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The crystal structure of 5′-Br-3′:5′-dideoxythymidine.

By M. M. Woolfson,* *Crystallographic Laboratory, Cavendish Laboratory, Cambridge, England*

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The X-ray analysis of 5′-Br-3′:5′-dideoxythymidine, which was produced during the study of the chemistry of certain thymidine derivatives (Michelson & Todd, 1955), was undertaken for the purpose of determining its chemical structure.

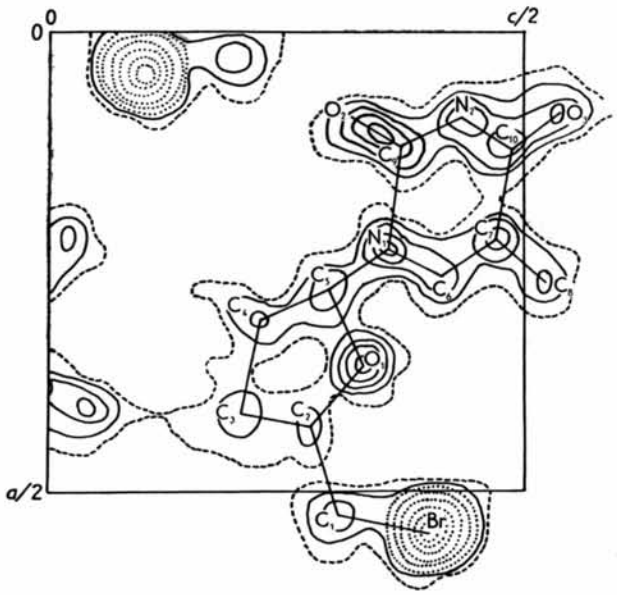

Fig. 1. 5′-Br-3′ : 5′-dideoxythymidine, electron-density map. The contours are at equal arbitrary intervals with the lowest contour broken. The dotted contours round the bromine atom are at twice the interval elsewhere.

The space group was found to be $P2_12_12_1$ with

$$a = 14{\cdot}78,\ b = 4{\cdot}89,\ c = 15{\cdot}17\ \text{Å},$$

there being four molecules per unit cell. The bromine parameters were readily found for the *h*0*l* projection from the two-dimensional Patterson synthesis. With the signs of the structure factors taken from the bromine contribution, an electron-density map was calculated which showed clearly the remainder of the atoms. One stage of refinement gave the map shown in Fig. 1, and, since the object of the investigation had been achieved, no further refinement was attempted. The atomic co-ordinates found for this projection are:

	x	*z*		*x*	*z*
C_1	0·533	0·304	O_1	0·364	0·330
C_2	0·430	0·274	O_2	0·091	0·318
C_3	0·416	0·202	O_3	0·089	0·535
C_4	0·314	0·221	N_1	0·236	0·356
C_5	0·280	0·294	N_2	0·093	0·434
C_6	0·266	0·413	Br	0·547	0·400
C_7	0·226	0·471			
C_8	0·274	0·522			
C_9	0·124	0·367			
C_{10}	0·128	0·486			

Reference

Michelson, A. M. & Todd, A. R. (1955). *J. Chem. Soc.* p. 816.

* Present address: Physics Department, College of Science and Technology, Manchester 1, England.

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The shrinkage of ribonuclease.

By C. H. Carlisle, *Crystallography Laboratory, Birkbeck College, London W.C.*1, *England*

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In the course of taking an X-ray photograph to record the *h*0*l* reflexions of a wet ribonuclease crystal of the monoclinic form (ex aqueous alcohol) in a sealed capillary in the normal manner, a small leak allowed the crystal to dry during its exposure. There was recorded on the photograph both the diffraction pattern of the wet crystal and that of the dry one, with smear lines indicating that a change had taken place. This is most clearly seen (Fig. 1) for the 003 reflexion. The unit-cell dimensions of this form of ribonuclease are:

	a (Å)	*b* (Å)	*c* (Å)	*β* (°)
Wet	30·90	38·8	54·06	106
Dry	29·10	30·08	51·03	114·0

It was possible to confirm from this photograph that the angle *β* increases when the crystal goes from the wet to the dry state.

The crystal did not move off the capillary wall during the drying process and so Fig. 1 shows the correct orien-

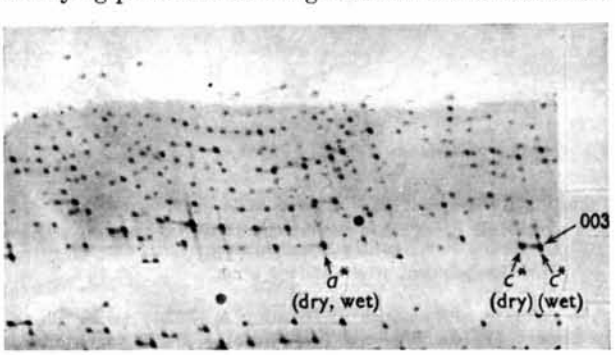

Fig. 1. Weissenberg photograph of ribonuclease, *h*0*l* reflexions.

tation of the two diffraction patterns relative to each other. It is clear from the continuous nature of the streak connecting the 003 reflexion in the wet and dry states that there is no intermediate stage in the drying process of these crystals, such as that observed for haemoglobin (Perutz, 1946). The shrinkage process here merely seems to involve the relative and continuous movement of the molecules from one state to the other.

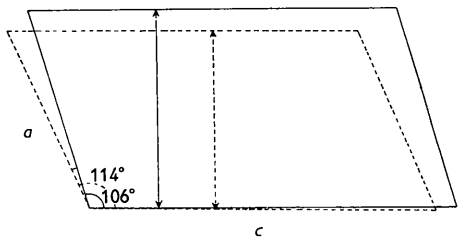

Fig. 2. Relationship of wet and dry unit cells. Full lines: wet cell; broken lines: dry cell.

Reflexions in the dried crystal can be observed to spacings of about 3 Å, indicating that shrinkage can take place in an ordered manner provided it proceeds sufficiently slowly.

Fig. 2 shows the relationship of the wet to the dry unit cell. The small angular shift of the *c* axis is consistent with elongated molecules lying with their major axes approximately parallel to *c* (Carlisle & Scouloudi, 1951); hence the shortening of *b* from 38·80 to 30·08 Å would suggest a movement of the elongated molecules perpendicular to their lengths.

References

Carlisle, C. H. & Scouloudi, H. (1951). *Proc. Roy. Soc.* A, **207**, 496.

Perutz, M. F. (1946). *Trans. Faraday Soc.* B, **42**, 187.

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Use of a fluorescent source in an X-ray diffractometer.

By R. A. Coyle and R. I. Garrod, *Aeronautical Research Laboratories, P.O. Box* 4331, *Melbourne, Australia*

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Weiss, DeMarco & Weremchuk (1954) have described a method for converting a Norelco X-ray spectrograph to a diffractometer in which a fluorescent X-ray source is used. With suitable geometry, the half breadth of the 110 reflexion from an annealed iron specimen (Co radiation) was found to be only about 20% larger than the value obtained with a standard Norelco diffractometer (22′ and 18′ respectively), whilst the background was reduced by a factor ~ 25. If these results were applicable generally, the method would be an attractive alternative to a conventional X-ray source and crystal monochromator for applications requiring high resolution and a low background, since the use of a monochromator leads to complexity in mechanical design if focusing conditions are to be satisfied for a range of X-radiations.

Calculations, however, suggest that favourable comparison in line breadths between the fluorescent and normal methods is likely to be obtained only when the specimen itself produces appreciable broadening. For example, applying the convolution methods developed by Alexander (1950, 1954) to the experimental conditions specified by Weiss *et al.* leads to values for *instrumental* broadening of 6′ (α_1) for the standard diffractometer and 27′ (unresolved $\alpha_1\alpha_2$ doublet) for the fluorescent arrangement. It would therefore appear that the values obtained experimentally by Weiss *et al.* were determined largely by (i) the specimen and (ii) instrumental factors, in the standard and fluorescent methods respectively.

This impression has been confirmed by measurements in these Laboratories on suitable reflexions from a 'standard' silicon specimen and annealed powdered iron, using the following experimental methods:

(*A*) A fluorescent source and a slit system identical with that described by Weiss *et al.*; goniometer radius 14 cm.

(*B*) Normal X-ray source and standard Philips (Eindhoven) goniometer; 1° divergence slit, 0·1 mm. receiving slit, 17 cm. radius.

(*C*) Normal X-ray source, quartz crystal monochrometer, Philips goniometer; 1° divergence slit, 0·2 mm. receiving slit, 11 cm. radius.

The half breadths observed are summarized in Table 1, together with the equivalent calculated values for instrumental broadening.

Table 1. *Half breadths for reflexions from silicon and iron obtained by three diffractometer methods*

(Values of half breadths are in minutes of arc)

			Method *A*		Method *B*		Method *C*	
Specimen	Radiation	2θ(°)	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
Silicon	Co	33	24*	25*	—	—	—	—
Silicon	Co	55	—	—	6½	6	9	10
Silicon	Cu	56	—	—	7	6	10	10
Iron	Co	52	30*	27*	12	6	12	9

* Unresolved $\alpha_1\alpha_2$; otherwise α_1 alone.