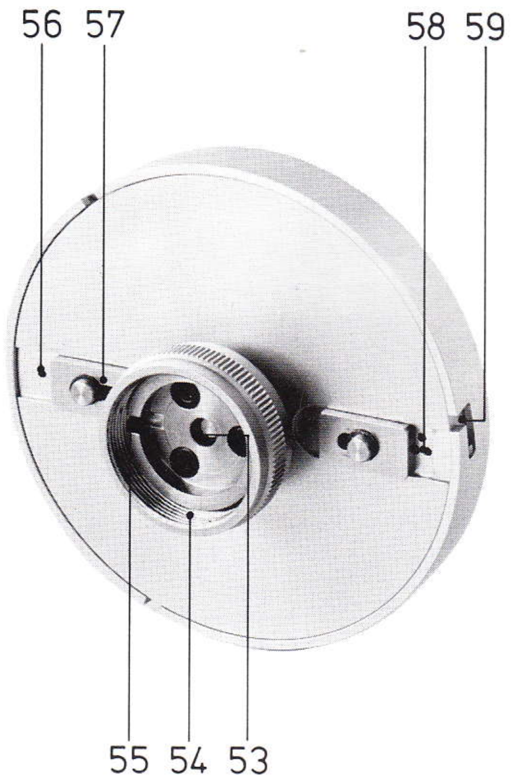


Fig. 6

Cassette

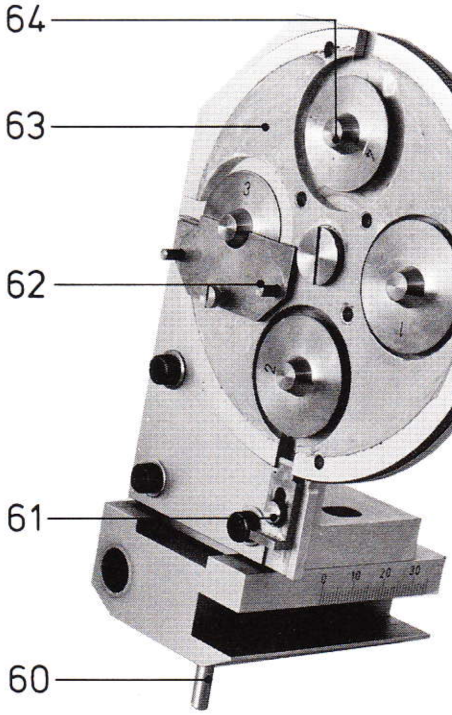


Fig. 7

Screen holder

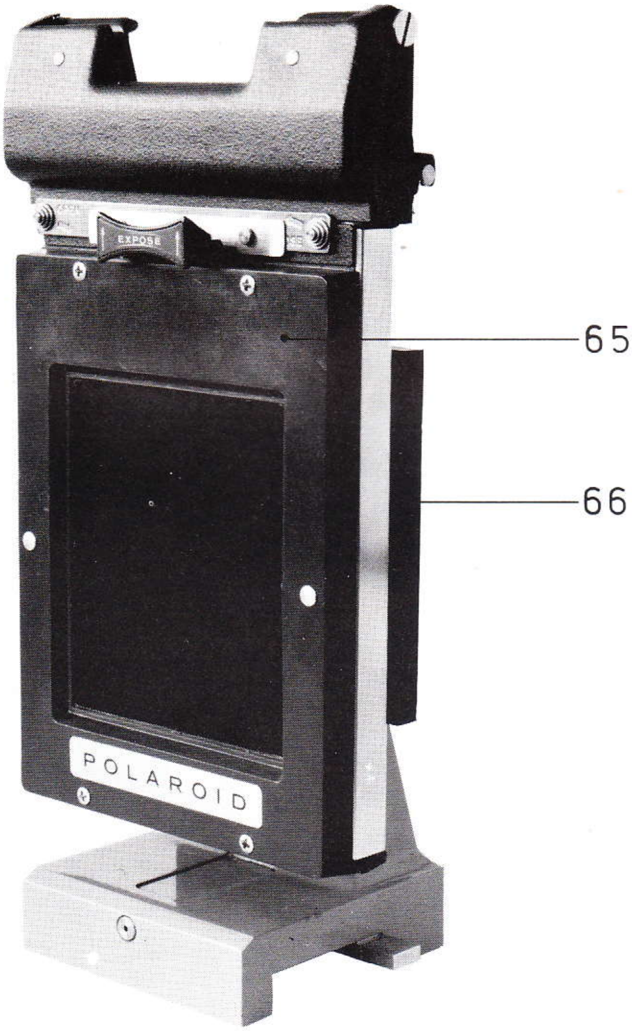


Fig. 8

Polaroid cassette and support

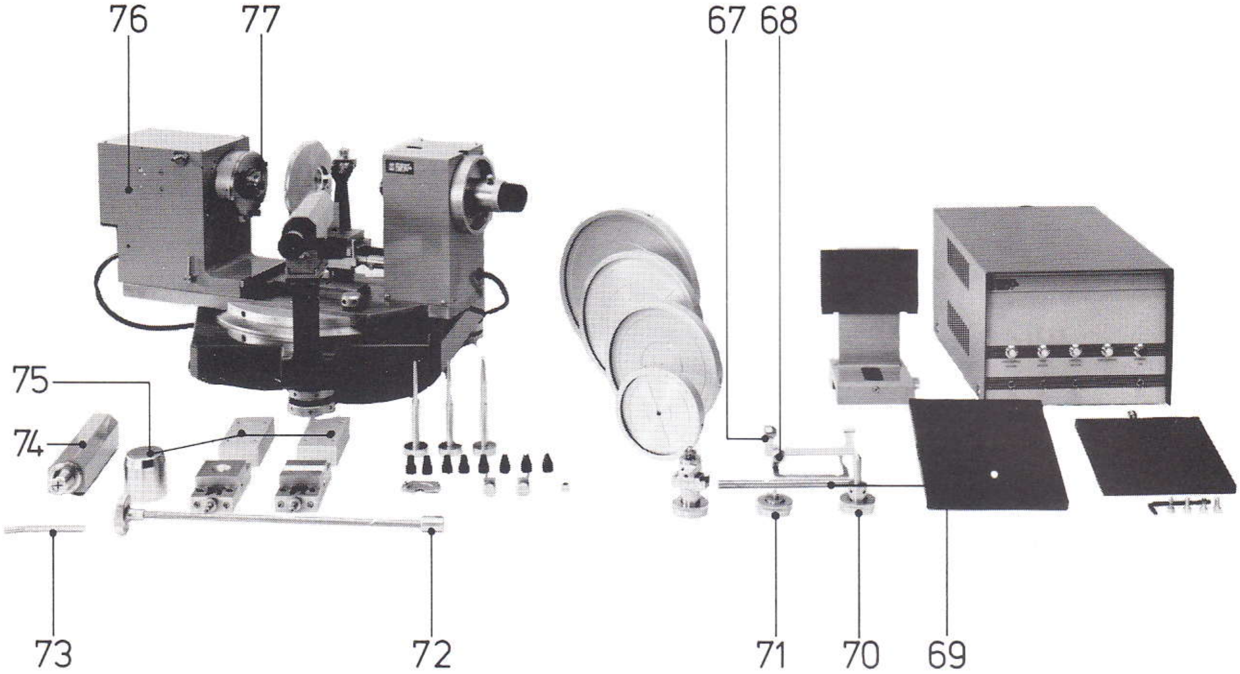


Fig. 9

Retigraph complete with standard accessories

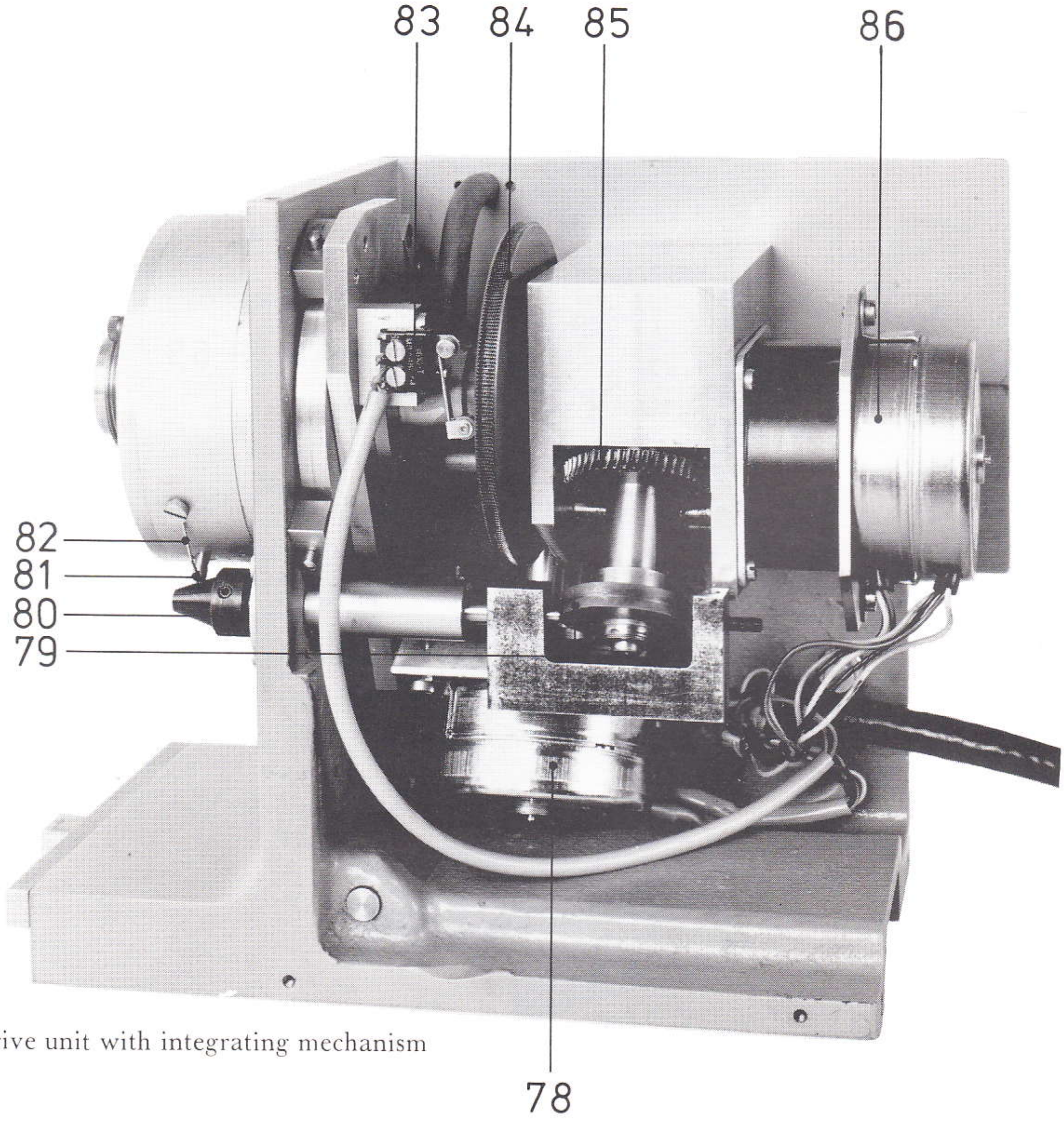
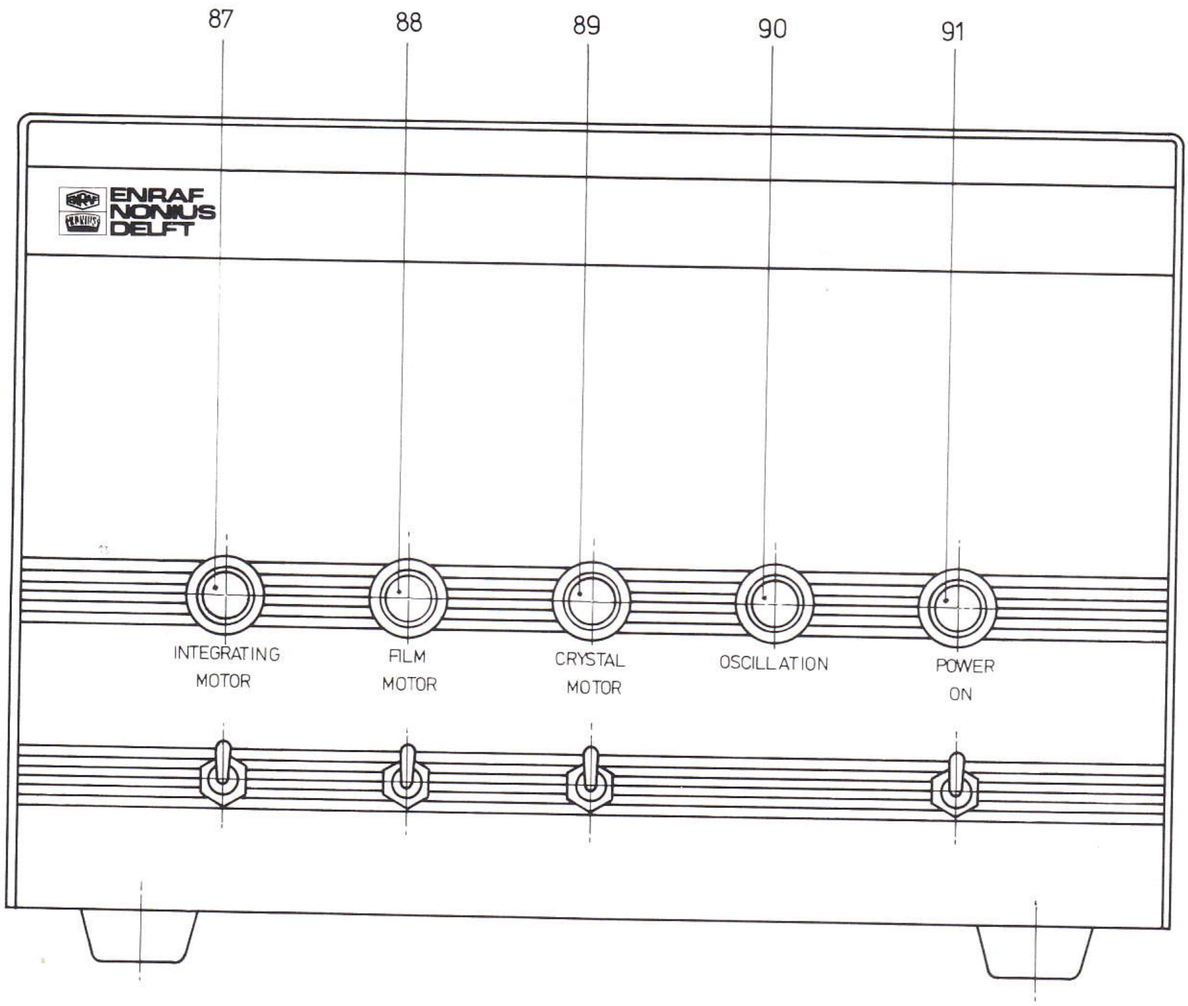


Fig. 10

Film drive unit with integrating mechanism


87
88
89
90
91
ENRAF NONIUS DELFT
INTEGRATING MOTOR
FILM MOTOR
CRYSTAL MOTOR
OSCILLATION
POWER ON

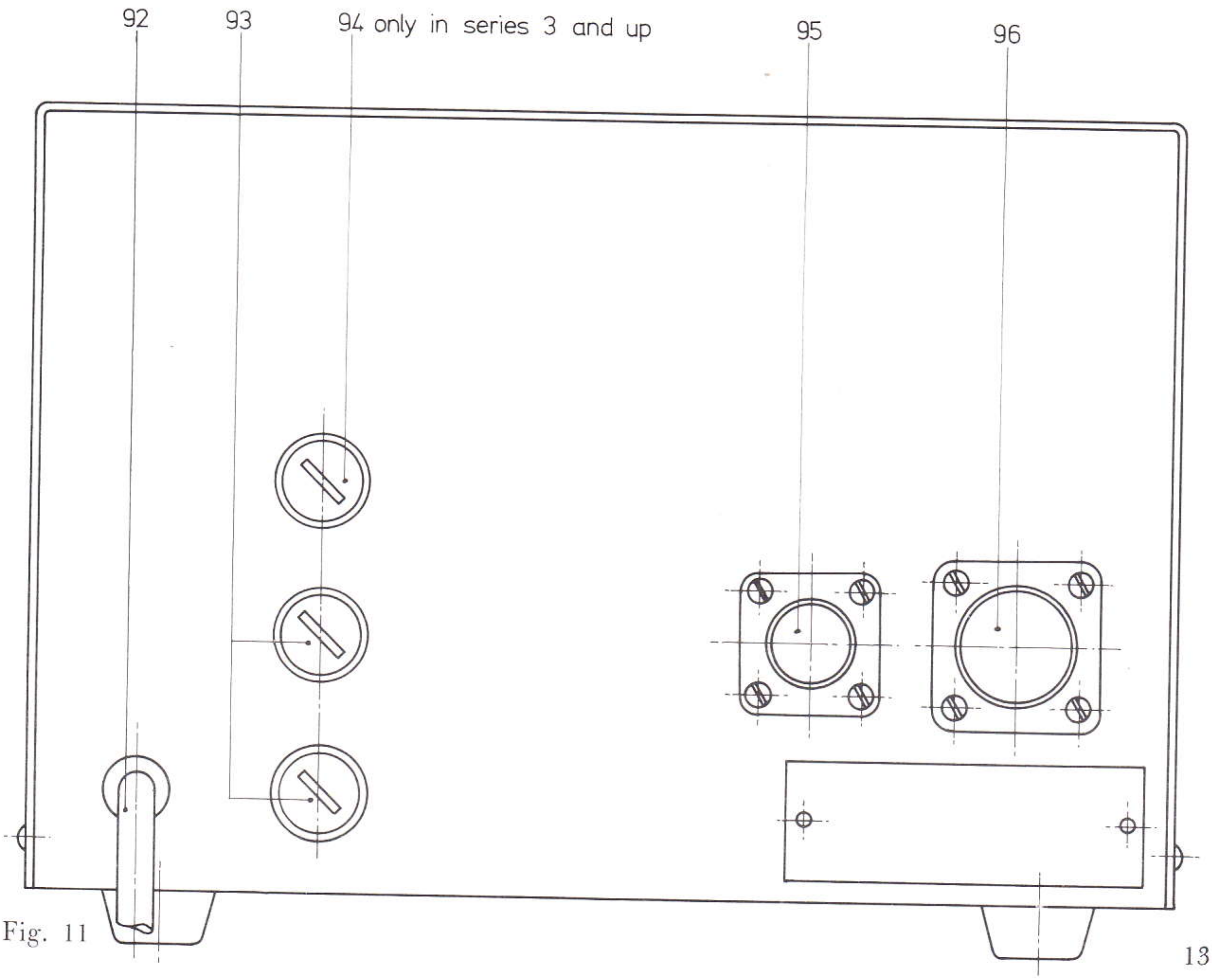

92
93
94 only in series 3 and up
95
96

Fig. 11

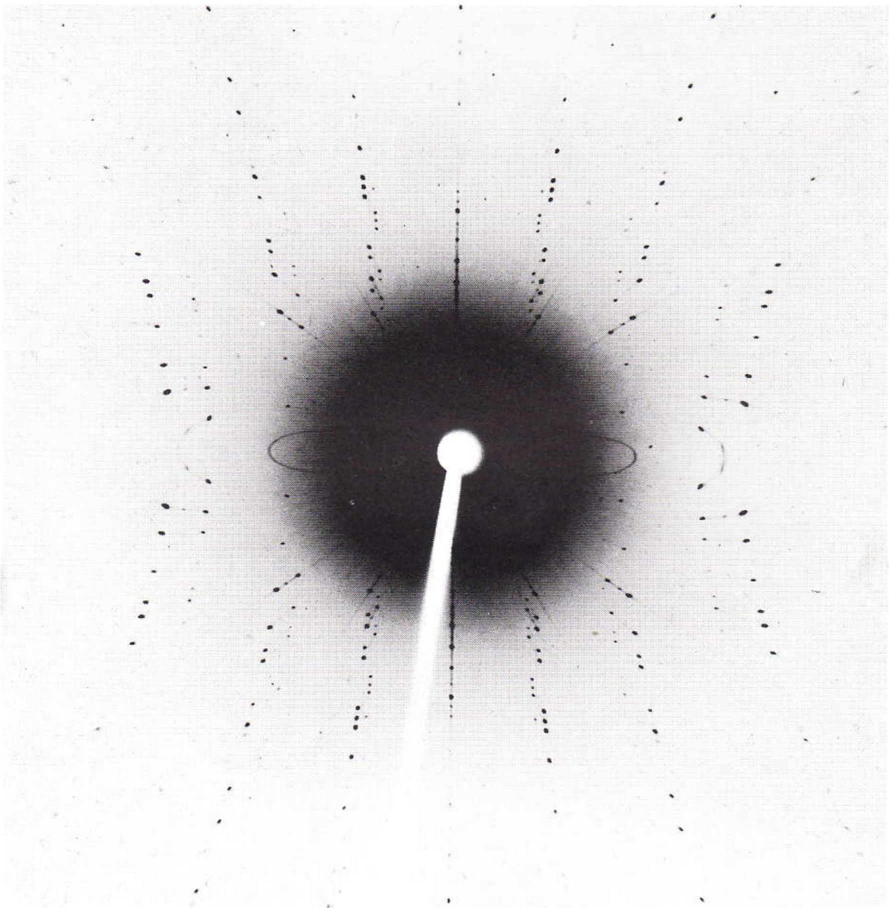


Fig. 19

Alignment exposure flat film technique radiation Cu kα + β

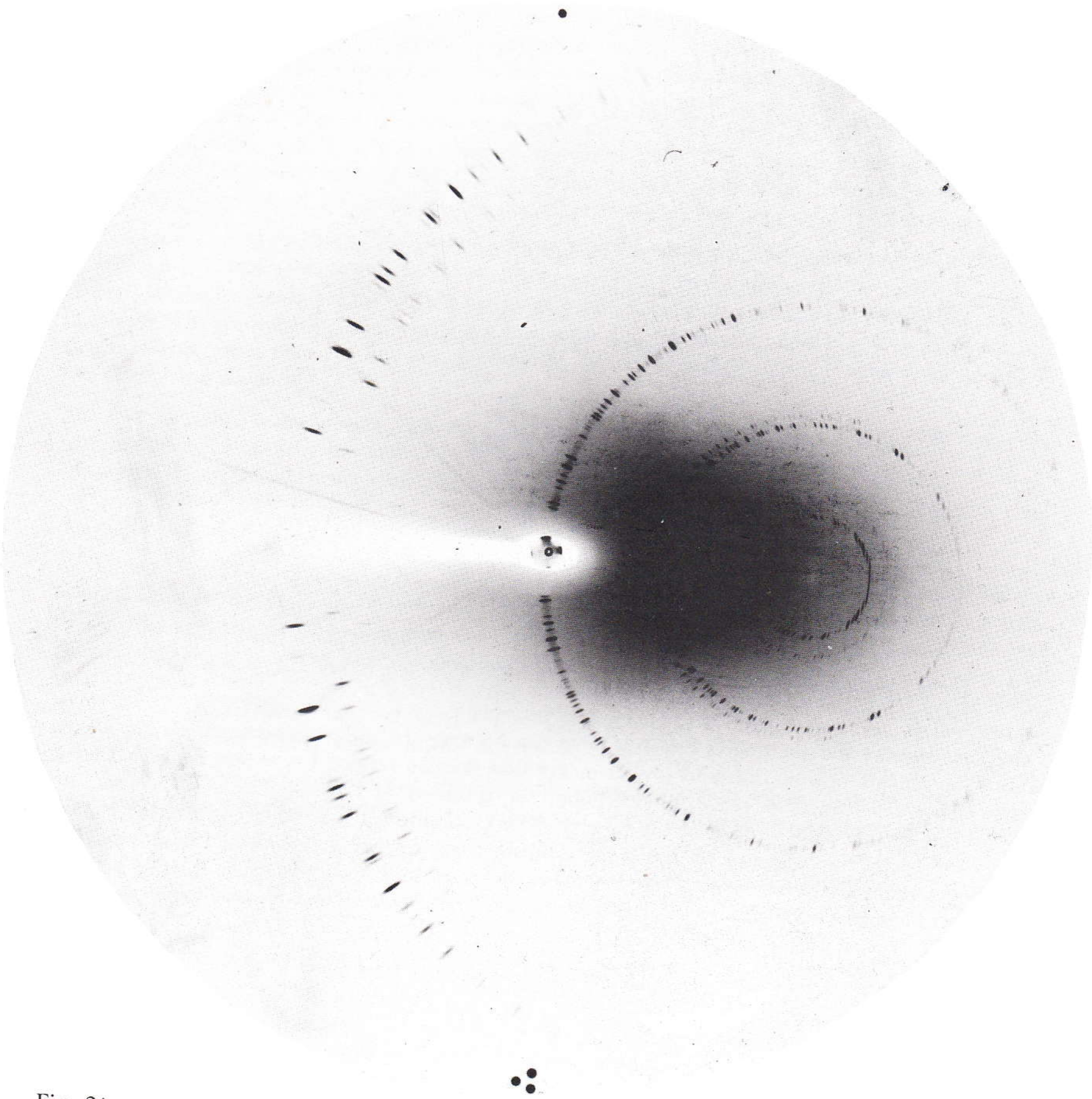


Fig. 24

Secants of Laue cones to determine crystal axis length along rotation axis.
$\mu = -30^\circ$ radiation Cuk$\alpha + \beta$

Pair C_n, C_{n-2}

$$n = 2 \times \frac{0.934 - 0.500}{0.934 - 0.494} \approx 2$$

$$D_o = \frac{50}{2} \times 0.440 = 11 \text{ mm.}$$

Pair C_{n-1}, C_{n-2}

$$n - 1 = 1 \times \frac{0.714 - 0.500}{0.714 - 0.494} \approx 1$$

Note

The theoretical value $\mu_n = |\mu_o|$ cannot be reached as the screen holder will interfere with the collimator. In practice the maximum inclination angle is $\mu_n = |\mu_o| - 10°$.

Integrating exposures

As described in section 5.2.6 the recording of a reciprocal lattice point on the film is a cross. These crosses cannot be used for intensity measurements and therefore one of the reflected rays must be stopped. This can be achieved by mounting the half shutter 62. When the crystal shape is irregular the shape of the reflections will not be identical. The built-in integration mechanism provides a possibility to obtain an area of uniform density in each reflection. Four sets of integration cones are available giving an integration of 0.5 - 1.0 - 1.5 and 2 mm. The choice of the integration cones depends on the size of the reflection and the size of the light spot of the densitometer used for density measurements.

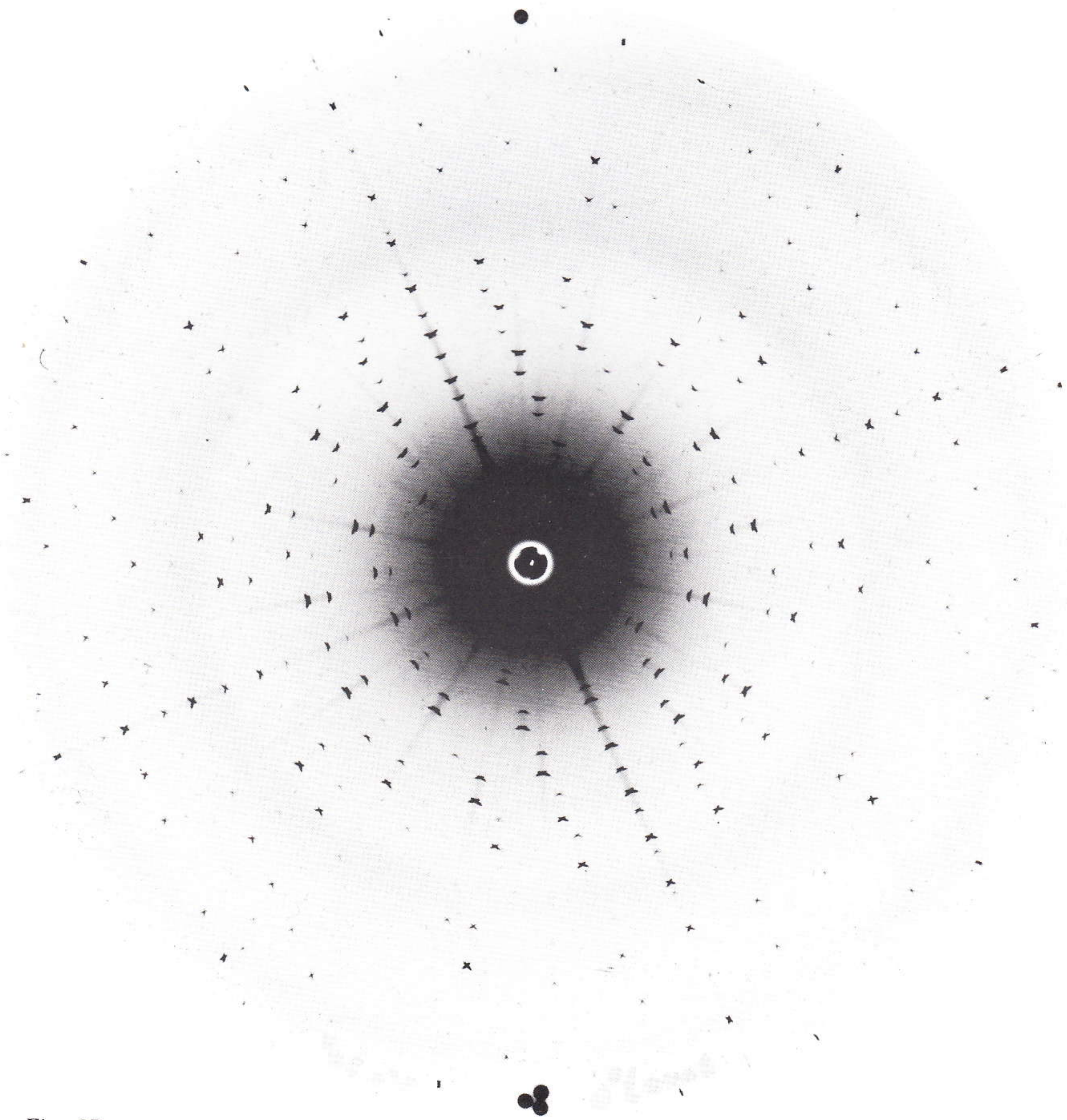


Fig. 27

Zero layer recording $\mu = -30°$ D = 0

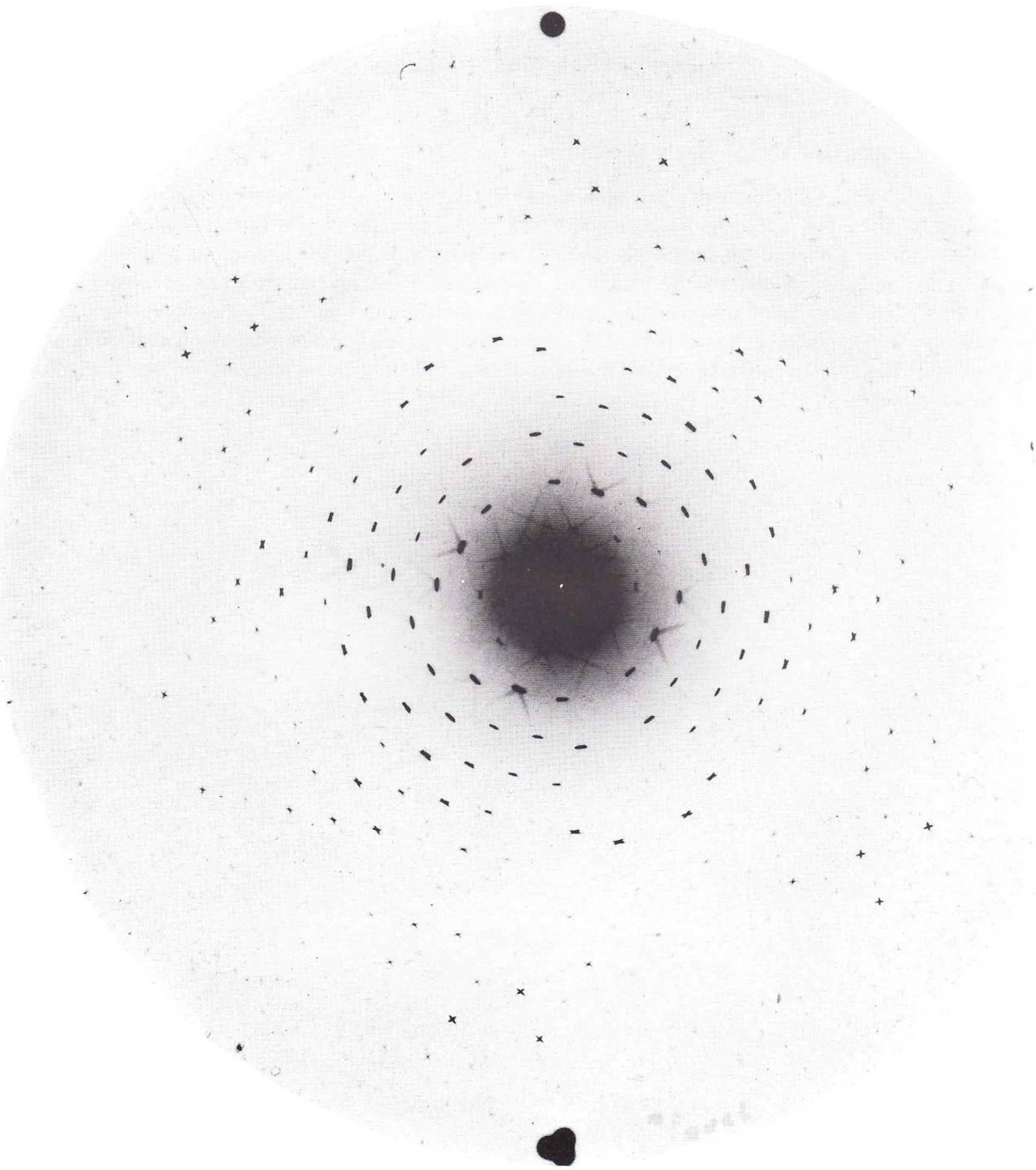


Fig. 28

First layer recording $\mu = -17^\circ$ D = 10.8 mm

Example

To integrate a reflection with sizes	
Along 'horizontal' integration axis	: 1.5 mm
Along 'vertical' integration axis	: 0.7 mm
The densitometer has a light spot	: 0.3 x 0.3 mm
The horizontal integration should be	: $\geq 1.5 + 0.3$; use 2 mm integration cone.
The vertical integration should be	: $\geq 0.7 + 0.3$; the 1 mm integration cone can be used.
The size of the integrated reflection becomes	
horizontal	: $2 + 1.5 = 3.5$ mm
vertical	: $1 + 0.7 = 1.7$ mm

We have: $C_b = \frac{1}{2}(C_n - C_\pi)$

C_n is known, the diameter can be measured on the film.

C_π can be calculated from:

$$C_\pi = 2\left[R^2 - (D^1 - R\sin\mu)^2\right]^{1/2} \quad \text{so that}$$

$$C_b = \tfrac{1}{2}C_n - \left[R^2 - (D^1 - R\sin\mu)^2\right]^{1/2}$$

Triangle abc can be considered to be rectangular at c which leads to the following equations:

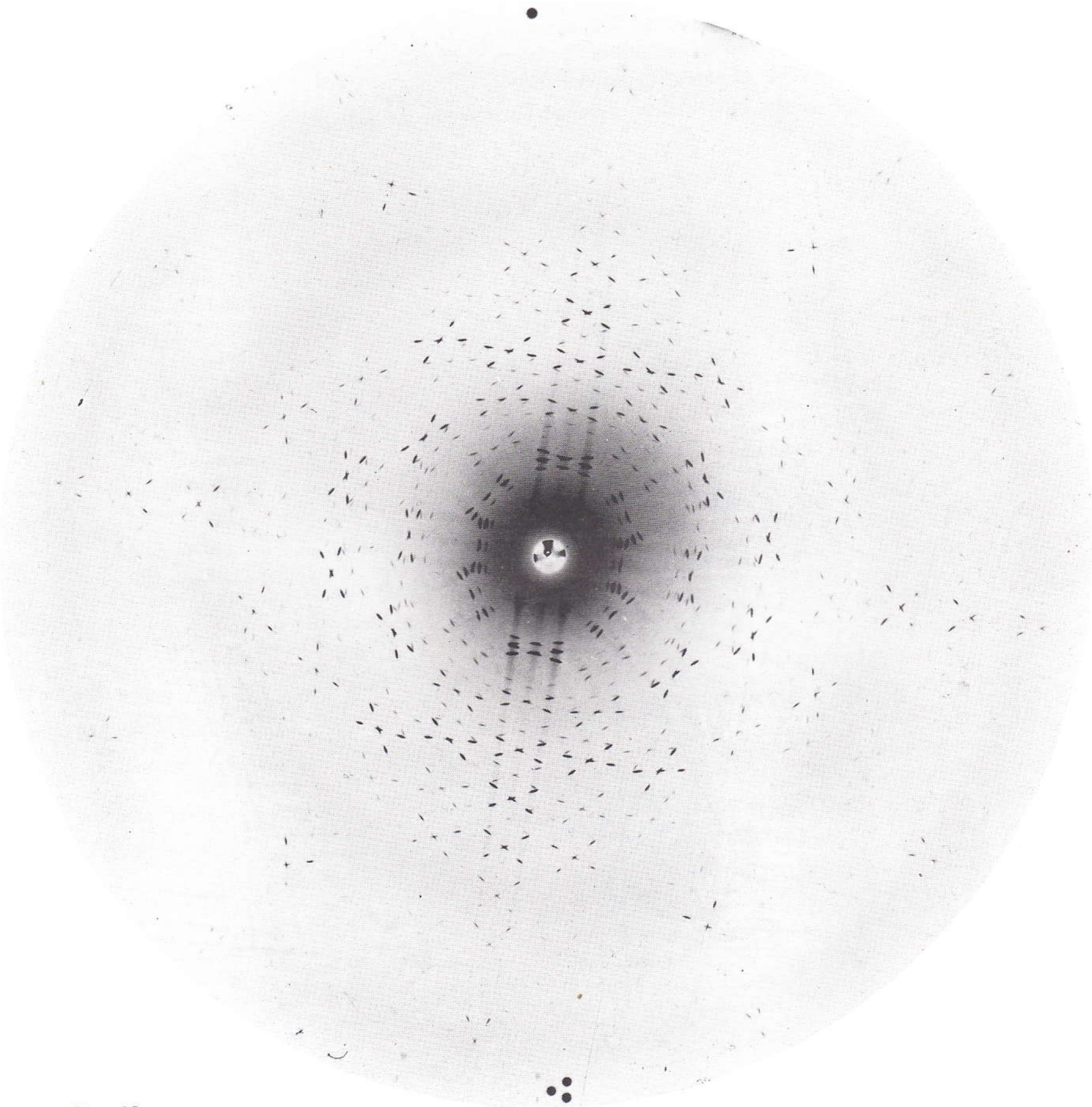


Fig. 39

Exposure showing the effect of
$\triangle D = -2$, $\triangle D = 0$, $\triangle D = +2$ on the same film.
$\mu = -30°$