Chapter III

EXPERIMENTAL

3.1 Taking Rotation photograph

Crystals of Stemonone were formed in transparent orange needle shape of various sizes (Fig. 15). The largest single crystal has dimensions of $.3 \times .05 \times .7 \text{ mm}^3$. From the external morphology of the crystal (Hartshorne, Stuart, 1964, p.7) the crystal axes \bar{c} and \bar{b} were chosen (Fig. 15) as follows:

 \bar{c} ([001] axis) along the needle axis of the crystal \bar{b}' ([010] axis) along the cleavage of natural growth.

Two well-formed single crystals were selected and mounted on the goniometer heads separately, with $\bar{\bf c}$ and $\bar{\bf b}'$ axes as rotation axes respectively. Rotation photographs of both crystals were recorded (Fig. 16) using a Unicam Vertical camera, 60 mm. diameter and Cu radiations (λ_z = 1.5418 Å, λ_β = 1.3922 Å). The mean cell parameters c and b' determined from the rotation photographs in Table 1 and 2 using equation (12) and (14) are

b' = 15.5 Å,

c = 8.25 Å.



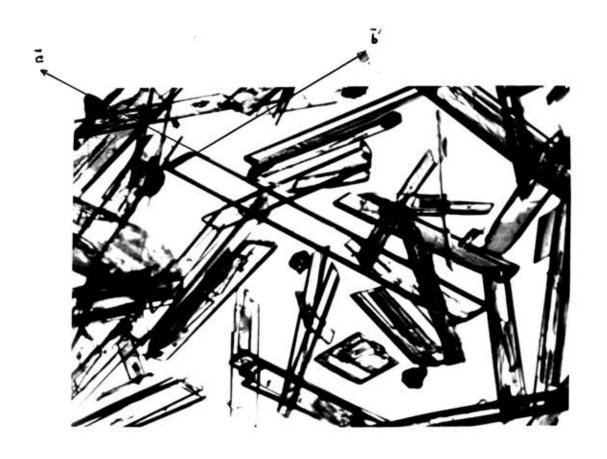
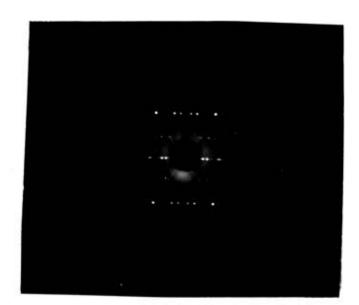


Fig.15 Stemonone Crystals.



b'Rotation axis





c Rotation axis
Fig. 16 Rotation Photograph.

TABLE 1

Determination of c axis length from [001] rotation photograph.

layer	2m	m	tanv	ν	3	c/n	(A)	
	ram •	mm.	=m/r		=sinv	= \/\x		
CuK, ra	diatio	n, λ =	1.5418 A			10 - 10 - 10 - 10 - 10 - 10 - 10 - 10 -	New Cold State	
1	11.4	5.70	.1900	10° 45	.1866	8.263	3.26	
2	24.1	12.05	.4017	21° 53	.3728	4.136	8.27	
3	40.6	20.30	.6767	34° 5'	•5604	2.751	8.25	
4	68.0	34.00	1.1333	480 34	•7498	2.056	8.23	
CuK _ß r	adiati	on, λ _β =	1.3922	Å				
1	10.3	5.15	.1717	9° 4.5	.1694	8.218	8.22	
2	21.4	10.70	•3567	19° 38	•3360	4.143	8.29	
3	35.3	17.65	-5883	30° 28'	•5070	2.746	8.24	
4	55.3	27.65	.9217	42° 40'	.6778	2.054	8.22	

r = 30 mm.



TABLE 2

Determination of b axis length from [010] rotation photograph.

layer	2m	m	tany		39	b/n	b'	
	mm.	mm.	=m/r	ν	=sinv	= \(\lambda/\xi\$	(A)	
CuK, r	adiati	.on, λ _ω =	1.5418	A				
1	5.9	2.95	.0983	5° 37'	.0979	15.75	15.7	
2	12.0	6.00	.2000	11° 19'	.1962	7.86	15.7	
3	18.7	9.35	.3117	17° 19	•2977	5.18	15.5	
L _t	26.1	13.05	.4350	23° 31'	.3990	3.86	15.5	
5	34.5	17.25	•5750	29° 54'	•4985	3.09	15.5	
6	44.6	22.30	•7433	36° 57	•5964	2.58	15.5	
CuK _B r	adiati	on, λ _β =	1.3922	A				
1	5.3	2.65	.0883	5° 3'	.0881	15.80	15.8	
2	10.9	5.45	.1817	10° 18	.1788	7.79	15.6	
3	16.9	8.45	.2817	15° 44'	.2712	5.13	15.4	
L _i .	23.0	11.50	•3833	20° 58'	•3578	3.89	15.6	
5	30.3	15.15	•5050	26° 43'	•4509	3.09	15.4	
6	38.7	19.35	.6450	32° 49'	•5419	2.57	15.4	

3.2 Taking Laue photographs

Laue photographs had been taken at 10 degree intervals about both \bar{c} and \bar{b}' rotation axes but no symmetry was found, so the crystals are expected to be a triclinic system.

3.3 Taking Weissenberg photographs

Necessary parameters for taking normal beam and equiinclination Weissenberg photographs were evaluated from rotation photographs in Table 3.

Weissenberg photographs from the zeroth to the third layer of \bar{c} and \bar{b} rotation axes were recorded using a Unicam Weissenberg camera, 57.3 mm film diameter with .5 mm. translation per 1 degree of rotation and CuK, radiation ($\lambda = 1.5418 \ A$), Fig. 17.

The reciprocal net of each layer was reconstructed. From \bar{c} rotation axis 0th to 3rd layer of reciprocal net the reciprocal axes were chosen to be

$$a^* = .15 \text{ rlu}, \quad x^* = 71^\circ 21^\circ,$$
 $b^* = .20 \text{ rlu}, \quad x^* = 79^\circ 8^\circ,$
 $c^* = .297 \text{ rlu}, \quad x^* = 53^\circ 50^\circ.$

The crystals belong to the triclinic system.

 a^* , b^* and y^* were read directly from the 0th layer, whereas c^* a^* a^* were obtained from a calculation by considering the projection of 1st, 2nd and 3rd layer on 0th layer (Fig. 18).

using cosine law as a method of calculation.

TABLE 3

Necessary parameters for taking equi-inclination

Weissenberg photograph.

layer	: 41	3/2	M	s = rtan A
	7	= sin_M		r = 24mm.
[001] r	tation axis			<u> </u>
1	.1866	•0933	5° 21'	2.2
2	.3728	.1864	100 44	4.5
3	•5604	.2802	16° 16	7.0
[010] ro	tation axis		· ·	
1	•0979	•0489	2° 49'	1.2
2	.1962	.0981	5° 38'	2.4
3	•2977	.1501	8° 32'	3.6

^{1 %} from Table 1 & 2. (reciprocal lattice unit)

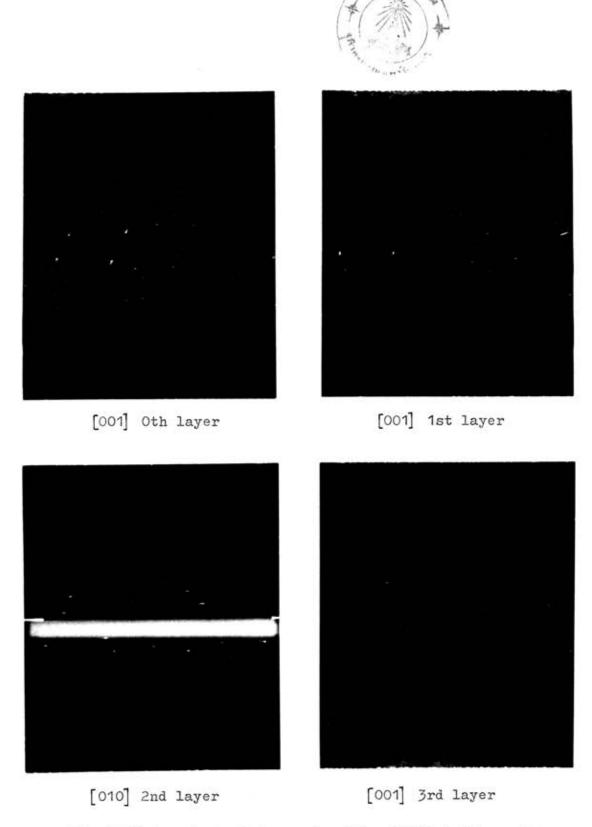


Fig. 17 Weissenberg Photographs of \bar{c} and \bar{b}' Rotation axis.

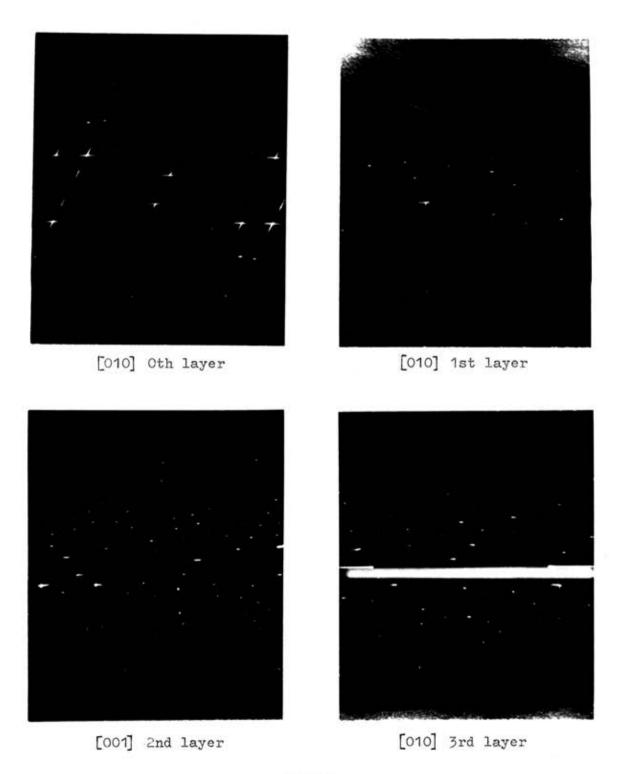


Fig.17

The reciprocal axes were chosen from \bar{c} rotation axis photographs because the projection of \bar{c}^* on a^*b^* plane is coincide with \bar{b}^* axis, which simplifies the problem of calculation in three dimensions.

The reciprocal net of \overline{b} rotation axes failed to relate with that we have chosen, since the crystals are triclinic crystals the unit cell chosen is therefore one of the infinitely many possible unit cells.

According to these chosen reciprocal axes, indices of each reflection spot on the film have been given in Table 4, and no extinction condition was found.

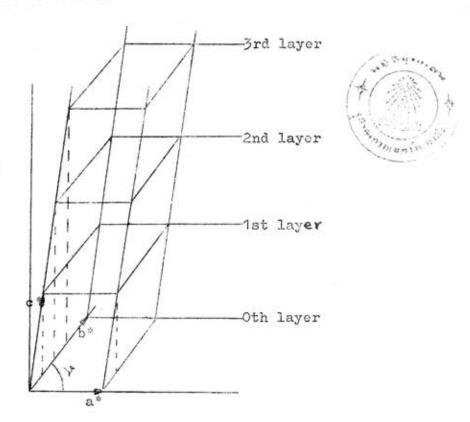


Fig. 18 Diagram of projection of 1st, 2nd, 3+1 layer on 0th layer.

3.4 Crystallographic data.

The unit cell parameters of the crystal lattice were calculated from the results obtained from section 3.1 and 3.3:

c = 8.25 Å

a* = .15 rlu.,
$$\mathcal{L}^*$$
 = 71° 21'

b* = .20 rlu., β * = 79° 8'

c* = .197 rlu., γ * = 53° 50'.

Using equations (International Table for X-ray Crystallography vol. II)

$$a^* = \lambda \frac{bc \sin 2}{v},$$

$$b^* = \lambda \frac{ca \sin \beta}{v},$$

$$c^* = \lambda \frac{ab \sin \gamma}{v},$$

$$v^* = \lambda^3,$$

$$v^* = a^*b^*c^* \sin 2 \sin \beta^* \sin \gamma^*$$

$$= a^*b^*c^* \sin 2 \sin \beta^* \sin \gamma^*,$$

$$= a^*b^*c^* \sin 2 \sin \beta^* \sin \gamma^*,$$

it was found that

$$a = 12.73 \text{ Å}$$
 $\mathcal{L} = 105^{\circ} 14^{\circ}$
 $b = 9.90 \text{ Å}$ $\beta = 90^{\circ}$
 $c = 8.25 \text{ Å}$ $\gamma = 124^{\circ} 42^{\circ}$
 $a:b:c = 1.28: 1: 0.83.$

The density of this Stemonone (C_{19} $^{\text{H}}_{14}$ $^{\text{O}}_{8}$) crystal determined by the floating method using the mixture of carbon tetrachloride ($d = 1.6 \text{ gm/cm}^3$) and petrolium ether (B.P. 60-110°c, $d = .6 \text{ gm/cm}^3$) is 1.514 gm/cm³, at 27.5°d.

The number of molecules per unit cell (Z) calculated from the observed density and the molecular formular ($^{\rm C}_{19}$ $^{\rm H}_{14}$ $^{\rm O}_8$) and unit cell parameters are

Z = 1.993

But Z must be integer, so the number of molecules per unit cell must be 2, which gave the calculated density of the crystal of 1.519 gm/cm³.

From the international Table for X-Ray Crystallography Vol. I, the space group of this Stemonone crystal is either P1 or P1, the structure being either non-centrosymmetrical or centrosymmetrical.

TABLE 4
Indices of spots of reflection

[[100	Oth lay	/er, 1 =	0								
h	k	h k	t h	lc	h	lc -	h	k	h	k	h	k
0	1	1 10	3	ŝ	6	1	7	2	3	6	6	0
0	2	2 0	3	10	6	2	ব	3	3	7	6	1
0	3	2 1	3	12	6	i	ī	<u>7</u>	3	8	5	2
0	1:	2 2	4	2	6	ā.	ī	7	3	9	3	4
0	5	2 3	4	ī	6	$\tilde{\mathcal{L}}_{\!$	ī	8	3	10	6	5
0	7	2 4	· I _t	Ž.	6	6	2	0	3	12	5	6
0	2	2 6	4	3	6	8	ż	1	3	ī	6	ī
0	3	2 1	4	T ₊	6	9	2	2	3	Ž	8	ž
0	$I_{\tilde{t}}$	2 2	4	5	7	0	Ž	3	3	3	7	0
0	5	2 3	4.	5	7	ż	Ž	4	3	7	7	2
1	0	2 4	4	7	7	$\bar{I}_{\!$	ž	5	4	0	7	4
1	1	2 6	4	8	7	5	ž	6	\overline{L}_{+}	1	7	5
1	2	2 8	4	13	7	7	2	8	14	2		
1	3	2 10	5	0	7	5	ž	10	4	3		
1	4	2 11	5	1	1	0	ż	ī	$\overline{4}$	L_{l^*}		
1	5	3 0	5	3	ī	1	ż	ā	\vec{I}_{\downarrow}	ž		
1	8	3 1	5	ī	ī	2	ā	3	4	8		
1	1	3 2	5	2	1	3	ż	$\tilde{L}_{\!$	5	0		
1	2	3 3	5	3	ī	<i>L</i> ₁ .	Ž	6	5	1		
1	3	3 1	5	$\tilde{I}_{\dot{\mathbf{l}}}$	ī	6	2	8	5	2		
1	I_{ν}^{τ}	5 2	5	5	1	8	3	.0	5	3		
1	2	3 3	5	š	1	9	3	1	5	4		
1	3	3 4	5	9	ī	10	3	2	5	5		
1	9	3 7	6	0	ī	1	3	3	5	ī		

[c	001]	1st l:	ayer	, 1 = 1					T)	able '	4	con	tinue	d)	
h	k	h	k	h	k	h	k	h	k		h	k		h	k
0	0	1	3	3	5	5	1	ĭ	5		2	5		5	3
0	2	1	14	3	ī	5	ź	ì	6		ž	3		5	4
0	3	1	5	3	2	5	3	ī	7		2	8		5	7
0	4	1	6	3	3	5	$\overline{l}_{\!4}$	i	10		3	0		5	8
0	5	1	3	3	$\vec{l}_{\!$	5	6	1	ī		3	1		5	9
0	6	1	9	3	5	5	$\vec{7}$	ī	2		3	2		5	i
0	7	1	10	3	7	5	13	ī	\vec{l}_{+}		3	3		5	2
0	8	1	17	3	8	6	0	i	3		3	L _k		5	0
0	9	2	0	3	9	6	2	ì	6	88	3	6		5	3
0	ī	2	3	3	17	6	i	ī	7		3	8		3	l_{\dagger}
0	2	2	4	4	0	6	4	ī	8		4	0		6	5
0	3	2	6	L _f .	1	6	5	à	0		4	1		5	1
0	Ĩ.	2	ī	14	2	6	8	Ž	1		4	3		5	$\overline{l}_{\!+}$
0	5	2	2	24.	3	7	0	ž	2		4	l_{l}		7	0
0	7	2	3	L_{l}	ī	7	1	2	3		Ī _†	5		7	1
0	9	2	74	4.	Ž	7	ž	à	4		4	7		7	2
0	10	2	5	4	3	7	\overline{l}_{+}	2	5	9	4	8		7	l_{l}
1	0	2	6	l_{k}	5	7	6	2	7		Ī ₄	9		7	5
1	3	2	7	L _F	3	7	9	ž	8	83	Ī,	10		7	6
1	$I_{\mathbb{H}}$	2	8	4.	7	8	5	ž	9	29	4	11		7	1
1	5	2	5	4	12	8	10	2	11	8	4	12		8	2
1	6	2	10	4	13	ī	0	2	12	15	Ī _‡	2		8	4
1	7	3	0	5	0	ī	1	2	ī		Ę	3			
1	8	3	1	5	1	ì	2	ž	2		4	14			
1	1	3	2	5	2	1	3	ž	3	23	5	0			
1	2	3	I _I .	5	3	i	4	Ž	\overline{l}_{i}		5	1			

(01	2nd	layer	, l =	2				(Tab	le 4	cont	inued)	
h	k	h	k	h	k	h	k	h	k	h	k	h	k
0	0	1	10	3	7	6	10	ž	3	\bar{l}_{r}	1	6	4
0	1	1	11	4	0	7	3	2	$l_{\dot{r}}$	4	2	7	0
0	2	2	0	4	1	7	4	2	5	4	3	7	1
0	3	2	1	4	2	7	3	Ž	6	<u>1</u>	4	7	3
0	$l_{\!+}$	2	3	Lį.	3	7	7	ā	7	<u> 4</u>	5	7	4
0	5	2	4	14	4	7	8	ż	8	4	6	7	5
0	6	2	1	4	1	7	10	2	1	$\overline{4}$	7	8	1
0	ī	2	2	$L_{\!\!\!\!+}$	3	7	17	ā	2	1 4	8	3	2
0	2	2	3	$L_{\!$	\tilde{l}_{l}	8	<u> 7</u>	2	3	$\overline{4}$	9	8	4
0	$\bar{l}_{\!$	2	4	4	5	ī	0	2	6	$\overline{L}_{\!$	1	9	4
0	5	2	5	4	6	1	1	3	0	\tilde{l}_{+}	2		
0	8	2	6	4	7	ī	2	3	1	$\bar{4}$	3		
1	0	2	7	4	8	i	3	3	2	4	6		
1	1	2	9	L_{r}	10	ī	4	3	3	5	0		
1	4	2	10	5	0	ī	5	3	4	5	4		
1	5	2	11	5	2	ī	6	3	5	5	5		
1	6	3	0	5	3	i	1	3	6	5	7		
1	7	. 3	1	5	5	ī	Ž	3	7	5	9		
1	7 1	3	2	5	6	ī	$\overline{I_{+}}$	3	8	5	2		
1	2	3	5	5	7	i	5	3	ī	5	5		
1	3	3	ī	5	9	ī	7	3	2	Ğ	1		
1	$\overline{I_{\Phi}}$	3	2	6	0	ī	8		3	6	2		
1	5	3	3	6	ž	i	9	3	\overline{l}_{+}	6	5		
1	6	3	$\overline{L}_{\!\downarrow}$	6	5	2	0		5		10		
1	8	3	5	6	7	2	1		7	6	1		
1	9	3	6	6	9	2	2	4	0	6	2		

0	01	3rd 3	Laye	r, 1 =	3				(Ta	ble	4	cor	tinu	ed)	l
h	lc	h	k	h	k	h	k	h	k		h	k		h	k
0	0	1	8	3	6	5	7	ā	0		ż	6		5	11
0	1	2	0	3	7	5	8	ĩ	1		3	0		5	i
0	2	2	1	3	9	6	2	ī	2		3	1		6	7
0	3	2	2	3	10	6	ž	1	I,		3	2		6	2
0	5	2	4	4	0	6	3	ī	5		3	3		7	0
0	7	2	5	4	$l_{\rm i}$	6	7	i	6		3	5		7	1
0	8	2	6	l_{\dagger}	5	6	8	ī	7		3	6		7	2
0	ī	2.	1	4	1	6	9	1	8		3	10		7	3
0	ž	2	2	$L_{\overline{k}}$	3	6	11	ī	7		3	ī		8	3
0	3	2	3	L_{\parallel}	$\overline{I_{l_{r}}}$	6	12	i	ž		3	Ž		8	4
0	<u>7</u>	2	$\tilde{I}_{\!$	4	5	6	13	1	3		3	3			
0	5	2	5	l_{\dagger}	6	7	2	1	<u>7</u>		3	$\overline{I_{\dot{\mathbb{P}}}}$			
0	3	2	5	I_{\dagger}	7	7	4	ī	5		3	5			
0	7	2	7	4	3	7	5	ī	6		<u>1</u>	1			
0	8	2	3	4	9	7	7	ā	7		4	3			
1	0	2	9	4	10	7	8	ī	8		$\bar{l}_{\rm l}$	G			
1	2	2	10	4	11	7	9	2	0		4	8			
1	3	2	11	5	0	7	11	2	1		Ī ₊	10			
1	4	3	0	5	1	8	ž	ž	2		\overline{l}_{\dagger}	ì			
1	5	3	1	5	2	8	3	ž	3		I ₊	3			
1	i	3	3	5	3	8	4	Ž	L _F		5	0			
1	2	3		5	4	8	6	2	5		5				
1	3	3	1	5	2	8	8	2	6		5				
1	$\vec{L}_{\!\!\!+}$	3		5		8		ž	7		5				
1	6	3		5		8		Ž	2			5			
1	7	3	5	5	6	9	8	ž	4		5	10			