CHAPTER IV

MEASUREMENTS AND RESULTS

IV.1 Taking Rotation Photographs

Transparent crystals of Latifolin (C_{17} H_{18} O_4) were formed in various shapes and sizes. The largest crystal was approximately 0.4 mm. long and has a cross section of 0.87 mm^2. From the external morphology of the crystal, two small single crystals with crystal axes $\bar{a}$ and $\bar{b}$ were chosen as follows :

$\bar{a}$ or [100] axis along the long axis of the crystal,

$\bar{b}$ or [010] axis along the axis perpendicular to the long axis as shown in Fig. 4.1

Each of the two selected crystals was supported by a very fine glass fiber, to which it was attached with adhesive glue. The other end of the fiber was fixed with wax and attached on the goniometer head. The crystals were mounted with their $\bar{a}$ and $\bar{b}$ axes as rotation axes and placed normal to the x-ray beam.

A Nonius Weissenberg Goniometer camera of diameter 57.3 mm. was used with CuK-radiation (λ_{K_α} = 1.5418 A°, $\lambda_{K\beta}$ = 1.3922 A°). The axis was first aligned normal to the beam by using a 15° oscillation photograph. Rotation photographs with a and b axes

as rotation axes were recorded on a cylindrical film coaxial with the rotation axis. The resulting diffraction pattern with layer lines is shown in Fig. 4.2 and the data are shown in tables 4.1 and 4.2. The mean cell parameters a and b obtained from the rotation photograph using eq. (19) and (20) are

$$a = 7.41 \ A^{\circ}$$

$$b = 13.48 \ A^{\circ}$$

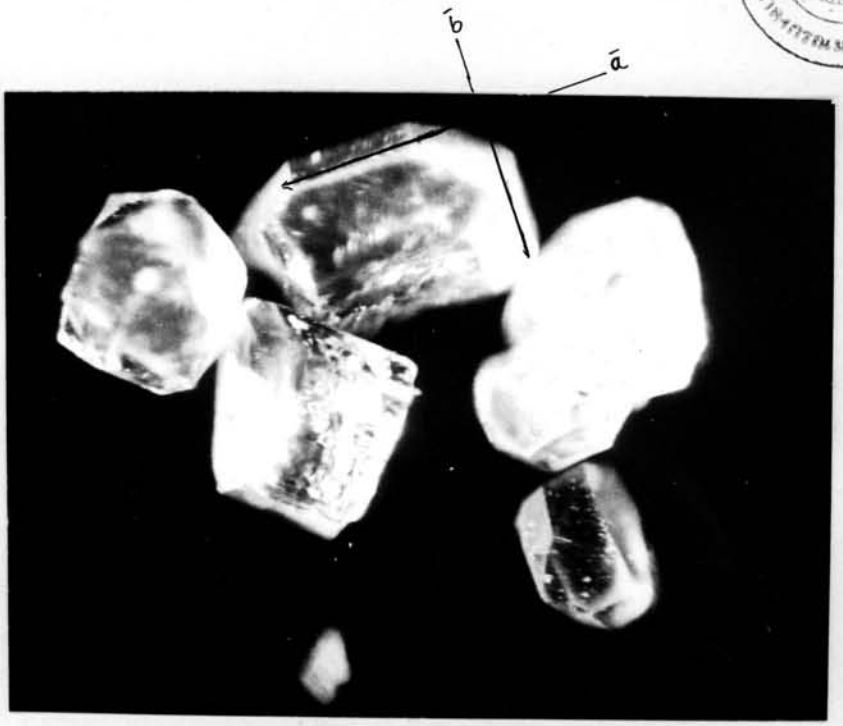

Fig. 4.1 Crystal of Latifolin ($C_{17}H_{18}O_4$) with the chosen axes $\bar{a}$ and $\bar{b}$.

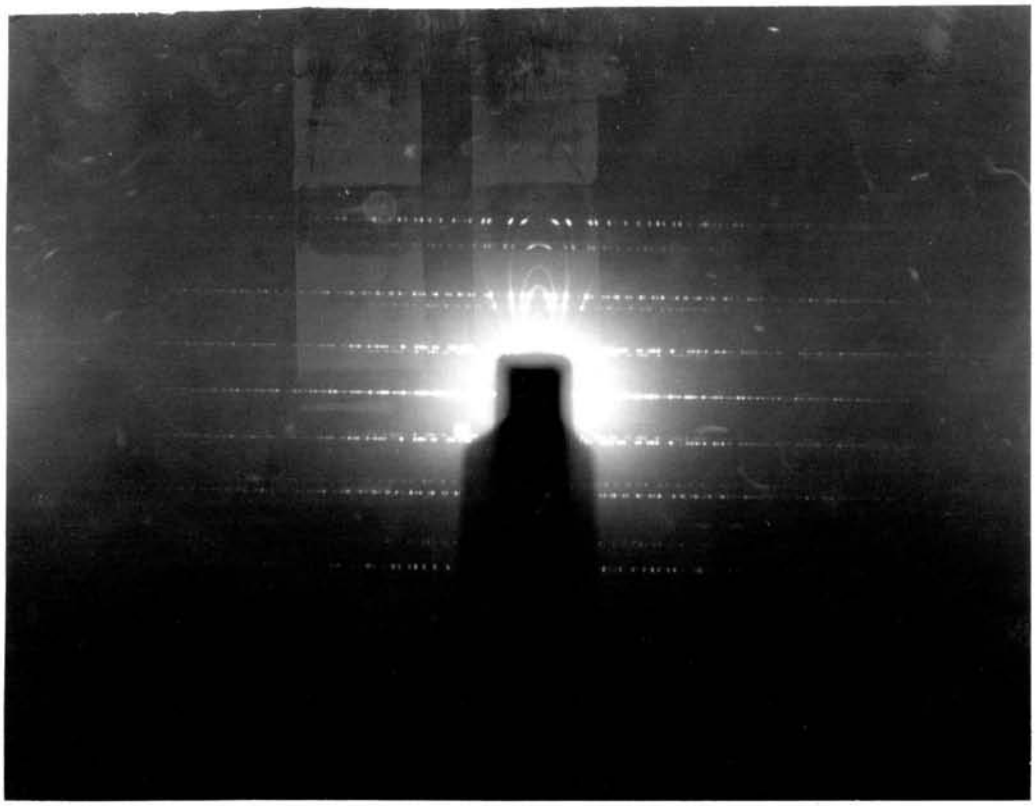

Fig. 4.2 Rotation photographs of Latifolin ($C_{17}H_{18}O_4$)
(a) 100 Rotation axis; CuK -radiation.
(b) 010 Rotation axis; CuK -radiation.

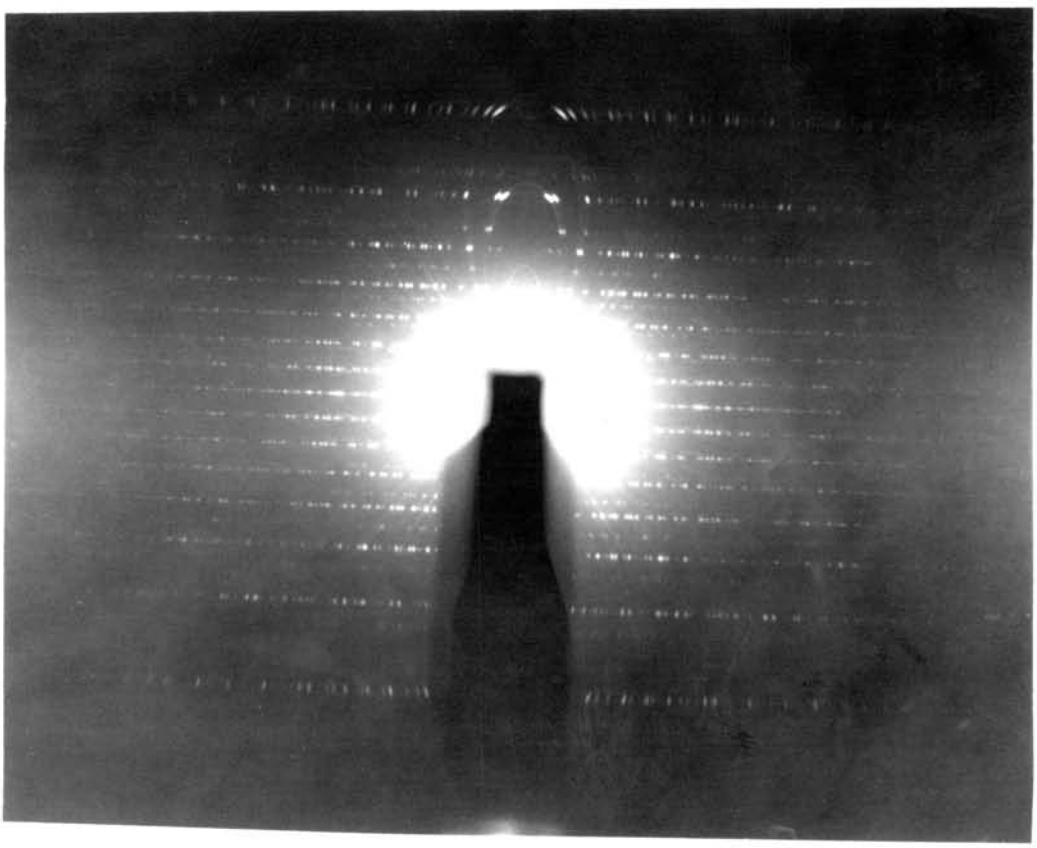

Table : 4.1

Determination of $\bar{a}$ axis length from [100] rotation photograph

layer	2m mm.	m mm.	tan ν = m/r	ν	$\sin\nu$	a/n = $\lambda/\sin\nu$	a (Å)
Cu K_α radiation, λ = 1.5418 Å							
1	12.20	6.10	.2129	12.2°	.2082	7.405	7.41
2	26.20	13.60	.4572	24.57°	.4159	3.707	7.41
3	45.95	22.975	.8019	38.72°	.6255	2.465	7.40
Cu K_β radiation , λ = 1.3922 Å							
1	10.97	5.485	.1914	10.83°	.1880	7.405	7.41
2	23.20	11.60	.4049	22.05°	.3754	3.709	7.42
3	39.20	19.60	.6841	34.38°	.5640	2.468	7.41

$$r = \frac{57.3}{2} \text{ mm} = 28.65 \text{ mm.}$$

Table 4.2

Determination of $\bar{b}$ axis length from [010] rotation photograph

layer	2m mm.	m mm.	tan ν = m/r	ν	ζ = sin ν	b/n = $\lambda/\sin\nu$	b (Å)
Cu K_α - radiation, λ = 1.5418 Å							
1	6.60	3.30	.1152	6.57°	.1144	13.477	13.48
2	13.45	6.725	.2347	13.21°	.2286	6.745	13.49
3	20.95	10.475	.3656	20.08°	.3434	4.490	13.47
4	29.45	14.725	.5140	27.20°	.4572	3.373	13.49
Cu K_β radiation, λ = 1.3922 Å							
1	5.95	2.975	.1038	5.93°	.1033	13.48	13.48
2	12.10	6.05	.2112	11.93°	.2067	6.735	13.47
3	18.675	9.3375	.3259	18.05°	.3098	4.494	13.48
4	25.975	12.9875	.4533	24.40°	.4132	3.370	13.48

$$r = \frac{57.3}{2} \text{ mm} = 28.65 \text{ mm.}$$

Table 4.3

Necessary parameters for taking equi-inclination Weissenberg photographs. Values of ζ were taken from table 4.1 and 4.2 .

Layer	ζ (r.l.u.)	$\zeta/2$ (r.l.u.)	μ	tan μ	$s=r_s \tan \mu$ $r_s=24.23$ mm.
[100] Rotation axis					
1	.2028	.1041	5.97°	.1046	2.5
2	.4159	.2080	12°	.2127	5.2
3	.6255	.3126	18.45°	.3293	8.0
[010] Rotation axis					
1	.1144	.0572	3.23°	.0573	1.4
2	.2286	.1143	6.57°	.1151	2.8
3	.3434	.1717	9.89°	.1743	4.2

IV.2 Taking the Weissenberg photograph

A Nonius Weissenberg camera with diameter of 57.3 mm. and a translation of 1 mm. per 2° of rotation was used with CuK_{α} -radiation ($\lambda_{K_{\alpha}} = 1.5418\ A^{\circ}$).

2.1 A normal - beam Weissenberg photograph was taken with $\bar{a}$ and $\bar{b}$ rotation axes as section IV.1 for the zero-order layer Weissenberg photograph as shown in Fig. 4.3 (a) and (e).

2.2 An equi-inclination Weissenberg photograph was taken with the necessary parameters derived from rotation photographs in table 4.3. The first, second and third layer equi-inclination Weissenberg photograph were taken with $\bar{a}$ and $\bar{b}$ as rotation axes, as shown in Fig. 4.3 (b), (c), (d) for $\bar{a}$ rotation axis and 4.3 (f), (g), and (h) for the $\bar{b}$ rotation axis.

The reciprocal net of each layer was constructed from reflections on these photographs by measuring the angle , and the reciprocal lattice coordinate $\frac{\xi}{\delta}$ of each diffracted spot on the film using a so called the reciprocal lattice unit triangle. The cooresponding data are presented in an appendix and the reciprocal nets with indicies of each spots can be seen in Fig. 4.4 (a)$\longrightarrow$(h).

From these reciprocal nets, the reciprocal axes and the interaxial angles were chosen :

1) from the Weissenberg photograph of the $\bar{a}$ rotation axis as

$$b^* = .115 \quad \text{r. } \ell\text{. u.}^*$$

$$c^* = .10 \quad \text{r. } \ell\text{. u.}$$

and

$$\alpha^* = 90^\circ ,$$

2) from the Weissenberg photograph of the $\bar{b}$ rotation axis

$$a^* = .21 \quad \text{r. } \ell\text{. u.}$$

$$c^* = .10 \quad \text{r. } \ell\text{. u.}$$

and

$$\beta^* = 90^\circ .$$

The indicies of reflected spots from the construction of the reciprocal lattice point of Fig. 4.4 (a)——>(h) are shown in table 4.4 (a)——>(h) (in the appendix).

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* The 'r. ℓ. u.' stands for the reciprocal lattice unit.

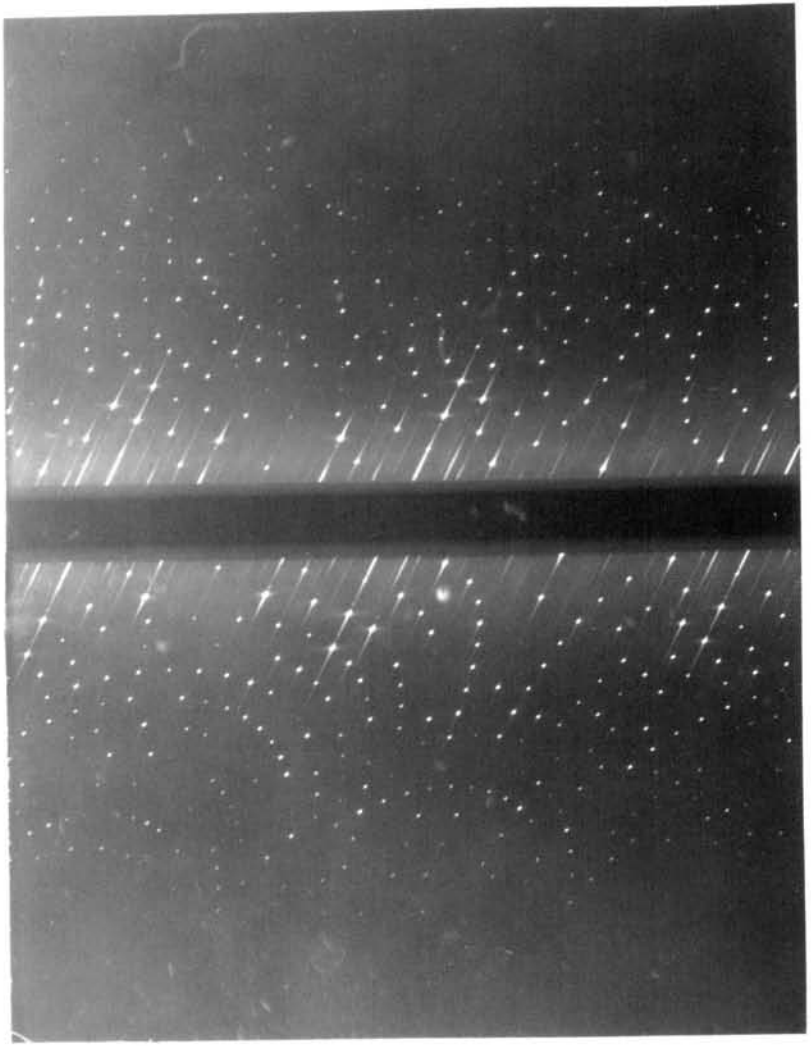


Fig. 4.3 (a) The zero-level Weissenberg photograph of Latifolin. [100] Rotation axis; CuK_{α} -radiation.

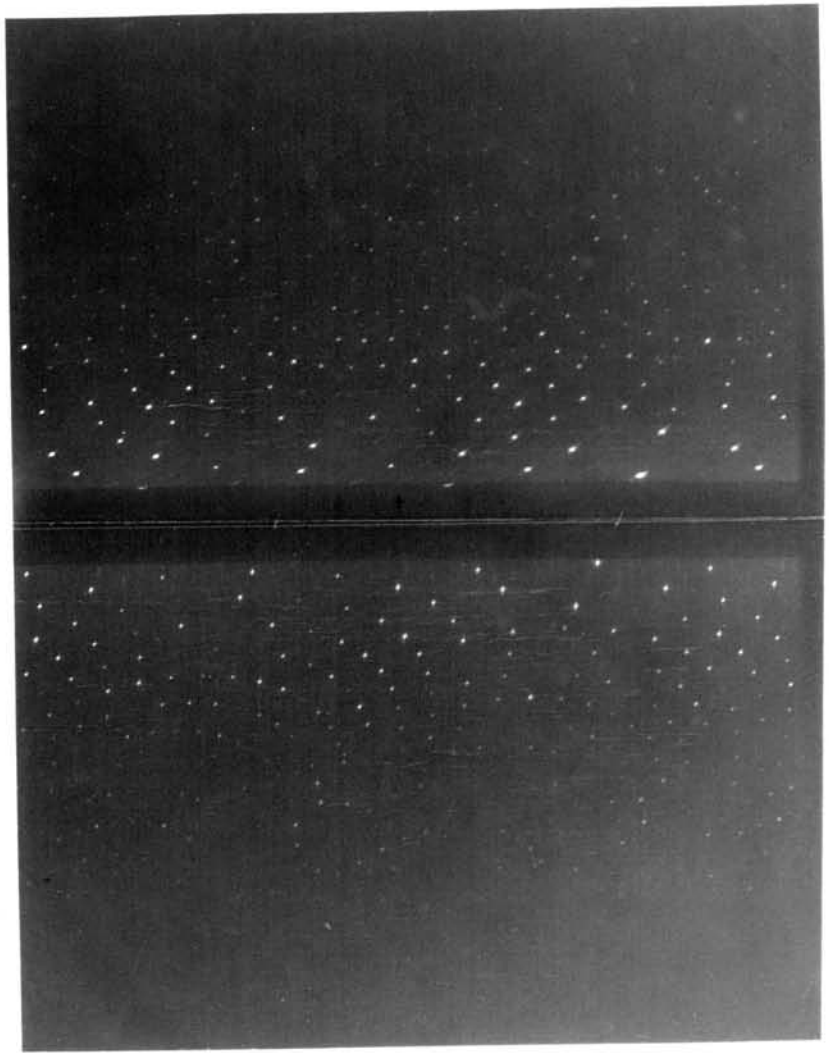


Fig. 4.3 (b) The first-level Weissenberg photograph of Latifolin. [100] Rotation axis; CuK_{α} -radiation.

Fig. 4.4 (a) The reciprocal net of indicies o k l [100] Rotation axis, 0th layer, with h = 0

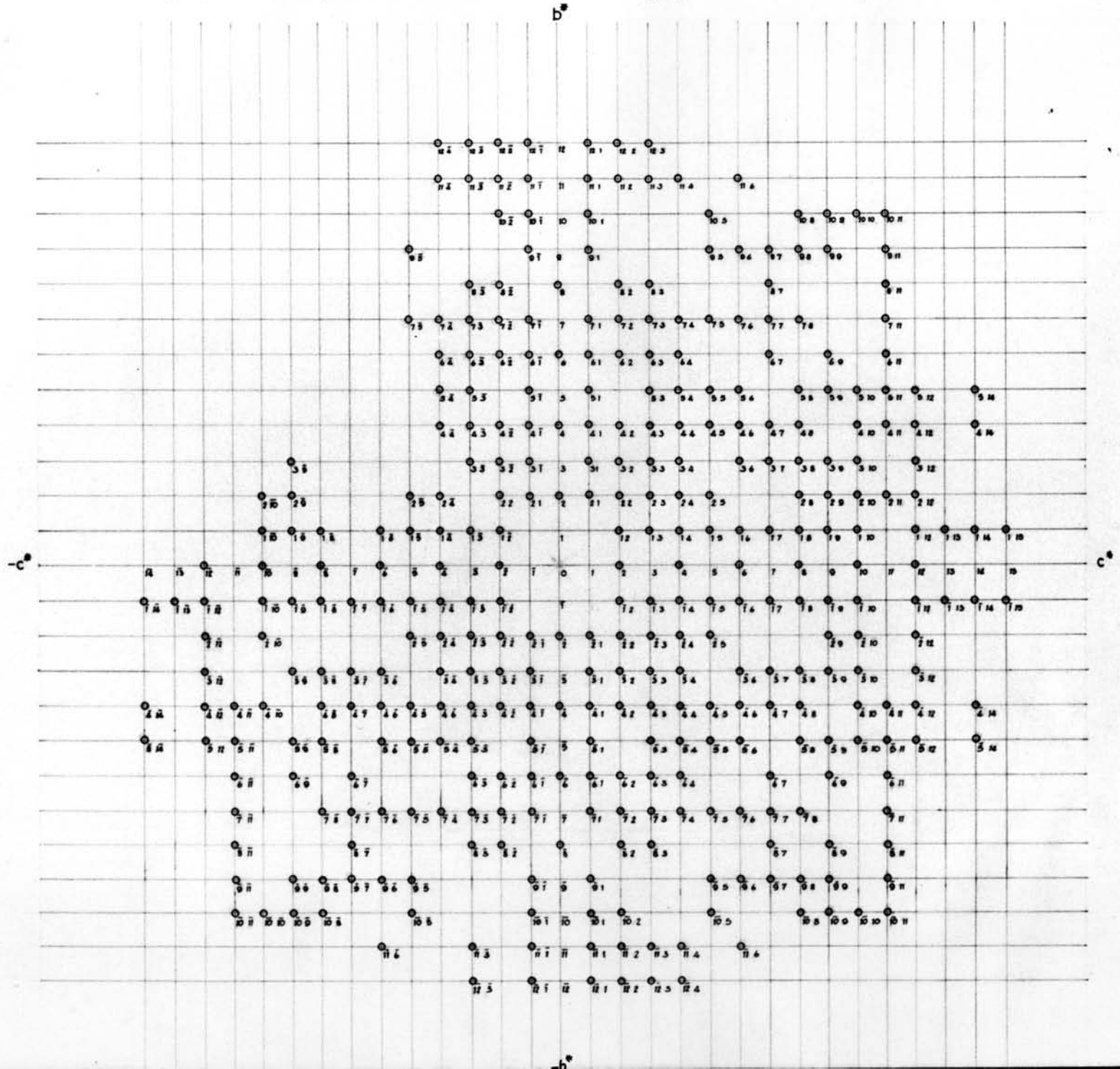
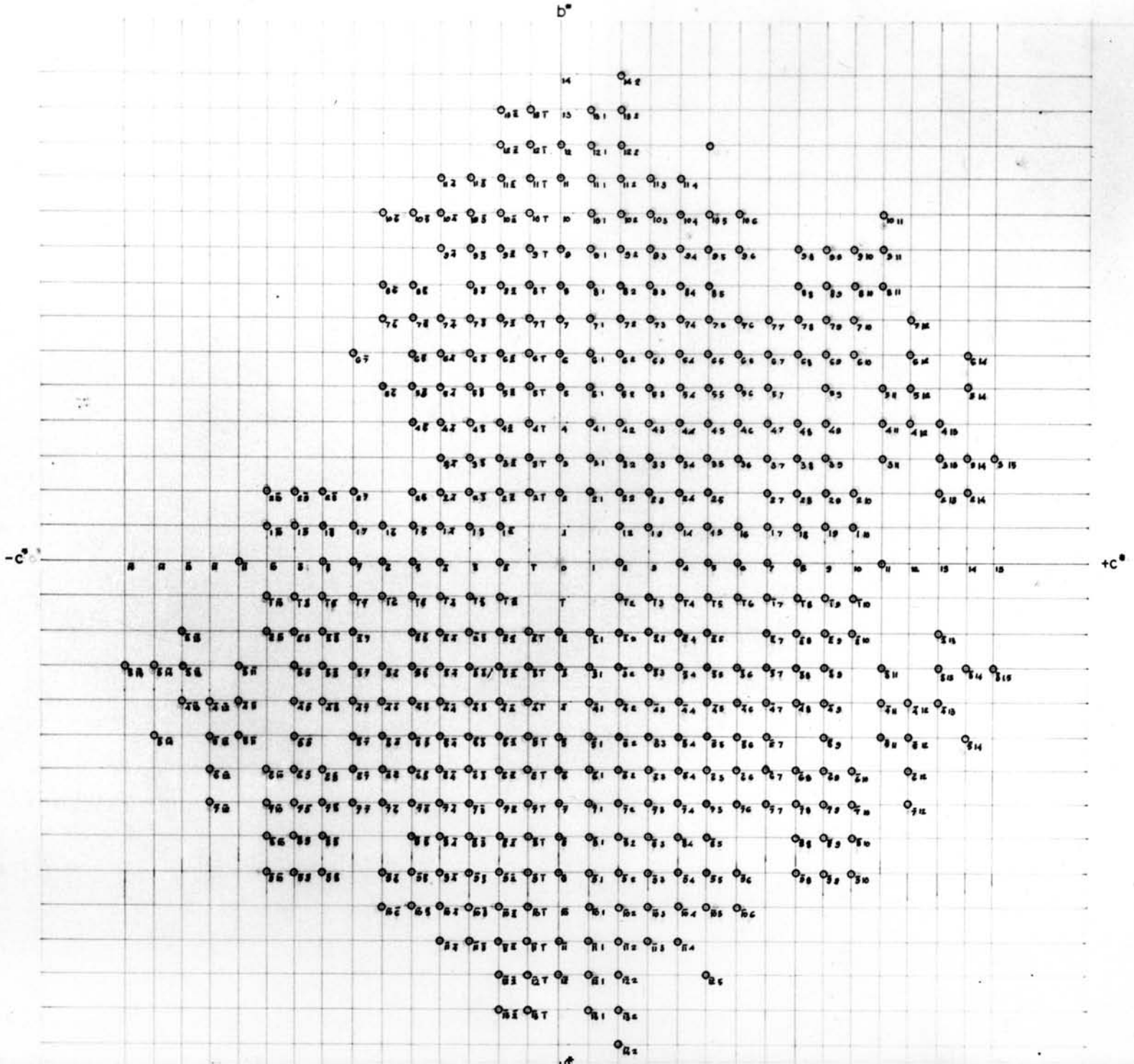


Fig 44 (b) The reciprocal net of indicies 1 k l [100] Rotation axis, 1st layer, with h = 1



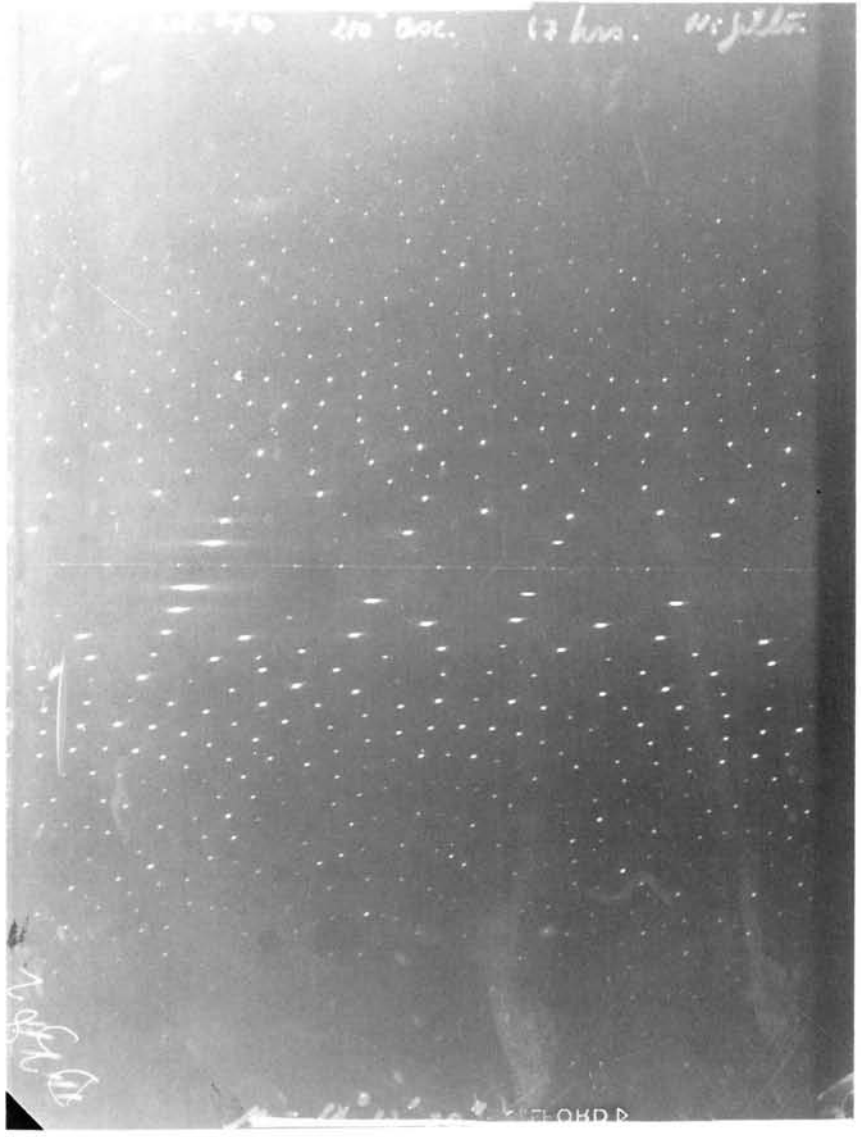


Fig.4.3(d) The third-level Weissenberg photograph of Latifolin. [100] Rotation axis; Cuk$_{\alpha}$ -radiation.

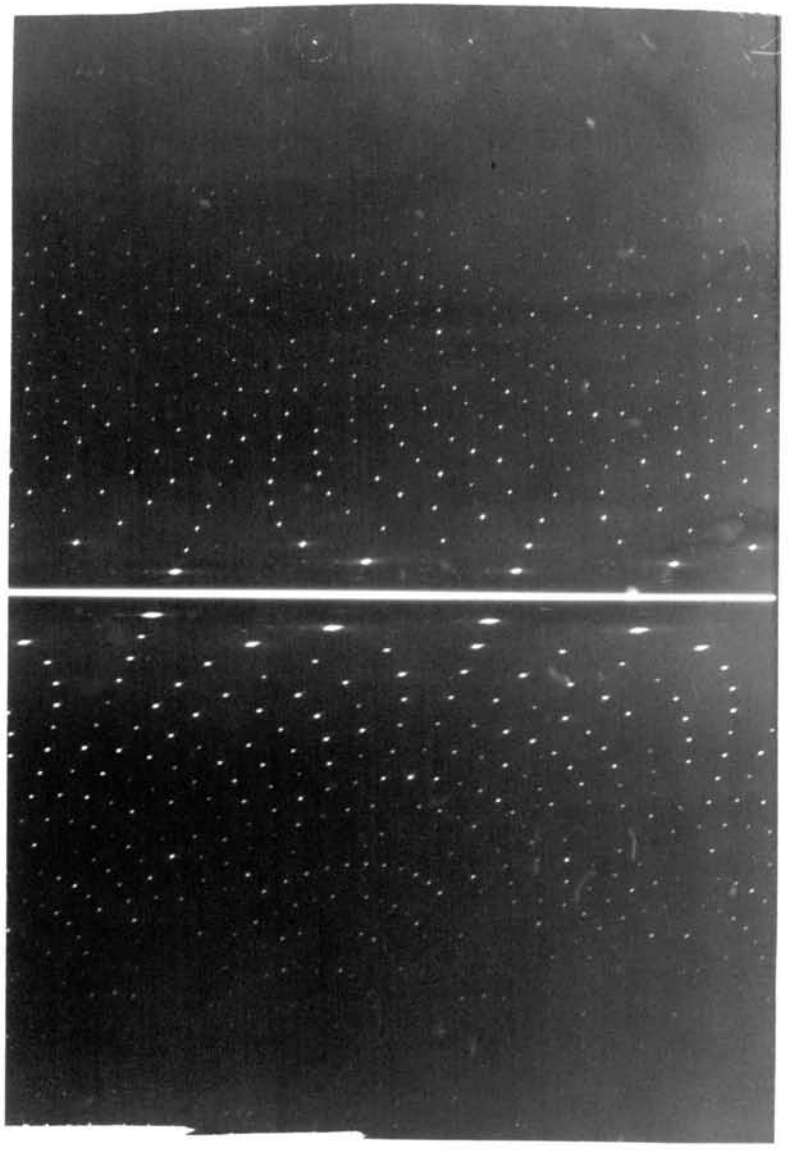


Fig.4.3(c) The second-level Weissenberg of photograph of Latifolin. [100] Rotation axis; CuK$_{\alpha}$ -radiation.

Fig. 4.4 (c) The reciprocal net of indicies 2 k l [100] rotation axis, 2nd layer, h = 2

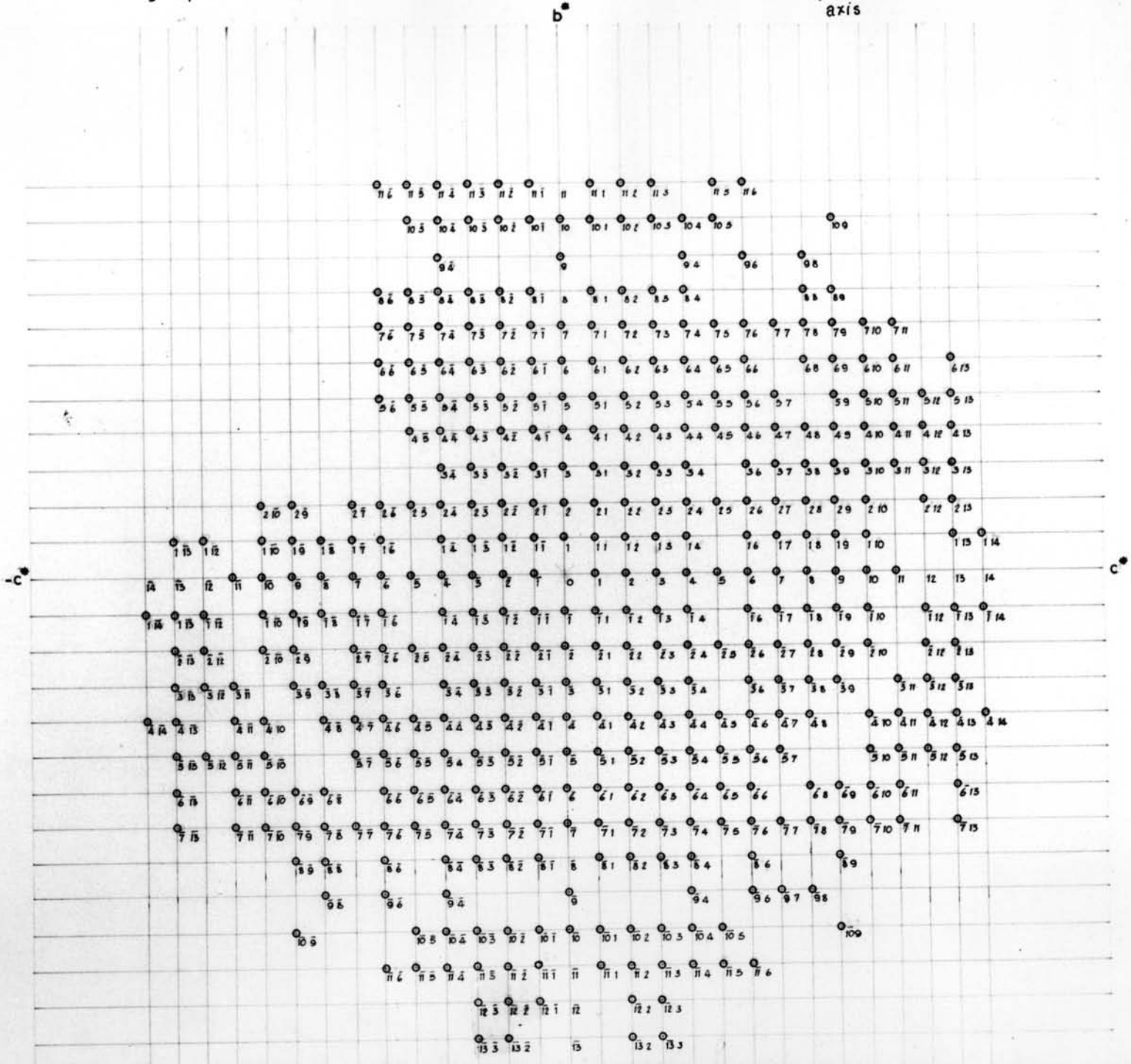
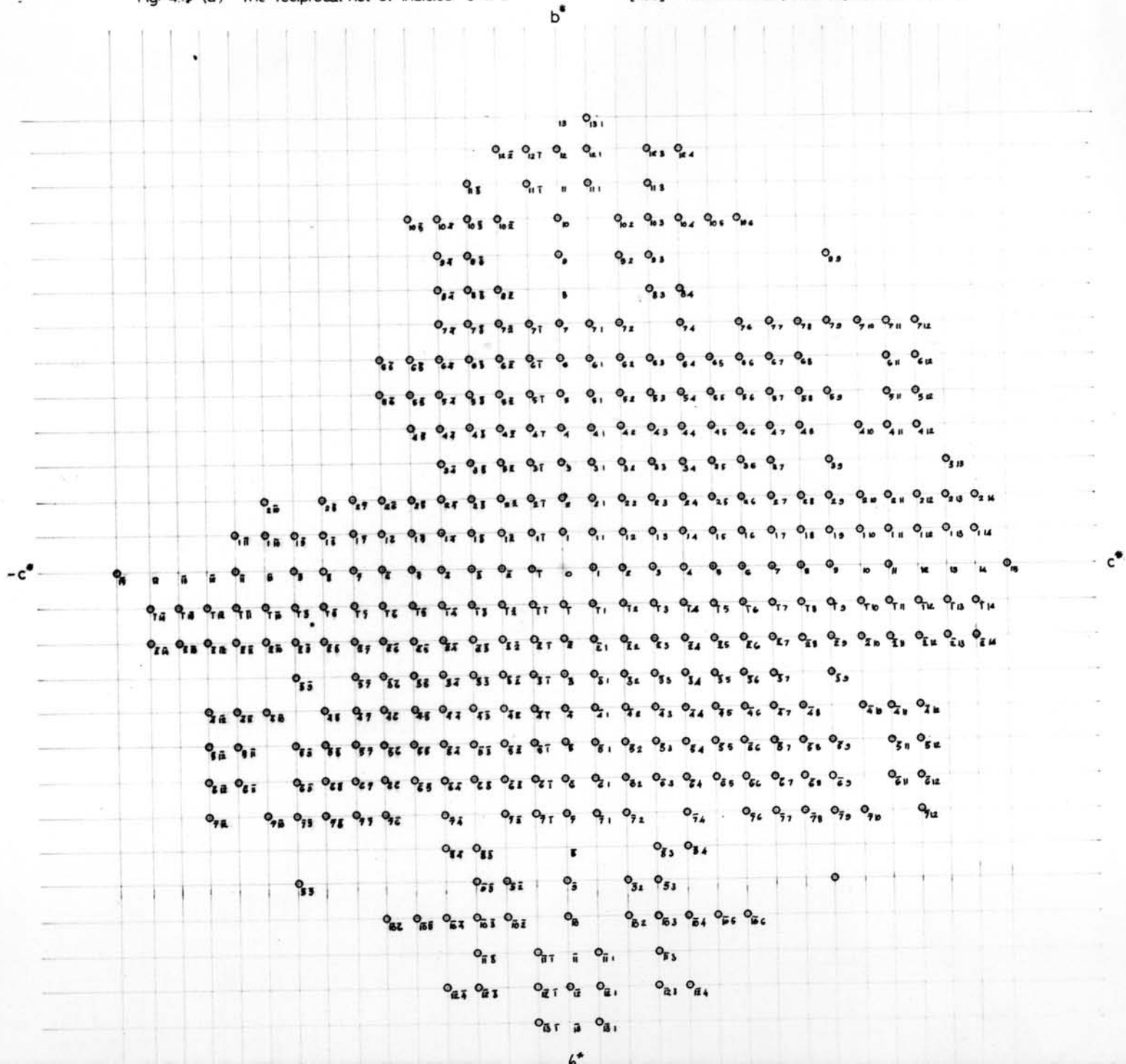


Fig. 4.4 (d) The reciprocal net of indicies 3 k l [100] Rotation axis, 3rd layer, with h = 3



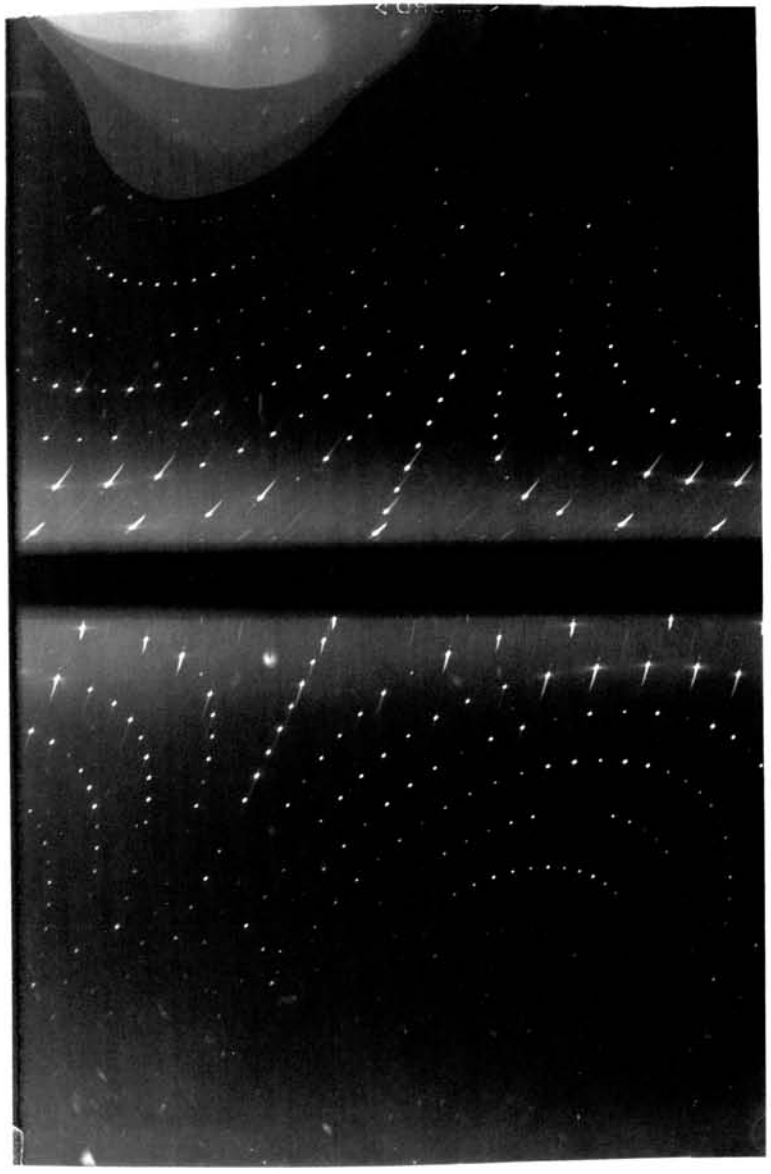


Fig.4.3(f) The first-level Weissenberg photograph of Latifolin. 010 Rotation axis; CuK -radiation.

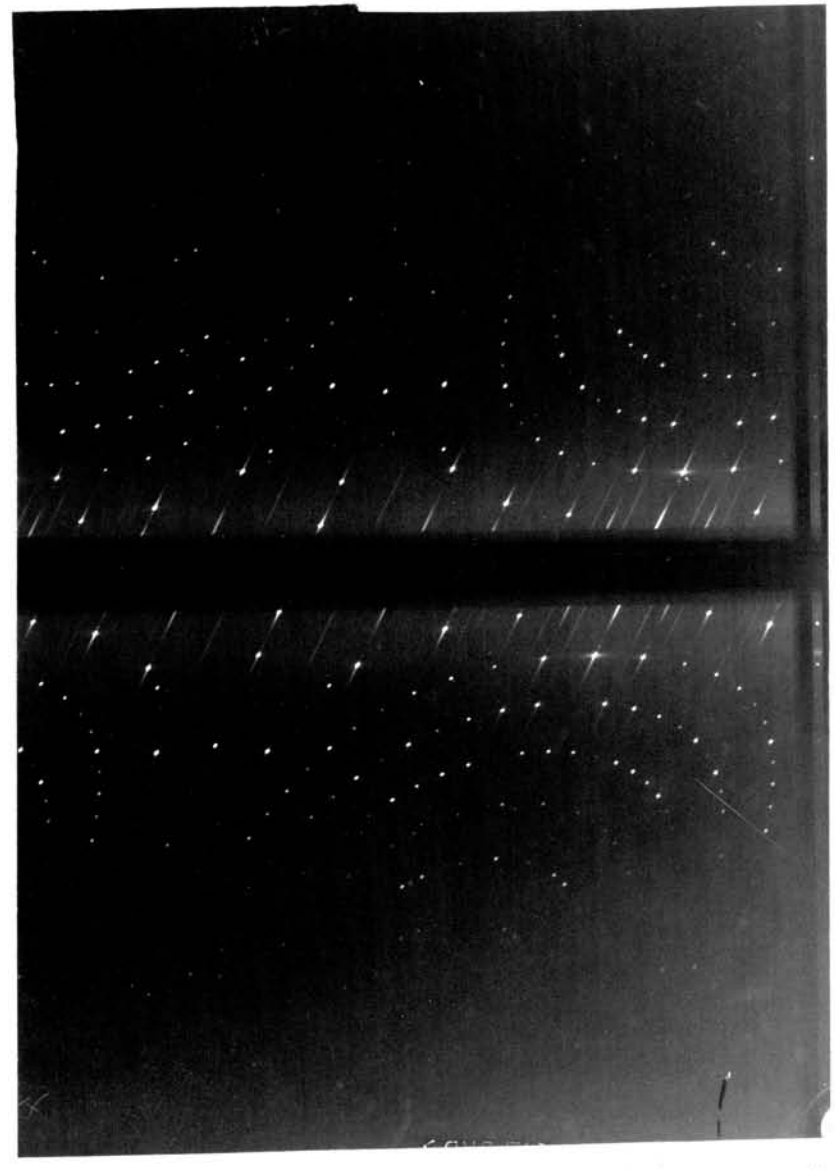


Fig.4.3(e) The zero-level Weissenberg photograph of Latifolin. 010 Rotation axis; CuK -radiation.

Fig 4.4 (e) The reciprocal net of indicies h o l [010] Rotation axis, 0th layer, with k = 0

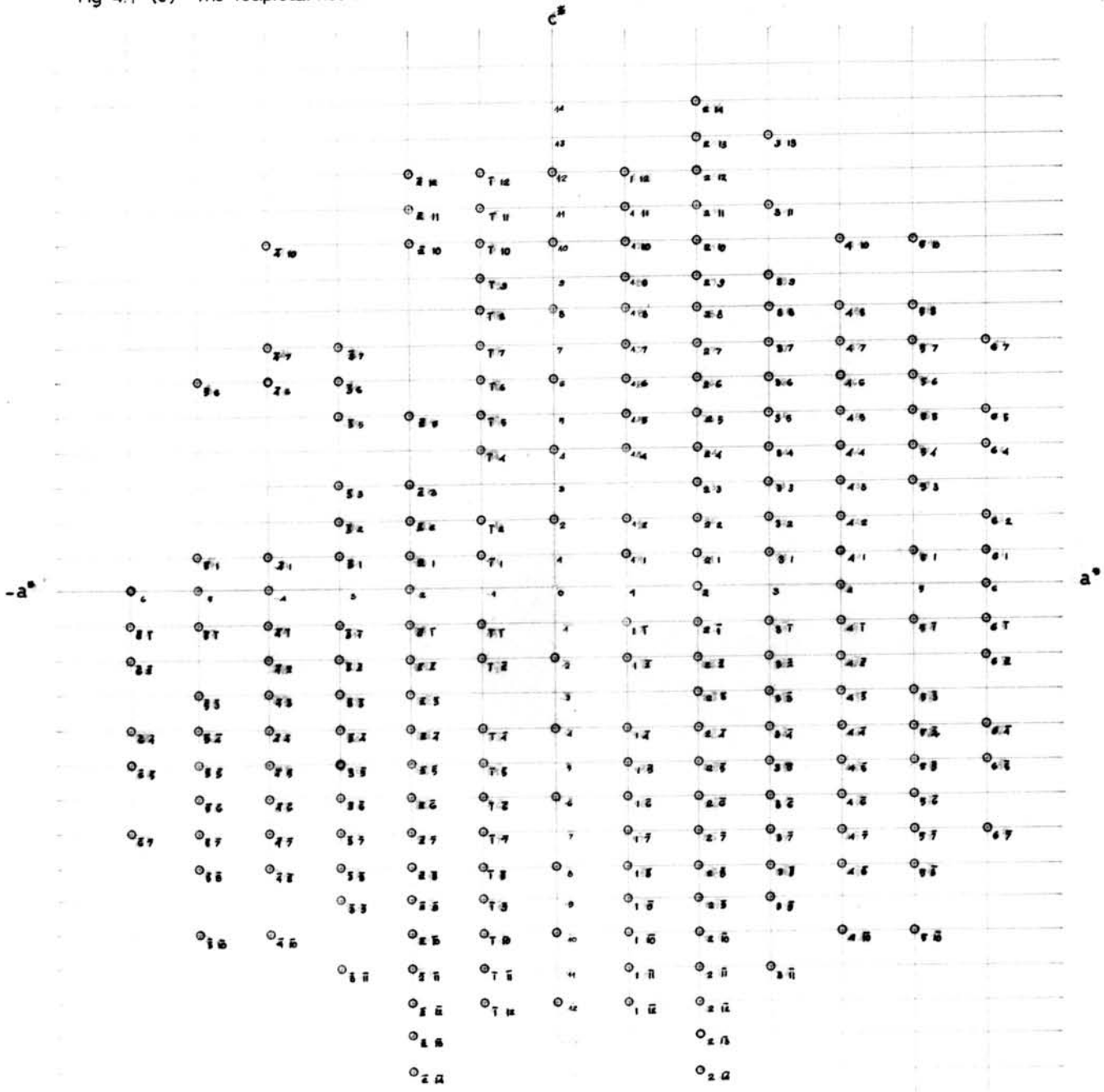
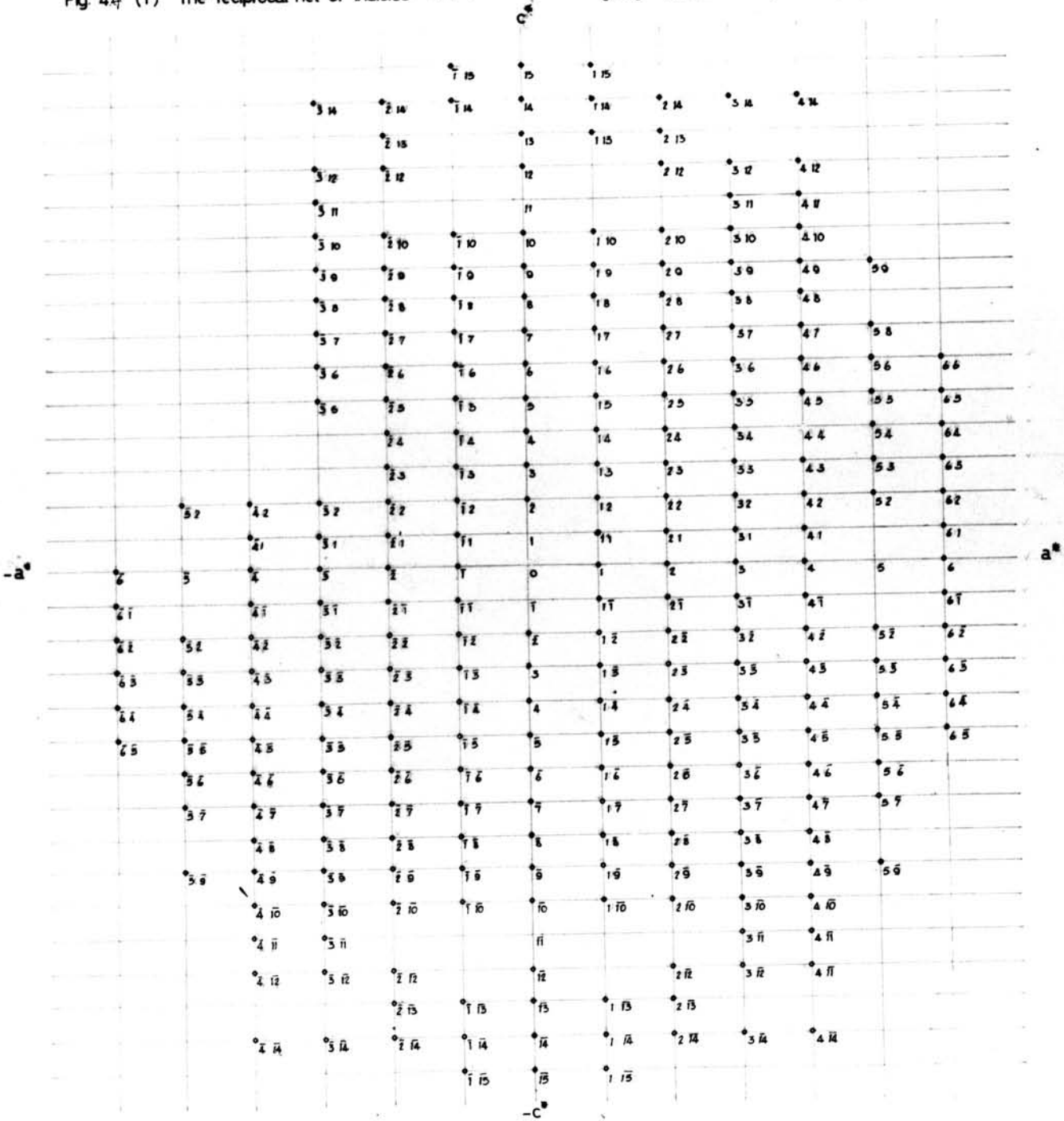


Fig. 44 (f) The reciprocal net of indicies h 1 l [010] Rotation axis, 1st layer, with k = 1



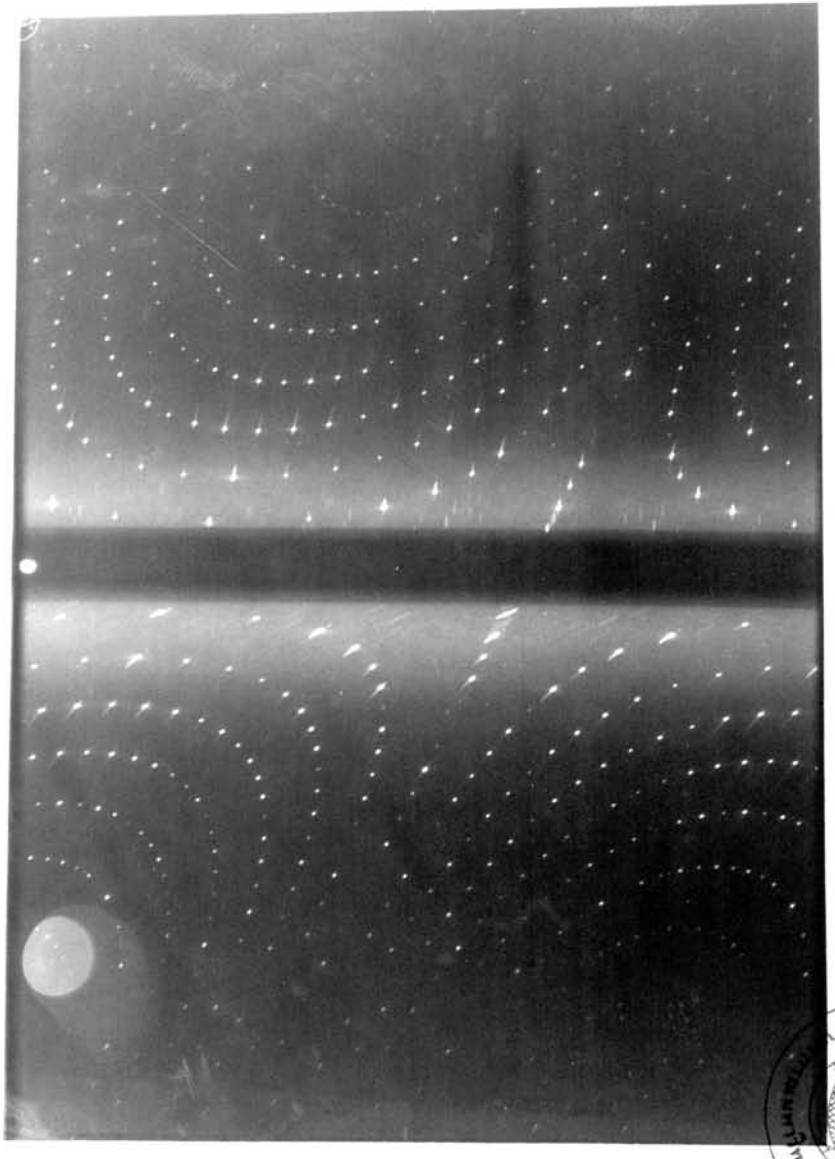


Fig.4.3(g) The second-level Weissenberg photograph of Latifolin. 010 Rotation axis; CuK -radiation.

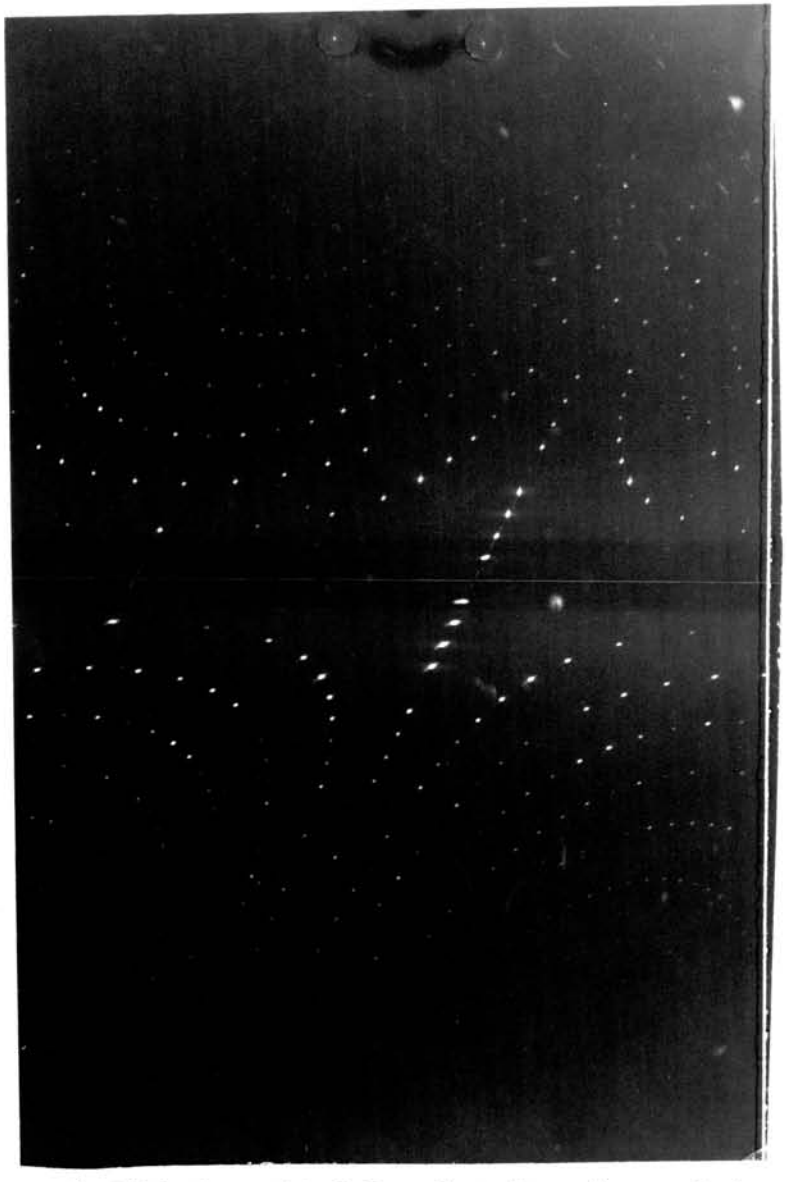
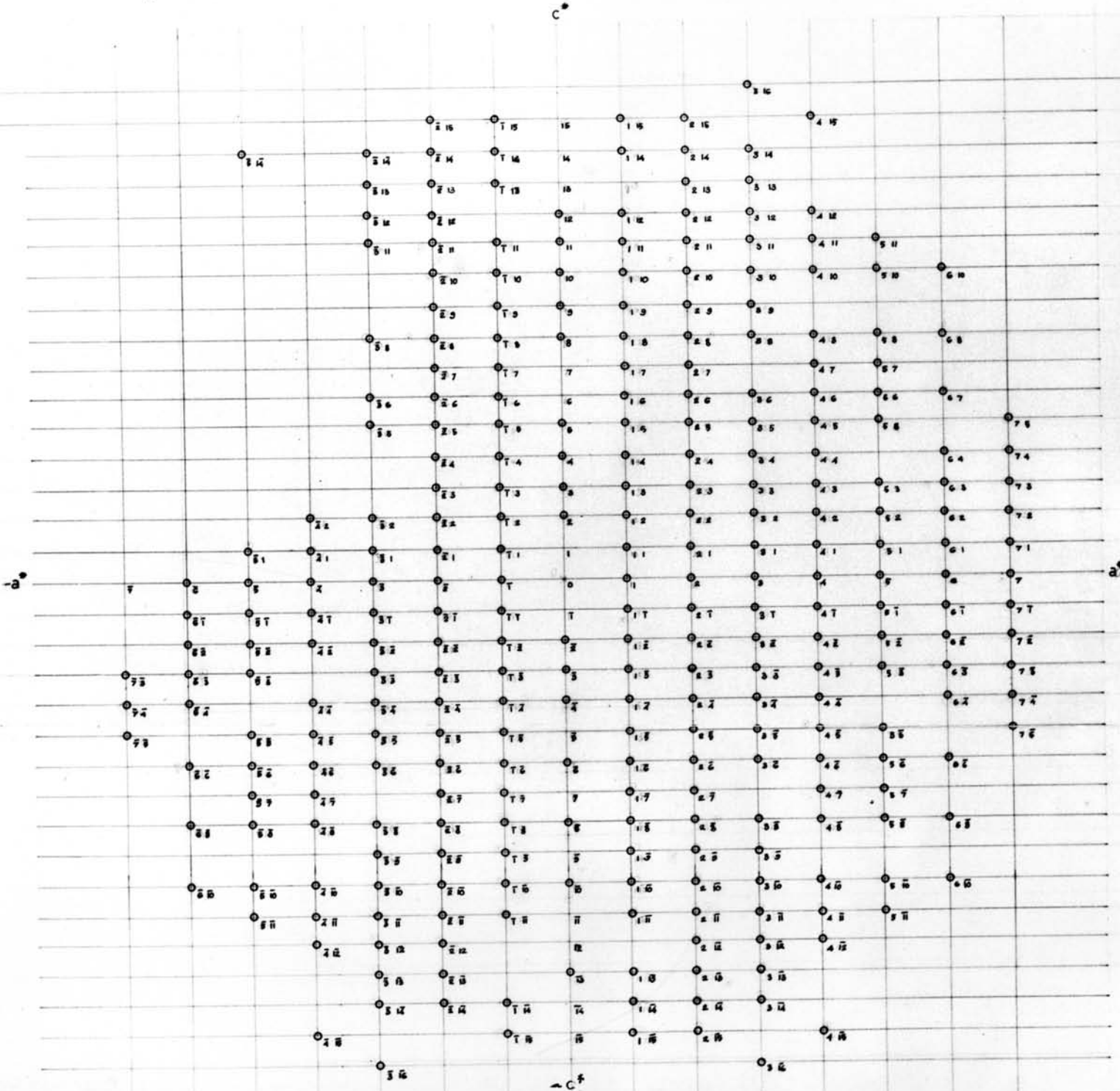


Fig.4.3(h) The third-level Weissenberg photograph of Latifolin. 010 Rotation axis; CuK -radiation.

Fig. 4.4 (g) The reciprocal net of indicies h 2 l [010] Rotation axis, 2nd layer, with $k_2 = 2$



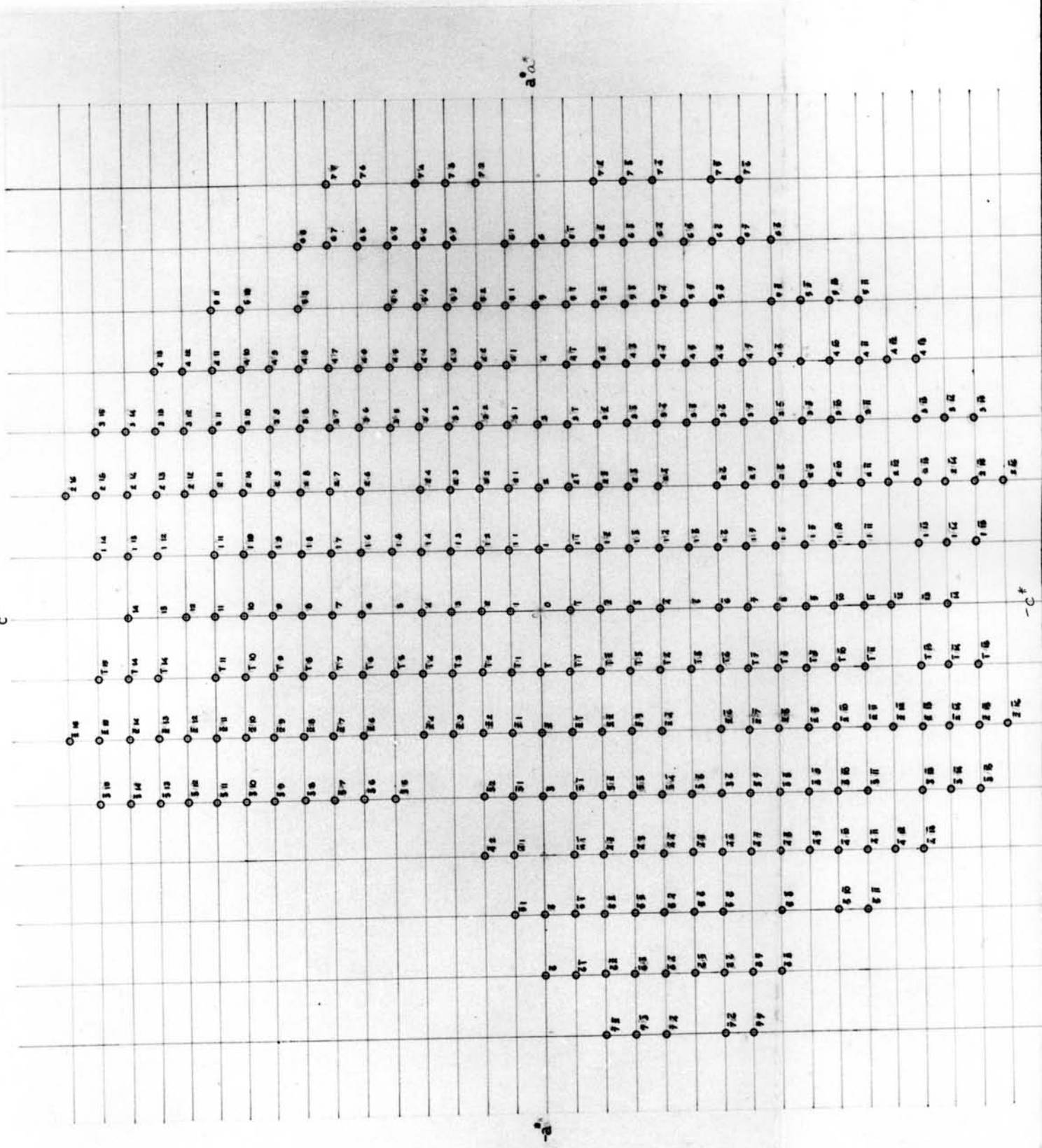


Fig. 4.4 (h) The reciprocal net of indicies h 3 l [010] Rotation axis, 3 rd layer, with k = 3

IV.3 Finding a unit cell

The unit cell parameters of the crystal lattice were calculated from the results of section IV.1 and IV.2 and using the relationship of the reciprocal lattice and the direct lattice from International Tables fro X-Ray Crystallography[5] as shown in table 4.5. It is an orthorhombic crystal with Laue symmetry m m m.

Table 4.5

Unit cell dimensions

Reciprocal lattice*	Direct lattics
$a^* = .21$ r.l.u. $\alpha^* = 90^\circ$	$a = 7.34\ A^\circ$ $\alpha = 90^\circ$
$b^* = .115$ r.l.u. $\beta^* = 90^\circ$	$b = 13.47\ A^\circ$ $\beta = 90^\circ$
$c^* = .10$ r.l.u. $\gamma^* = 90^\circ$	$c = 15.42\ A^\circ$ $\gamma = 90^\circ$
From table 4.1 and table 4.2 $a = 7.41\ A^\circ$, $b = 13.48\ A^\circ$	
Average cell dimensions from rotation and Weissenberg photographs : $a = 7.38\ A^\circ \pm 0.01\ A^\circ$ $\alpha = 90^\circ$; $b = 13.48\ A^\circ \pm 0.03\ A^\circ$ $\beta = 90^\circ$; $c = 15.42\ A^\circ \pm 0.15\ A^\circ$ $\gamma = 90^\circ$	

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5 International Tables for X-Ray Crystallography, (Vol.II; Bermingham : Kynoch Press, 1967), p.p. 108.

* The measurements are based on the assumed error of the equipment with $\pm$ 0.2 mm.

IV.4 Determining a space group of Latifolin ($C_{17}H_{18}O_4$)

Table 4.4 was used together with table 2.6 to construct table 4.6

Table 4.6

Determination of space group of Latifolin ($C_{17}H_{18}O_4$)

Class of Reflection	Condition for nonextinction n = integer	Interpretation of extinction	Symbol
h k ℓ	no conditions	Primitive	p
0 k ℓ	no conditions	-	-
h 0 ℓ	no conditions	-	-
h k 0	no conditions	-	-
h 0 0	h = 2n	[100] screw axis comp. a/2	2_1
0 k 0	k = 2n	[010] screw axis comp. b/2	2_1
0 0 ℓ	ℓ = 2n	[001] screw axis comp. c/2	2_1
Point Group			2 2 2
Laue Group			m m m
Space Group			$P2_1$ 2_1 2_1

IV.5 Determination of the density and the number of molecules per unit cell (N) of Latifolin ($C_{17}H_{18}O_4$)

Flotation method[6] was used to find the density of Latifolin ($C_{17}H_{18}O_4$) by using the mixture of :

1) carbon tetrachloride (d = 1.60 g/cm^3 at 25° C)
   and kerosene (d = 0.79 g/cm^3 at 25° C),

2) bromobenzene (d = 1.40 g/cm^3 at 25° C)
   and kerosene (d = 0.79 g/cm^3 at 25° C).

The crystal of Latifolin neither rises nor sinks in the miscible solution of

1) carbon tetrachloride and kerosene,

2) bromobenzene and kerosene,

in the proportion of x : y. The observed density, the number of molecules per unit cell (N) calculated from the observed density, the molecular formula ($C_{17}H_{18}O_4$), and the unit cell dimensions from table 4.5, and the calculated density were shown in table 4.7.

6 International Tables for X-Ray Crystallography (Vol.III; Bermingham : Kynoch Press, 1962), p.p.18.

Table 4.7

Densities and the number of molecules per unit cell (N) of Latifolin ($C_{17}H_{18}O_4$)

Observed at room temperature 27.5°C	Observed density g/cm^3	$N = \frac{D_{obs} \times V \times A}{mol.mass.}$	Calculated density $D = \frac{N \times mol.mass.}{V \times A}$ g/cm^3
Carbon tetrachloride and kerosene with $V_1 : V_2 = 1.20 : 1$	1.233		
		3.998	
Bromobenzene and kerosene with $V_1 : V_2 = 2.29 : 1$	1.235	which implies	
	$D_{ave} = 1.234$	4	1.235

N = number of molecules per unit cell

V = volume (Taken from table 4.5) = 1540.2×10^{-24} cm^3

A = Avogadro's number = 6.02×10^{23}

mol.mass = molecular mass of $C_{17}H_{18}O_4$ = 286.161 a.m.u.

IV.6 Refinement of the unit cell dimensions of Latifolin ($C_{17}H_{18}O_4$)

The refinement was taken from the powder photograph of Latifolin ($C_{17}H_{18}O_4$) taken at University of Uppsala, Sweden. A Guinier - Hägg focusing powder camera with Cr - K_{α_1} radiation (λ = 2.28962 A°)

The unit cell dimensions of Latifolin after the least square refinement based on 47 reflections using a computing program with IBM 1800 were shown in table 4.8.

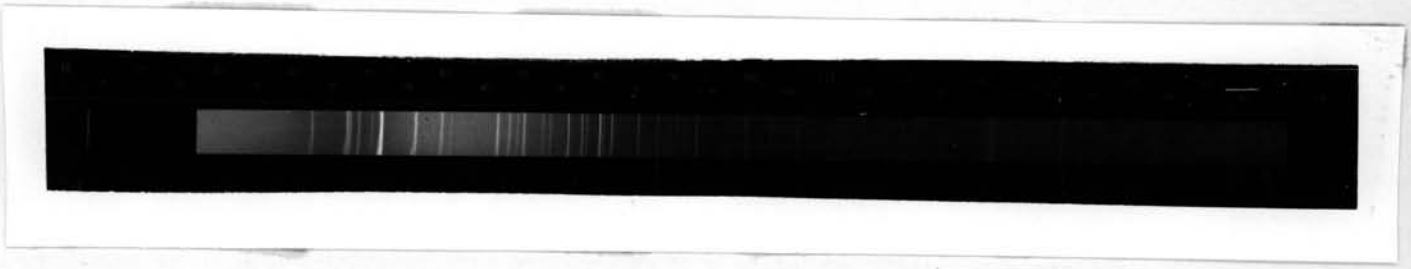

Fig. 4.5 The powder photograph of Latifolin ($C_{17}H_{18}O_4$).

Table 4.8

Refinement of cell dimensions

$a = 7.3887 \pm 0.0006$ A°

$b = 13.4581 \pm 0.0008$ A°

$c = 15.6157 \pm 0.0010$ A°

Volume = 1552.8 A°

h	k	ℓ	$\sin^2\theta_{obs}$	$\sin^2\theta_{cal}$	$\Delta = (\sin^2\theta_{obs} - \sin^2\theta_{cal})\ 10^{-5}$
0	0	2	0.021479	0.021498	- 1.9
0	1	2	0.028712	0.028734	- 2.2
1	0	1	0.029382	0.29381	0.1
1	1	0	0.031231	0.031242	- 1.0
0	2	1	0.034309	0.034318	- 0.9
1	1	1	0.036561	0.036617	- 5.5
1	0	2	0.045484	0.045505	- 2.0
0	1	3	0.055537	0.055607	- 7.0
0	3	1	0.070513	0.070498	1.4
1	2	2	0.074422	0.074449	- 2.6
0	2	3	0.077280	0.077315	- 3.5
1	1	3	0.079588	0.079613	- 2.4
0	3	2	0.086646	0.086622	2.4
1	3	0	0.089134	0.089130	0.4
2	0	0	0.096024	0.096027	- 0.2
2	1	0	0.103202	0.103263	- 6.0
2	1	1	0.108612	0.108637	- 2.5
1	0	4	0.110077	0.109999	7.7
0	3	3	0.113444	0.113495	- 5.1
0	2	4	0.114840	0.114937	- 9.6
0	4	1	0.121156	0.121150	0.5
2	1	2	0.124796	0.124761	3.5
1	2	4	0.138881	0.138943	- 6.1
0	1	5	0.141656	0.141600	5.6
1	4	1	0.145193	0.145157	3.5
0	3	4	0.151116	0.151117	- 0.0
0	2	5	0.163367	0.163308	5.8
1	1	5	0.165624	0.165606	1.8

h	k	ℓ	$\sin^2\theta_{obs}$	$\sin^2\theta_{cal}$	$\Delta = (\sin^2\theta_{obs} - \sin^2\theta_{cal})\ 10^{-5}$
2	3	1	0.166516	0.166525	- 0.8
1	3	3	0.175084	0.175123	- 3.8
1	2	5	0.187355	0.187314	4.0
1	4	3	0.188212	0.188154	5.8
0	4	4	0.201742	0.201769	- 2.7
2	3	3	0.209490	0.209522	- 3.2
3	0	1	0.221560	0.221435	12.5
1	4	4	0.225804	0.225776	2.8
2	2	5	0.259255	0.259335	- 8.0
3	2	2	0.266550	0.266503	4.7
1	4	5	0.274089	0.274147	- 5.7
1	3	6	0.282772	0.282615	15.6
1	5	4	0.290986	0.290900	8.9
2	5	2	0.298500	0.298425	7.5
1	6	2	0.305975	0.306001	- 2.6
2	5	3	0.325229	0.325298	- 6.9
1	6	3	0.332827	0.332874	- 4.6
1	0	8	0.367919	0.367978	- 5.8
1	5	6	0.398538	0.398391	14.6

$$\Sigma \Delta^2 = 1{,}542.19 \times 10^{-10}$$

$\Sigma \Delta^2$ represents the minimum values (among those of the other cycles) which determined the accuracy of the refined measurements.