March - April 1952

Long series of microphotographs at 75% RH (not preserved) in which trial short-exposure films were generally good — specimen subsequently went non-crystalline during long exposure. Twice drying at room T. over P_2O_5 in attempt to recover photograph apparently spoilt specimen. Specimen non-crystalline at 75% was never re-converted to crystalline

Tilting camera

Series of tilting photographs of minor adjustments to camera.

All specimen rifle thick fibres (40-70μ) @ 75% RH

18.4.52 75%, Ni filter

(45) 3 films. Exposure unknown (3–7 days, ∴ Easter)
Single fibre ~40μ
→ good photo, showing some double orientation

(T0) Specimen previously x-talline, now gives "wet" diagram
(2 films, ~3 days)

(T1) Single fibre, ~50μ, 16 hrs
Tilt ~14° (edge set to 61)

(T2) Chromium 21 hrs, thin V filter
Tilt as above

(T3) As T2. 2 films
On ~18.4.52, 6 p.m. (Friday)
Still running Sat., flames turned out before Mon. 21st
Developed 21.4.52 3 p.m. Dirty centre.
~~Black~~ Specimen deteriorating → "wet" ∴ changed

(46) As (45), but Cr and V filter
Exposure as for T3 – result blank film

(T4) Exposure 16 hrs, 1 film, no filter Tilt ~18°
→ "wet" diagram v well oriented. Dirty centre
∴ specimen transferred to Urecan

T4 specimen was dried 4 hrs over P_2O_5 ∴ this apparently destroyed x-tallinity. ∴ New specimen not dried

(T5) New specimen. Beam centred photographically
Tilt ~18°. Lead pinhole removed from tube. No filter
X specimen moved. V dirty centre ∴ replace pinhole

(47) As (46). On 7 p.m. 21.4.52
Exposure to 26.4.52 10 a.m. ~~10 a.m. 22.4.52~~
Result – X-tal diffraction pattern of foreign body + diffuse ring
Specimen has "necked" over collimator hole → ~⅔ diameter remaining

(T6) Black paper over film ∴ dirty centre blackened
due to soft radiation scattered from glass collimator
– shadow of DNA fibre (~50μ) appears sharply
on dirty centre. Specimen as ∴ T5, no filter
Exposure 3 days. Weak x-talline diagram ∴

28.4.52 Copper target replaced

(48) 3 rolled fibres viewed edge on, Industrial Cr film,
no filter, exposure 16 hrs
→ mainly wet photo, v strong exposure
– no trace of X-tals shown by (47) although
collimator had not been cleaned meanwhile
∴ X-tals were decomp product of DNA fibre

Filament life ~ 250 hrs

(49) Specimen from TO (which gave good "wet" photo) re-exposed at 75% RH. 2 films, Ni

29.4.52 11 a.m. — night of May 1-2 (filament burned out)

i.e. exposure 33 – 44 hrs

NB Film B back to front. V good "wet" photo

New filament

(50) Rolled fibre, 75%, no filter, Industrial G.

2.5.52 3.20 p.m. – 5.00

⇒ wet photograph, mod. well oriented

∴ put specimen & dry over P_2O_5

(51) Specimen as (49) with holder centred over collimator so as to include both 3.4 Å arcs

2.5.52 7.30 p.m. – 6.5.52 5 p.m. with interruption of 32 hrs - i.e. 62 hrs

(50) Streaks = outer pair of diagram (( ))) are v strong & differently shaped from normal "wet" diagram, being more rounded. Is this an effect due to double orientation of e.g. elliptical helices?

(47) $2l = 2 \times 14.2$ to 20

Show rings of spots. Diameters measured

Assume specimen-film distance 14.2 mm

$2l$ (cm)	strength	$\tan 2\theta$	θ	d	$\log d$
1.65	m	.581	15°5′	4.38	.6415
1.89	v w	.665	16°48′	3.95	.597
2.13	m	.750	18°26′	3.61	.5575
2.29	w	.806	19°26′	3.43	.535
2.63	s	.926	22°24′	3.13	.4955
2.70	w	.951	21°47′	3.08	.489
3.14	s	1.106	23°56′	2.82	.450

Also faint diffuse ring at 8–9 Å

Cell spots too small to be phosphate

Wyckoff gives As_3PO_4 cubic, $a = 6.00$

KH_2PO_4 tetrag. $a = 7.43$, $c = 6.97$

$(NH_4)_2H_2PO_4$ " " 7.53 7.54

Li_3PO_4 orthorhombic 4.86, 6.1026, $c = 6.07$

$NaHCO_3$ $a = 7.51$, $b = 9.70$, $c = 3.53$, $\beta = 93° 19'$

NH_4HCO_3 7.29 10.79 8.76

(49) 49B

Rough measurements on projection drawing (2.5.52) (gypsum layer-lines straight)

Layer-line spacings, mm :— (= 2z)

14·8, 30·5, 46, —, 79, —, —, 131

Suppose specimen-film distance = 14·4 mm

3·4 A reflexions are for 2z = 164 mm, θ = 13°4′, tan 2θ = ·491

~~tan 2θ = 2z/2×167 =~~

∴ Specimen-film distance for projection = $\frac{164}{2} \times \frac{1}{\cdot 491}$ = 167 mm

∴ tan 2θ for layer-lines = $\frac{2z}{2\times 167} = \frac{2z}{334}$

= ·0443, ·0914, ·1378, —, ·2365, —, —, ·392, —, ·501

θ = 1°16′, 2°37′, 3°55′, —, 6°39′, —, —, 10°42′, 13°23′

d = 34·8, 16·8, 11·3, —, 6·64, —, —, 4·14

Multiplying by ~~1~~ 1, 2, 3, —, ~~5~~, ~~6~~, —, —, 8, 10 gives =

34·8, 33·6, 33·9, 33·2, 33·2, 33·2

∴ layer-line spacing = 33–34 A

∴ 3·4 A corresponds to 10th layer-line

(if helix is non-integral no. residues per turn, 3·4 A are not necessarily lie on a layer-line)

Equator strong doublet at 20·8, 23·2 mm

On above approx (using 3·4 A) this gives 24·6 A, 22·1 A

This suggests co-existence of 2 phases differing only by 1 molecular layer of water separating chain units

Taking mean pos^n of 1st doublet and centre of diffuse equatorial spot gives 23·3 A, 13·8 A, 9·3 A, 5·42 A

~~These do not fit long close-packing~~

this fact, together with the 3·4 A are lying on a layer-line, suggest that there is an integral no. (or right fractional no.) of residues per turn of helix (if there is a helix) even in the "wet" state.

In passing from "crystalline" to "wet" the predominant equatorial spacing is approximately doubled, and the fibre-axis period is extended by ~ 25%

(27 A → 34 A)

(52) As 51, with Na_2CO_3
on 6.5.52 6 p.m.

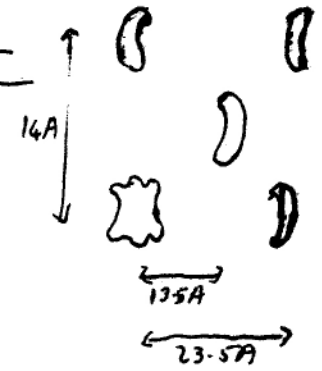
51A

Measuring on projection, and taking meridional arc to be 3.4 gives equatorial spots <u>24.5</u> Å and 9.83 Å (weak and streaky)

2.7.52

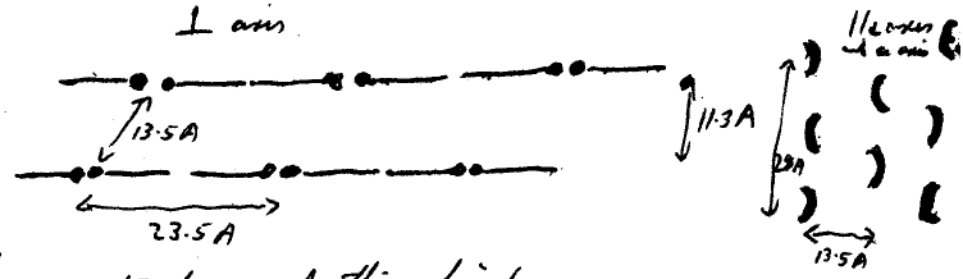
Notes on first cylindrical Patterson

General form



There is no indication of a helix of diameter 11A
The central banana-shaped peak fits curve calc. for helix of diameter 13.5 A having 2 turns / unit cell
If a helix there is only one strand
(2-strand helix would give ⊗)
If a helix, it is v far from continuous uniform density. Has deep trough for x 6-7 A.
Helix does not explain vertical short vector ≈ 4A (this is superimposed on peak at origin, so represents true distance of > 4A, i.e. not 3.4A). But if there is a flat banana-like unit in structure, with banana axes ⊥ fibre axis this vector is explained.

Cylindrical Patterson should give unit cell. Projection of a and b axes on plane ⊥ fibre axes being ≈ 13.5 and 23.5 A.) differs little from 60°
~~If bananas are flat & vertical, not helical, this explains why those of 23.5 A are best ~~ ~~they are~~
i.e. triclinic cell ≈ ½ size of previously considered primitive cell of monoclinic face-centred lattice.
Again, the dimensions of this cell make a helical structure improbable - if helices of 13.5 A diameter there is the wrong spacing - the long period filled?
Suggests rather a double sheet structure



Also a structure of this kind seems necessary to explain double orientation in diagram 45.
Effect poss. due to preferential orientation of sheets w.r.t. surface of fibre, and not all of fibre in beam (though nearly all of fibre was in beam)

July 7th

Pattern max. at $x \sim 13A$, $z \sim 7A$ can not be lattice point

∴ projected all 

would have a lattice vector with both x and z values intermediate between those of the 2 vectors observed. No trace of this.

∴ Pattern was extended to 46 A

This shows a large peak at $x \sim 35$ A, $z \sim 7$

i.e. it is to the peak of 23 A as is the 13 A peak to the origin

∴ this is not a lattice point

It seems probable that both lattice vectors are contained in the 23 A peak. Banana-like form might be due to one peak on x-axis and one slightly off nearer origin. This would give $\beta \sim 93°$, which seems reasonable for measured z value

There is no lattice peak between 24 A and, at the nearest 36 A. ∴ the cell is thought 2-dimensional

July 15th

G. Patt. has no cutbacks near. // fiber axis between 4–5 Å and ~14 Å

∴ there is no narrow straight chain of high density // axis.
∴ high-density regions are discrete regions, not continuous
Gap between x = 6 — 1 x = 10

14.8.52

(60) Fat fibre of Sigiex 10 scale divisions (~120μ) covering ½ of 80μ collimator, to look for double orientation due to edge effect
14.8.52 12.15 p.m. – 15.8.52 10.45
Film goes. Trace of double orientation ?

(61) Sigiex (2), fibre ~15μ ✗ see whether Xtalline
15.8.52 11.45 – 18.8.52 9.45
→ not diagram (v weak)

(62) Sigiex (3). Pulling properties resemble (1) rather than (2). Fibre ~50μ
18.8.52 4 p.m. – 19.8.52 3.30 p.m.
→ Xtalline photograph (poor)

(63) 19.8.52 6 p.m. – 25.8.52 11 a.m.
As (60), some fibre, covering ~⅓ hole

no double orientation

(64) Fibre Sigiex (1) ~40μ, 75% RH
25.8.52 12 p.m. – 26.8.52 6 p.m.
Good photograph, but with strong trace of Xtalline impurity – collimator blocked

(65) Fibre used in (60) & (63) replaced. 75% RH
26.8.52 7 p.m. – 27.8.52 3 p.m. – 20 hrs

(66) As above, using "$Ca(NO_3)_2$ sat."
27.8.52 4 p.m. – 28.8.52 2 p.m. – 22 hrs
→ similar to (65) but with one Xtalline impurity

(67) As above, using $CaCl_2$ (in jar : blocks sucked bubbler)
Start over table 28th!
28.8.52 6.30 p.m. – 29.8.52 3 p.m.

(68) Over $Ca(NO_3)_2$ 3.50 p.m. 29.8.52
29.8.52 Exposed 5 p.m. – 30.8.52 12 p.m. – 19 hrs
→ photograph similar to 67

(69) Over P_2O_5 in hydrogen, 12.30 p.m. 30.8.52
1.30 p.m. on → 31.8.52 8 a.m.
Film fogged ✗. repeat

(70) As (69)
1.9.52 10.30 a.m. – 2.9.52 2 p.m.
again fogged (though less)
= this due to acid vapour ?

(71) Repeat as (70). On 2.9.52 4 p.m. → 3.9.52 4.30
fogged again
– must be ∴ acid vapour. Why?

(72) $NaClO_3$
3.9.52. 4.45 in camera
Exposed 6.15 – ~~dg~~ 2.15 4.9.52

(73) Na_2CO_3
4.9.52 5.30 in camera
Exposed 6.30 — 5.9.52 2.30
→ X-talline photo

(74) Wetted by standing ~10 minutes over dil. sol $NaClO_3$
→ length change, moved off collimator hole
then exposed to air till came back over hole &
exposed to Na_2CO_3 in camera 4 p.m. 5.9.52
Exposed 5 p.m. – 6.9.52 11 a.m.
Fibre has flowed – too wet

from Sheet (3)

(75) New fibre, ~100 μ dia, glue v close to collimator hole (~0.2 mm of fibre exposed)
In camera, with Na_2CO_3 1 p.m. 6.9.52
Exposed 3 p.m. — 8.9.52 10 a.m.
→ wet photo with trace of crystalline

(76) Same fibre as (75), with $NaClO_3$ 11 a.m.
exposed 11.40 — 3.30
→ "wet" photo
∴ specimen dry dried over P_2O_5 – before
repeating w $NaClO_3$

(77) 9.9.52 exposed 1.20 – (?)
→ wet photograph

(78) 14.10.52 New fibre, 60-70μ. Over $NaClO_3$
Dyed 48.m. —