

together in the *c* direction mainly through N–H···O hydrogen bonds between the amino nitrogen and phosphate oxygen atoms.

The arginine molecule is linked to the phosphate group by O–H···O hydrogen bonds from the phosphate to carboxyl oxygen atoms and N–H···O hydrogen bonds from the amino and guanidyl nitrogen atoms N(1), N(3) and N(4) to the phosphate oxygen atoms. As shown in Figs. 4 and 5 these hydrogen bonds bind the molecules on one side of the phosphate layer. The other side is attached to another arginine molecule. The layer of arginine which lies between the two phosphate layers is, therefore, made up of double layers of arginine molecules and chains of water molecules. The water molecules and the carboxyl oxygen atoms O(5) are arranged about the twofold screw axis ($\frac{1}{2}, y, 0$) to form a hydrogen-bonded chain. As shown in Table 8, the directions of the hydrogen bonds around the water oxygen atom are approximately tetrahedral.

The ϵ -nitrogen atom N(2) forms a bifurcated hydrogen bond to the water oxygen atom O(*W*) and the carboxyl oxygen atom O(6) of the neighbouring molecule translated by *b*.

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Crystal Structure of *n*-Bromoacetamide, *n*-CH₃CO–NH–Br

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n-Bromoacetamide has been studied by X-ray diffraction. The crystals are orthorhombic, space group *Aba*2, $a = 8.74 \pm 0.02$, $b = 12.58 \pm 0.02$, $c = 8.80 \pm 0.03$ Å, $Z = 8$. Three-dimensional data collected by precession photography gave a final *R* index of 10.68%. The atom Br bonded to the amide N atom has a bond length of 1.82 Å.

Introduction

n-Bromoacetamide is of interest as it inhibits the enzyme action of rennin (G. C. Cheeseman, private communication), the structure of which is under investigation (Bunn, Camerman, T'sai, Moews & Baumber, 1970). The compound was synthesized by the method described by Oliveto & Gerald (1951). The crystal

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structure of orthorhombic acetamide has been described by Hamilton (1965).

Experimental

Crystallographic data

C₂H₄NOBr, $M = 137.9$, orthorhombic, *mm*2, space group *Aba*2 (C_{2v}^7), $a = 8.72 \pm 0.02$, $b = 12.58 \pm 0.02$, $c = 8.80 \pm 0.03$ Å, $V = 967.5$ Å³, $d_m = 1.95$, $d_c = 1.90$ g.cm⁻³, $Z = 8$, $F(000) = 528$, ($\lambda = 0.71$ Å; Mo $K\alpha$).

Crystals suitable for X-ray diffraction measurements were obtained from a slightly supersaturated aqueous

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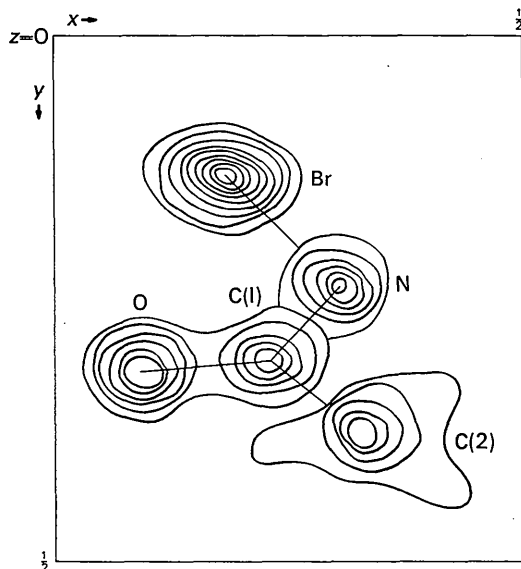


Fig. 1. Three-dimensional electron-density distribution indicating the positions of the atoms.

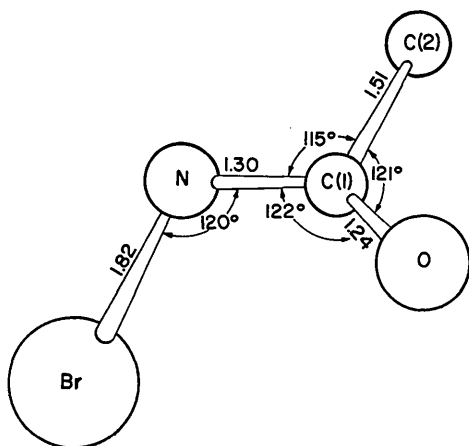


Fig. 2. Bond lengths and angles in the molecule.

solution at about freezing temperature. The density was determined by the flotation method in a mixture of bromobenzene and bromoform. Rectangular crystals of approximately $0.5 \times 0.5 \times 0.3$ mm were used for collecting intensities. Unit-cell dimensions were obtained by the precession method. The condition for reflexion, hkl , $k+l=2n$; $0kl$, $k=2n$, $l=2n$, $hk0$, $k=2n$, and the diffraction symmetry mmm are consistent with the space group $Aba2$.

A series of precession photographs ($\bar{\mu}=30^\circ$) to obtain 0.7 \AA resolution were taken. Intensities were measured with a recording microdensitometer giving peak heights, which were taken to be proportional to intensities. No absorption correction was applied. Relative scaling of 28 photographs was accomplished by the method of Hamilton, Rollett & Sparks (1965)

after applying Lorentz and polarization corrections. From 1450 measured points, 547 independent structure amplitudes were obtained.

Structure analysis

The positions of all light atoms were found from a three-dimensional electron-density map phased on the bromine atom whose position had been determined from two-dimensional Patterson syntheses. Fig. 1 shows the three-dimensional electron-density distribution. The structure was refined by least squares. Hydrogen atoms were not included in the analysis. The final atomic coordinates and anisotropic thermal parameters are listed in Table 1. Very strong reflexions and unobserved reflexions which were given half the minimum observable intensity were eliminated from the final refinement. The final R value is 10.68%. Bond distances and angles are listed in Table 2 and Fig. 2. The observed and calculated amplitudes are given in Table 3. The $\text{NH}\cdots\text{O}=\text{C}$ hydrogen bond has a distance of 2.80 \AA . Fig. 3 shows the molecular packing and the hydrogen bonding scheme. The bromine, nitrogen, carbon and oxygen atoms are almost coplanar.

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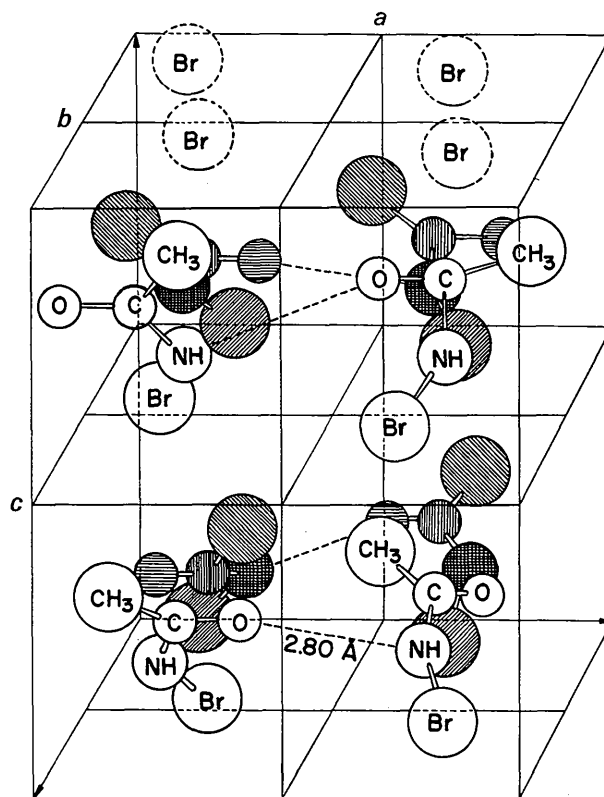


Fig. 3. Packing of the molecules. Hydrogen bonding is depicted by the dotted lines.

Table 1. Atomic coordinates and temperature factors

	Final atomic coordinates			Anisotropic thermal parameters*					
	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
Br	0-211	0-139	-0-003	0-010	0-005	0-011	0-004	0-004	-0-000
O	0-120	0-331	0-178	0-009	0-010	0-030	0-004	-0-020	0-002
N	0-325	0-237	0-109	0-009	0-006	0-024	-0-008	-0-002	0-003
C(1)	0-259	0-312	0-185	0-003	0-011	0-012	0-009	0-006	0-003
C(2)	0-366	0-388	0-263	0-015	0-008	0-028	-0-008	-0-014	-0-002

* The anisotropic temperature factor is of the form:

$$\exp [-(h^2 B_{11} + k^2 B_{22} + l^2 B_{33} + kl B_{23} + hl B_{13} + hk B_{12})].$$

Table 2. Interatomic distances and angles and their standard deviations

	Distance	σ	Angles	σ	
Br—N	1-820 Å	(0-025)	Br—N—C(1)	120-03°	(1-93)
O—C(1)	1-240	(0-031)	O—C(1)—N	122-34	(3-35)
N—C(1)	1-296	(0-040)	O—C(1)—C(2)	121-22	(3-27)
C(1)—C(2)	1-509	(0-050)	N—C(1)—C(2)	115-17	(2-59)

Table 3. Observed and calculated structure amplitudes

The four columns give l , F_o , F_c , α .

Section A lists very strong reflexions not included in the final refinement. Section B lists unobserved reflexions which were given half the minimum observable intensity; these also were not included in the final refinement.

h k l	F_o	F_c	α
0 0 0	10000	10000	
0 10 0	1000	1000	
0 20 0	1000	1000	
0 30 0	1000	1000	
0 40 0	1000	1000	
0 50 0	1000	1000	
0 60 0	1000	1000	
0 70 0	1000	1000	
0 80 0	1000	1000	
0 90 0	1000	1000	
1 0 0	1000	1000	
2 0 0	1000	1000	
3 0 0	1000	1000	
4 0 0	1000	1000	
5 0 0	1000	1000	
6 0 0	1000	1000	
7 0 0	1000	1000	
8 0 0	1000	1000	
9 0 0	1000	1000	
10 0 0	1000	1000	
11 0 0	1000	1000	
12 0 0	1000	1000	
13 0 0	1000	1000	
14 0 0	1000	1000	
15 0 0	1000	1000	
16 0 0	1000	1000	
17 0 0	1000	1000	
18 0 0	1000	1000	
19 0 0	1000	1000	
20 0 0	1000	1000	
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22 0 0	1000	1000	
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24 0 0	1000	1000	
25 0 0	1000	1000	
26 0 0	1000	1000	
27 0 0	1000	1000	
28 0 0	1000	1000	
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30 0 0	1000	1000	
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32 0 0	1000	1000	
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35 0 0	1000	1000	
36 0 0	1000	1000	
37 0 0	1000	1000	
38 0 0	1000	1000	
39 0 0	1000	1000	
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50 0 0	1000	1000	
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97 0 0	1000	1000	
98 0 0	1000	1000	
99 0 0	1000	1000	
100 0 0	1000	1000	

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