CHAPTER IV

MEASUREMENTS AND RESULTS

IV.1 Taking Rotation Photographs

Transparent crystals of Latifolin (C_{17} H₁₈ O₄) were formed in various shapes and sizes. The largest crystal was approxi. mately 0.4 mm. long and has a cross section of 0.87 mm². From the external morphology of the crystal, two small single crystals with crystal axes \bar{a} and \bar{b} were chosen as follows :

ā or [100] axis along the long axis of the crystal, 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 ($\lambda_{K_{\chi}} = 1.5418 \text{ A}^{\circ}, \lambda_{K\beta} = 1.3922 \text{ A}^{\circ}$). 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} .

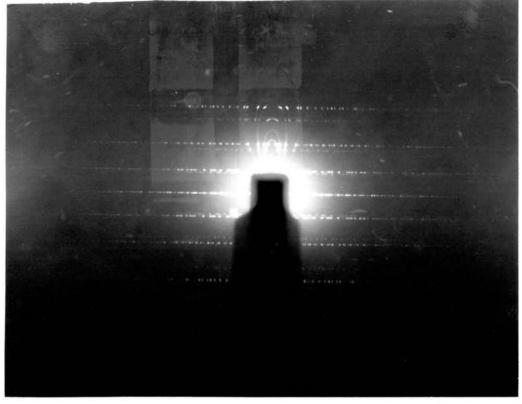


Fig. 4.2 Rotation photographs of Latifolin (C₁₇H₁₈O₄) (a) 100 Rotation axis; CuK -radiation. (b) 010 Rotation axis; CuK -radiation.

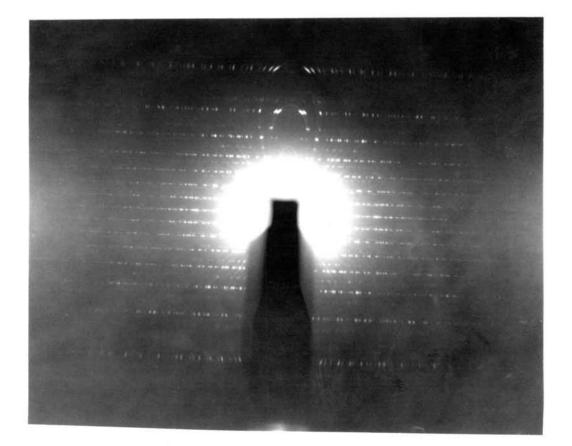


Table : 4.1

Determination	of	ła	axis	length	from	[100]	rotation	photograph	

layer	2m	m	tan V	r	B	a/n	a
	mm.	mm.	= m/r		= sin V	= Kin V	(A)
Cu Ka	radiation	, λ= 1	• L.5418 A		-		
l	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	radiatio	n, λ =	1.3922	•			
1	10.97	5.485	.1914	10.83	.1380	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

$$r = \frac{57 \cdot 3}{2}$$
 mm = 28.65 mm.

Γa	b.	le	4		2
				-	

Determination of b axis length from [010] rotation photograph

layer	2n mm.	m mn.	tan 💙 = m/r	Ŷ	s = sin v	$= \frac{b/n}{\gamma_{sin}}$	b • • • (A)
Cu K	- radiati	lon, A	= 1.5418	•• A			
l	6.60	3.30	.1152	ر•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 ß	radiati	lon, 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

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

Table 4.3

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

Layer	(r.l.u)	9/2 (r.l.u.)	μ	tan µ	s=r _s tan μ r _s =24.23 mm
[100]	 Rotation axi	ļ .s			
l	.2028	.1041	5.97	.1046	2.5
2	•4159	.2080	12°	.2127	5.2
3	.6255	.3126	18.45	.3293	8.0
[010]	 Rotation axi	l .s .			
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_{χ} -radiation ($\lambda_{K_{\chi}}$ = 1.5418 A°).

2.1 A normal - beam Weissenberg photograph was taken with a and 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 \overline{a} and \overline{b} as rotation axes, as shown in Fig. 4.3 (b), (c), (d) for \overline{a} rotation axis and 4.3 (f), (g), and (h) for the \overline{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 $\bigotimes^{(2)}$ 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 a rotation axis as

b*	=	.115	rl.	u.*
с*	=	.10	r. l.	u.

and

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

2) from the Weissenberg photograph of the b rotation axis

a*	=	.21	r.	l.	u.	
c *	=	.10	r.	l.	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).

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

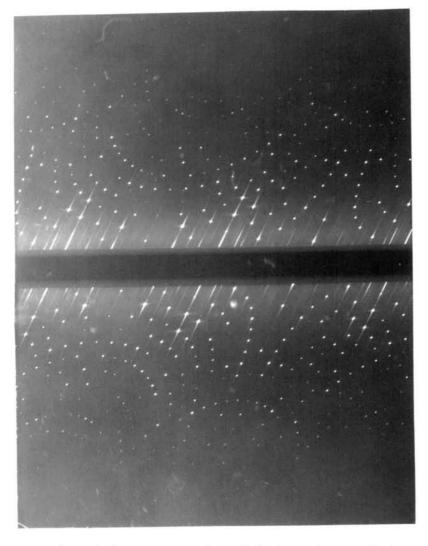


Fig. 4.3 (a) The zero-level Weissenberg photograph of Latifolin. [100] Rotation axis; Cuk, -radiation.

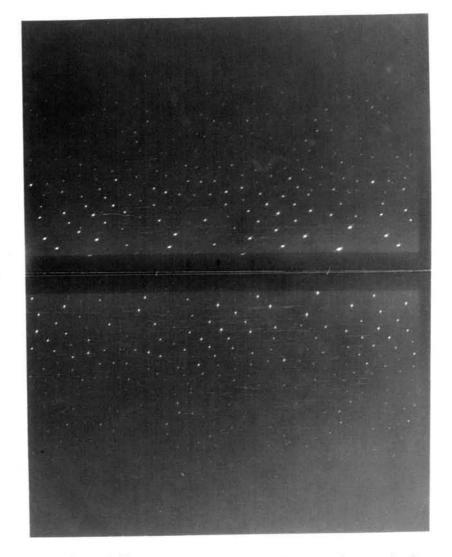


Fig. 4.3 (b) The first-level Weissenborg photograph of Latifolin. [100] Rotation axis; CuK_{χ} -radiation.

al al al a 2 1 .* : 1 0 . [100] Rotation axis, 0 th layer, with h = -2. 2. 2. 2. 2. 2. 5. 5. 5.0 5.0 5.0 5.0 21 30 9 0 0 0 0 0 1 0 M 3.8 -----8 80 80 8 B ----..... ā -1. 10 3. 3. 3. 3. 3. 3. 3. 5. 5. 5. 50 50 2 al .! . : ... 167 100 160 . : 9. 9. 9. 0. 0. 0. 0. 0. 0. 0° 10 · · · · · · · · · · · 3 5 4 6 2 de . -÷ - ----. - --* 13 0 0 0 0 0 0 V 0° ° 13 61 11 0 ----50 200 . 23 24 25 1 33 34 1 24 . 12 S. ut ut us us us 013 014 13 14 2 735 74 a 0 0 0 0 -2.04 .= --... --. 2 2 • 1 12 12 11 ----12 0. 0. 0. 0. 1 2 2 5 0 0 14 14 0² ... 0 k 54 54 54 -----1 2 1 11 21 43 4F 1. 2. 5 5 5 S 0 T 0 I The reciprocal net of indicies 22 22 22 27 42 1. .. 55 54 15 02 ... 17 21 1.0 12 55 40 64 49 66 65 ... 11 31 1. 11 -1 5 11 11 5E 23 -10. 10 44 44 48 --------10 Fig. 4.4 (a) -" T O 1 æ •

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••• ŝ 0 10 0 H 0 H Sea 9.4 9.15 E 2 8 0 KH x н = Ч 4 T T T 01. 01. 01.0 ÷ 1 0 H * -** NO Rotation axis, 1 st layer, with -............ ÷. 34 0. 0, 0, 0, 0, 1 0 0 0 N 0,1 0,1 0,4 0, 0, 0, 0, 0, 0, 0, 0, es 0.0 0.0 or. or. o. 02° 020 ", ", ", ", "," 60 020 02. 31. 91, 01. 01. Q. 91. Or. 41 41 44 45 45 44 41 41 41 87 31 40 ---..... ----0° 0° 1 -TA 75 016 047 4 31 38 St 47 64 025 026 067 ----Que Que Que Que Que Que mo Que Que Que Que Que 0,1 0,1 0,1 0,7 0, 0,1 0,1 0, 0,4 0,1 0,1 34 95 96 54 0 BS 066 3.0 ř 8. 3. 0. 0H 61 50 79 15 19 ---å Q. Q. Q. Q. Q. na 0.5 0. <u>8</u> ----... ň Qua Our Ou Ou Our A. 04. .n. 041 Qar 15 Qa. Qa. 0 i 0 ita -----Qa Que Que Que O 0, 0, 0, 0, 0, 0, 0, BI OLT OR 4 44 6' 6' 6' 6' 6' 6' 6' • 2.6 Q.1 Q.5 Q.6 Q.1 4 Par Or Or Or Or TIO IN Q1 Q1 Q1 Q1 Q1 Q1 10 10 10 10 10 10 10 365 061 061 061 0BI 0BI 947 05 04 04 05 T - Te 12 34 12 120 110 1k ITA TE 150 130 120 120 120 120 11 6 925 926 921 928 928 928 921 921 92 927 15 051 051 051 051 051 081 LL 31,/11, 740 24 ALOLD SE OF OF Fig 44 (b) The reciprocal net of indicies 1 h l 50 1.5 5 240 240 N ... 16 34 34 94 9 . 5 °55 °56 10 20 91 10 110 3 15 • 74 94 975 TI OIL 8.8 -EA VER SE --. 13 -0

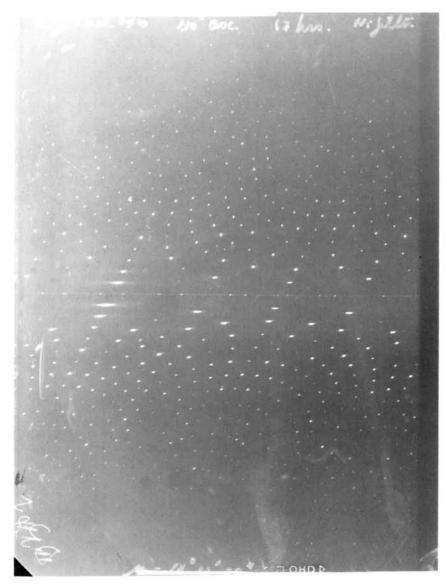


Fig.4.3(d) The third-level Weissenberg photograph of Latifolin. [100] Rotation axis: Cuk₂ -radiation.

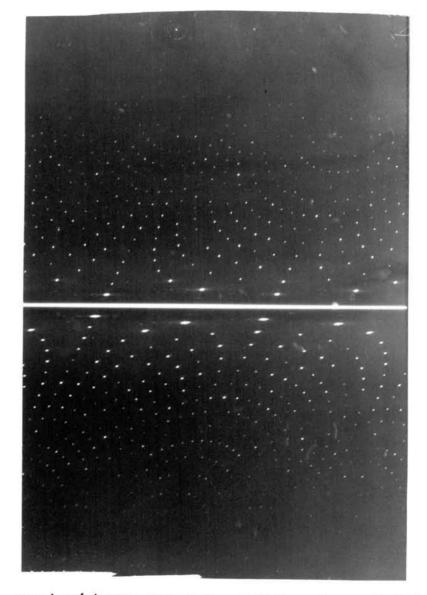


Fig.4.3(c) The second-level Weissenberg of photograph of Latifolin. [100] Rotation axis; CuK_d -radiation.

e. •t* or cu or 026 Q4 Q7 Q8 Q29 Q80 Q80 Q34 Q34 024 024 0,0 0,4 3 °--..... 3 9 " 4 02" O.E. 10 97 8 91 8 91 E 0 0 0 O 3 α, 0 0 0 0 0 0 T 0 0 0 as × at es es es es es et es es es es es 0 ° ° " 01, 010 019 010 011 Rotation axis, 3 rd layer, with •3 • o* • 041 94 041 042 041 041 041 041 94 04 041 052 051 044 056 041 041 043 45 061 061 94 061 061 061 061 061 06 021 061 061 061 061 06 061 061 063 0.0 05, 01, 9, 5, 5, 9, 05, 9. 07. 07. 04 04 04. 07. 07. ••• . 0, 0, . • • °° 9. 0. 01 0 BE 0 BJ 0 B4 0 B6 0 BC ř • Que Qua Que Que Que : •-" o" ---oa1 0 64 --. . OKS 04. à 031 053 0^{11 3} 0.5 [100] * 3 ÷ ÷ . š OZI 0 0 01. 012 0ar a 0a. OBT A OB! đ •¹⁰ 0 o⁼ ō GL GI GI GI GI GI OB o" 1, 9, 6, 9, 9, 9, 9, 0, 9, 0, 0, 0 Q1 9, 9, 9, 4, 9, 0, 0, 0, 0, 0, 9, 0, " - " ° On E 01 0 • 0 6 • • ء* ÷ 5 025 GE 1. di la 0, 2, 9, 1 0, 0, 1 õ 9, 9, 9, 9, 0, 1 10 10 10 10 10 10 10 10 10 9" 0" 0" 0" 1 0" 1 0" 0, 0, 0, 0, 0, 0, 1 36 96 0. S ... Fig. 4.4 (d) The reciprocal net of indicies 3 k l 1.0 1.0 0a1 0a1 011 015 0,1 0,1 0,1 ... 1 -0.0 94 94 974 91 94 95 95 -..... 10 10 9.4 °ze °za 5 0 E 190 BS -0.FE * 8 ••

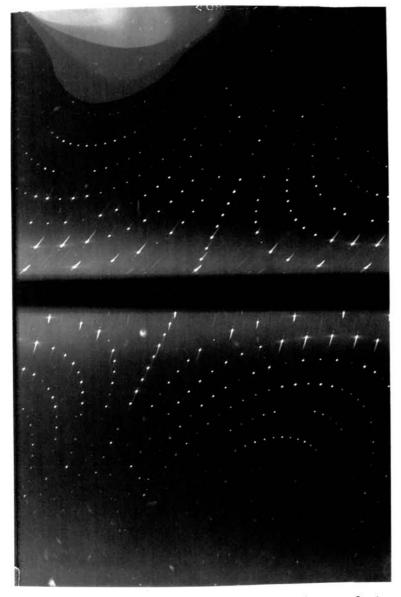


Fig.4.3(f) The first-level Weissenberg photograph of Latifolin. OlO Rotation axis; CuK -radiation.

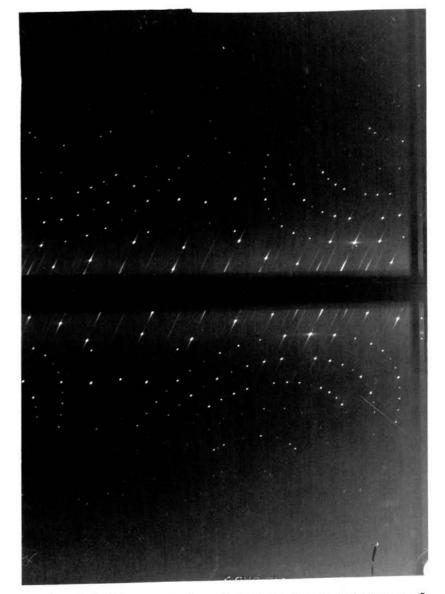


Fig.4.3(e) The zero-level Weissenberg photograph of Latifolin. OlO Rotation axis; Cuk -radiation.

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Rotation axis , 0th layer, with k = 0.... 5 1 2 : 5 1 020 0. 1 . . . 0.0 5 1 1 į. 0.0 . . -..... 1 [010] ы 0 9 ... e . o 510 -..... . 1 -. " 0 -Ļ 2 Ę 6 01.0 Fig 4.4 (e) The reciprocal net of indicies h o l ĩ į, 2 r, 110 100 1 2 of -F h 0.0 0 a n l 2 **... ..** 0.5 3 ... -. 0. B 510 · 20 -

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			-						96	\$ 9	40		25		•	-	52	35	14 S	5 S	2 5	5 5	-	20		-	-	-	-
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	¥ 2 H	2 15	212		210	20		11	3.6	52	24	23	11		2	52	2 E	2.5	14	5 Z	26	£2	, î.	52 •	2 IG		•2.R	• 3 B	2 14
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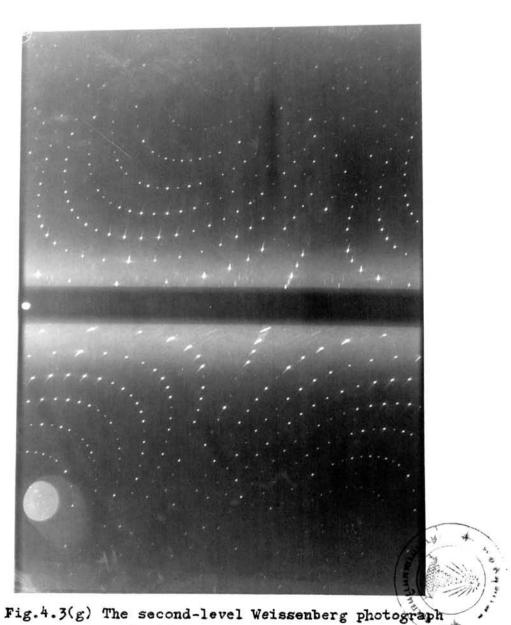


Fig.4.3(g) The second-level Weissenberg photograph of Latifolin. Olo Rotation axis; CuK -radiation.

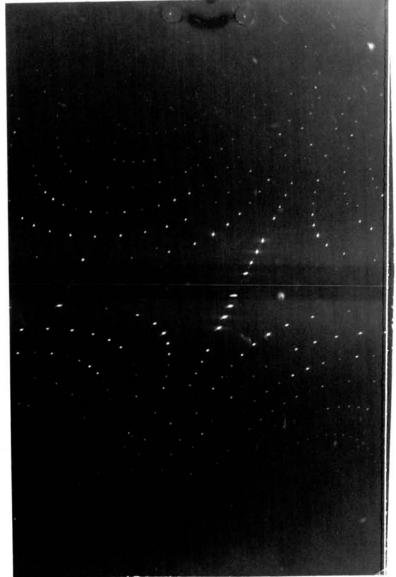
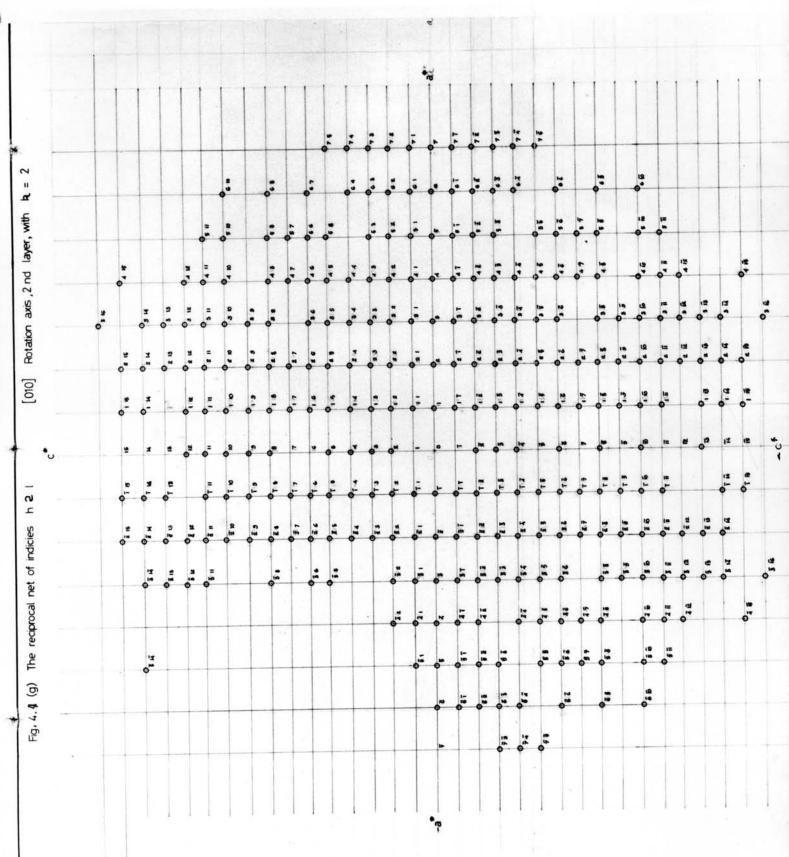


Fig.4.3(h) The third-level Weissenberg photograph of Latifolin. 010 Rotation axis; Cuk -radiation.



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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 Crystallography⁵ as shown in table 4.5. It is an orthorhombic crystal with Laue symmetry m m m.

Table 4.5

	R	ecipro	ocal	lattic	e*			Dir	ect	lat	tics	5	THE	No.	
a*	=	.21 r	. l.u	. d*	=	90°	a	. =	7.	34	Ao	X	=	90.00	Jup
b*	=	.115	r.l.	u. β*	=	90°	b	=	13.	47	A o	β	=	90°	
c *	=	.10 r	.l.u	. ð*	=	90°	c	=	15.	42 .	Ao	8	=	90°	
From	ta	ble 4.	l an	id tabl	e 4.	2 a	= 7.4	l A ^C	, t	=	13.	.48	Ao		
														togra	phs
			dime	ensions	fro	om rot	ation	and	l Wei	sse	nbei	rg 1		togra	phs
		cell a	dime =		fro A ⁰	om rot ± (tation	and A ^O	l Wei	sse =	nbei 90	rg 1 o		togra	phs

⁵ <u>International Tables for X-Ray Crystallography</u>, (Vol.II; Bermingham : Rynoch Press, 1967), p.p. 108.

* The measurements are based on the assumed error of the equipment with + 0.2 mm.

IV.4 Determining a space group of Latifolin (C17H18O4)

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

Table 4.6

Determination of space group of

Clas Refl		1	Condition for nonextinction n = integer	Interpretation of extinction	Symbol
h	k	l	no conditions	Primitive	р
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	21
0	k	0	k = 2n	[010] screw axis comp. b/2	21
0	0	8) = 2n	[001] screw axis comp. c/2	21
Po	int	Grouj	<u>ò</u>		222
La	ue (iroup			m m m
Sp	ace	Grou	2		P21 21 21

Latifolin (C₁₇H₁₈O₄)

IV.5 Determination of the density and the number of

molecules per unit cell (N) of Latifolin (C17H1804)

Flotation method⁶ was used to find the density of Latifolin $(C_{17}H_{18}O_4)$ by using the mixture of :

l)	carbon tetrachloride	(d	=	1.60	g/cm ³	at	25°	C)
	and kerosene	(d	=	0.79	g/cm ³	at	25°	с),
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.

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 N= Dobs ^{xVxA} mol.mass.	Calculated densit: $D = \frac{N \times mol.mass.}{V \times A}$ g/ cm ³
Carbon tetrachlo- ride and kerosene with	1.233		
V ₁ : v ₂ = 1.20 : 1		3.998	
Bromobenzene and			
kerosene with	1.235	which	
V ₁ : V ₂ = 2.29 : 1		implies	
	D _{ave} = 1.234	4	1.235

N = number of molecules per unit cell

V = volume (Taken from table 4.5) = $1540.2 \times 10^{-24} \text{ cm}^3$ 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 (C17H1804)

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_A, radiation $(\lambda = 2.28962 \text{ A}^{\circ})$

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 (C17H18O4).

Table 4.8

Refinement of cell dimensions

	a	=	7	•3887	+	0.0006	Ao
	b	=	13	.4581	+	0.0008	Ao
	с	=	15	.6157	+	0.0010	Ao
V	olu	me	=	1552.	8	Ao	

h	k	¢	$\sin^2 \theta_{obs}$	$\sin^2\theta_{cal}$	$\Delta = (\sin^2 \theta_{obs} - \sin^2 \theta_{cal}) 10^{-5}$
0	0	2	0.021479	0:021498	- 1.9
Ō	1	2 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	0 1 1	0.034309	0.034318	- 0.9
1	1	1	0.036561	0.036617	- 5.5
1	0	2 3 1	0.045484	0.045505	- 2.0
0	1	3	0.055537	0.055607	- 7.0
0	3		0.070513	0.070498	1.4
1	2 2 1 3 3	23320	0.074422	0.074449	- 2.6
0	2	3	0.077280	0.077315	- 3.5
1 0	1	3	0.079588	0.079613	- 2.4
0	3	2	0.086646	0.086622	2.4
1 2 2			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	l	1	0.108612	0.108637	- 2.5
1	0	4	0.110077	0.109999	7.7
0	32	34	0.113444	0.113495	- 5.1
0	2		0.114840	0.114937	- 9.6
0	4	1	0.121156	0.121150	0.5
2	1	2 4	0.124796	0.124761	3.5
1	2		0.138881	0.138943	- 6.1
0	1	5	0.141656	0.141600	5.6
1	4 3 2	5 1 4	0.145193	0.145157	3.5
0	3	4	0.151116	0.151117	- 0.0
0	2	5 5	0.163367	0.163308	5.8
1	1	5	0.165624	0.165606	1.8

h	k	2	sin ² 0 _{obs}	$\sin^{2}\theta_{cal}$	$\Delta = (\sin^2 \theta_{obs} - \sin^2 \theta_{cal}) 10^{-1}$	-5
2	3	1	0.166516	0.166525	- 0.8	
2	3	3	0.175084	0.175123	- 3.8	
1	2	1 3 5	0.187355	0.187314	4.0	
1	4	3	0.188212	0.188154	5.8	
ō	4	4	0.201742	0.201769	- 2.7	
2	3		0.209490	0.209522	- 3.2	
2 3 1	õ	3 1 4	0.221560	0.221435	12.5	
1	4	1	0.225804	0.225776	2.8	
2	2	5	0.259255		- 8.0	
2		2		0.259335 0.266503		
3 1	24	2	0.266550	0.200505	4.7	
1		5	0.274089	0.274147	- 5.7	
l l	3	6	0.282772	0.282615	15.6	
T	5 5	4	0.290986	0.290900	8.9	
2 1	5	2	0.298500	0.298425	7.5	
1	6	2338	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		0.367919	0.367978	- 5.8	
l	5	6	0.398538	0.398391	14.6	

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

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