My numbering
A.K.

1

March - April 1952

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

Tilting camera

Series of setting photographs and minor adjustments to camera.

All specimen single 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 xtalline, 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., filament turned on before Mon. 21st

Developed 21.4.52 3 p.m. Dirty centre.

~~Best~~ Specimen deteriorating → "wet" ∴ changed

(46) As (45), but Cr and V filter

Exposure as for T3 – result blank films

(T4) Exposure 16 hrs, 1 film, no filter. Tilt ~18°

→ "wet" diagram v well oriented. Dirty centre

∴ specimen transferred to Urecam

T4 specimen was dried 4 hrs over P_2O_5 & this apparently destroyed xtallinity. ∴ 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 – Xtal diffraction pattern of foreign body + diffuse ring

Specimen has "rocked" over collimator hole → ~⅔ diameter smaller

(T6) Black paper over film ∴ dirty centre believed

due to soft radiation scattered from glass collimator

– shadow of DNA fibre (~50μ) appears sharply

on dirty centre. Specimen as in T5, no filter

Exposure 3 days. Weak xtalline 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 Xtals shown by (47) although

collimator had not been cleaned meanwhile

∴ Xtals were decomp^n 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 ~~stay~~ 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 A 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 part of diagram ((( ))) are v strong and differently shaped from normal "wet" diagram, being more rounded. Is this an effect due to double orientation of e.g. elliptical helices?

(47)

$2l = 2 \times 14.2 \tan 2\theta$

Shows rings of spots. ~~Ø~~ Diameters measured. Assume specimen-film distance 14.2 mm

$2l$ (mm)	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	21°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 A

Cell prob. 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, β = 93° 19′
NH_4HCO_3 7.29 10.79 8.76

(49) 49B

Rough measurements on projection drawing (2.5.52) (supposing 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 meridian arc has 2z = 164 mm, θ = 13°4′, tan 2θ = .491

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

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

= .0443, .0914, .1378, —, .2365, —, —, .392, —, .504

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, 2, 3, —, 5, —, —, 8, 10 gives :—

34.8, 33.6, 33.9, 33.2, 33.2, 33.2

∴ layer-line spacing = 33 – 34 A

and 3.4 A ~ corresponds to 10th layer-line

(if helix is non-integral no. residues per turn, 3.4 A need 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 centres of diffuse equatorial spots gives 23.3 A, 13.8 A, 9.3 A, 5.42 A

~~These do not fit hexag. close-packing~~

This fact, together with the 3.4 A lying on a layer-line, suggests that there is an integral no. (or simple 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.

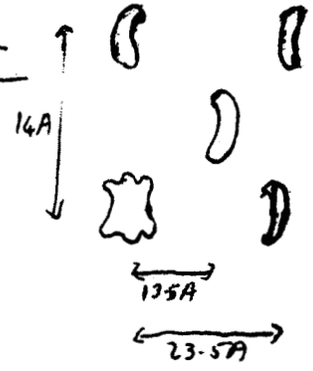
51A

Measuring on projection, and taking meridional arc of be 3.4
gives equatorial zeros 24.5 and 9.0.3 (weak and sharp)

2.7.52

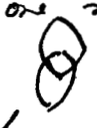
Notes on first cylindrical Patterson

General form



There is no indication of a helix of diameter 11A. The central banana-shaped peak fits curve calc. for helix of diameter 13.5A 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 axis ∥ fibre axis this vector is explained.

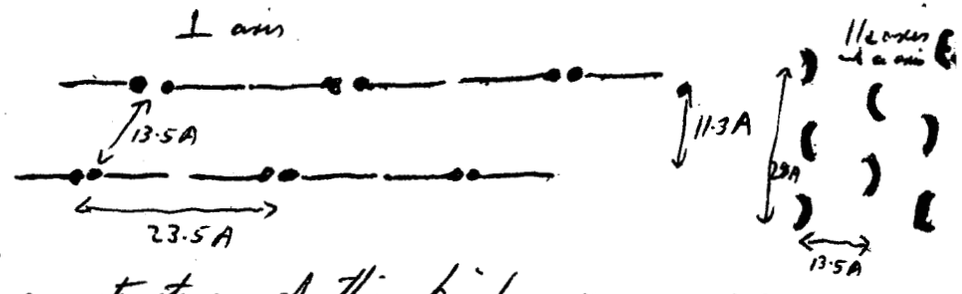
Cylindrical Patterson should give unit cell. Projections of a and b axes on plane ⊥ fibre axis being ~ 13.5 and 23.5A. γ differs little from 60°.

~~If banana are flat and vertical, not helical, this explains why those at 23.5A are less curved 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 13.5A diameter how is the remaining space in the long period filled?

Suggests rather a double-sheet structure

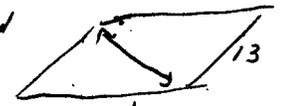


Also a structure of this kind seems necessary to explain double orientation in diagram 45. Effect poss. due to preferential orientation of sheets wrt surface of fibre, and not all of fibre in beam (though nearly all of fibre was in beam)

July 7th

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

∴ projected cell



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 of $x \sim 35$ A, $z \sim 7$

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

∴ this is not a lattice point

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

There is no lattice peak between 24 A and, of the nearest, 36 A. ∴ the cell is thought quasidimensional

July 15th

Cy. Patt. has no ~~[illegible]~~ max. ∥ fibre axes between 4–5 A and ~14 A

∴ there is no narrow straight chain of high density ∥ axis.

i.e. high-density regions are discrete regions, not continuous

Gap between x = 6 and x = 10

14.8.52

(60) Fat fibre of Signer 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 grey. Trace of double orientation?

(61) Signer (2), fibre ~15μ to see whether Xtalline

15.8.52 11.45 – 18.8.52 9.45

→ wet diagram (v weak)

(62) Signer (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), same fibre, covering ~ ⅓ hole

no double orientation

(64) Fibre Signer (1) ~40μ, 75% RH

25.8.52 12 p.m. – 26.8.52 6 p.m.

Good photograph, but with strong have of xtalline impurity – collimator blocked

(65) Fibre used in (60) and (63) replaced. 75% RH

26.8.52 7 p.m. – 27.8.52 3 p.m. – 20 hrs

(66) As above, using "$Ca(NO_3)_2$ sol."

27.8.52 4 p.m. – 28.8.52 2 p.m. – 22 hrs

→ similar to (65) but with some Xtalline impurity

(67) As above, using $CaCl_2$ (in jar: blocks sintered bubbler)

Stood over $CaCl_2$ 2 hrs

28.8.52 6.30 p.m. – 29.8.52 3 p.m.

(68) Over $Ca(NO_3)_2$ 3.30 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)

is this due to acid vapour?

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

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

(73) Na_2CO_3
4.9.52 5.30 in camera
Exposed 6.30 – 5.9.52 2.30
→ Xtalline 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 4p.m. 5.9.52
Exposed 5p.m. – 6.9.52 11a.m.
Fibre has flowed – too wet

from Signer (3)

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

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

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

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