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 $O-H\cdots O$ 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 laver 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 Aba2, $a=8.74\pm0.02$, $b=12.58\pm0.02$, $c=8.80\pm0.03$ 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 structure of orthorhombic acetamide has been described by Hamilton (1965).

Experimental

Crystallographic data

C₂H₄NOBr, M = 137.9, orthorhombic, mm2, space group Aba2 ($C_{2\nu}^{17}$), $a = 8.72 \pm 0.02$, $b = 12.58 \pm 0.02$, $c = 8.80 \pm 0.03$ 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 α).

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 Å 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 NH···O=C hydrogen bond has a distance of 2.80 Å. Fig. 3 shows the molecular packing and the hydrogen bonding scheme. The bromine, nitrogen, carbon and oxygen atoms are almost coplanar.

The author wishes to thank Dr C. W. Bunn for his guidance.



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

Table 1. Atomic coordinates	and temper	ature factors
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Final atomic coordinates				Anisotropic thermal parameters*					
	x/a	y/b	z/c	B_{11}	B ₂₂	B ₃₃	B ₂₃	<i>B</i> ₁₃	B_{12}
Br	0.211	0.139	-0.003	0.010	0.002	0.011	0.004	0.004	-0.000
0	0.120	0.331	0.178	0.009	0.010	0.030	0.004	-0.050	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	0.012	0.008	0.028	-0.008	-0.014	-0.005

* The anisotropic temperature factor is of the form:

 $\exp\left[-(h^2B_{11}+k^2B_{22}+l^2B_{33}+klB_{23}+hlB_{13}+hkB_{12})\right].$

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)
OC(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.

8 93 93 349 1 38 85 175	1 85 90 183 HH 4 KH 30	5 84 88 110 7	64 70 174	••••••	******		
10 61 68 343 3 41 26 166	3 115 110 164 0 17 14 184	7 58 63 348 •H	H. 7 K				
eHe o Ke 30 0 108 105 0	7 46 49 157 4 44 38 170	•He 5 Ke 8e 1	50 57 550	3 1 3 3 30 0 31	•He o Ke 8•	4 13° 135 343	0 14 5 Ko ge
8 111 94 853 8 77 75 10	9 31 31 157 •He 4 Ke 3•	0 34 37 180 •H	He 8 Ke of 4	4 149 208 341	0 140 137 0	3 131 106 170	•H= 3 K= 6+
6 30 41 182 6 53 55 358	3 133 139 160 1 64 11 1	6 31 25 189 4	47 39 313	•H= 0 K= 3*	•H# 1 X# 1*	0 176 113 186	• 110 139 15e
eHe o Ke 40 8 31 41 1	4 139 141 357 5 46 50 333	•H• 5 K=110 •H	He 8 Xe 10 0	0 87 159 180	0 113 170 180	8 166 101 193	1 130 150 180
10 54 53 161 0 50 47 180	8 64 70 100 0 11 10 11	1 68 76 178 1	18 49 3	0 1 10 340 180	1 100 309 171	4 150 150 150	• H= 5 Ke 34
•K= 0 K= 6+ 3 43 44 174	10 35 37 354 .He 4 Ke 40	5 51 57 178 5	41 47 3 4	8 131 170 165	6 134 133 166	•He Ke 4	5 181 181 171
3 74 75 9 4 39 45 176 4 93 100 118 6 16 11 178	•He 3 Ke 3* 0 68 69 180	• 7 34 49 169 •H	He 8 Ke 30 4	4 153 833 170	•H• : K• 6•	0 133 171 0	•H= 5 K= 7•
6 101 95 17 eHe s Ke of	3 117 144 349 4 88 64 187	1 33 37 357 3	44 47 343	•H= 0 K= 6*	3 146 167 357	4 130 163 354	,
8 38 33 17 8 69 77 171 elle o Xe 8e to 54 77 167	5 87 84 3 6 50 56 178	•H• 6 K• 0• 5	40 36 355				
4 100 100 6 eHe s Ke te	9 38 43 341 •Hu 4 Ku 5*	6 38 46 235 +H	Ha 8 Ka 4				
6 54 68 384 8 101 87 170	•H= 3 X= 4• 3 114 115 188	• • • • • • • • • •	18 9 0 **	•••••	**************		*****************
eHe o Kejoe 5 98 99 163	3 57 59 183 7 67 75 165	3 188 138 175 4	19 19 338				
e 109 109 180 7 48 41 191	4 36 41 197 9 46 46 16	5 65 68 185 6	28 25 328	•Hw o Y = 3*	5 30 17 335	•H= 5 K= 5*	6 31 10 251
4 79 76 188 11 18 17 151	•H= 3 K= 4* a 68 45 4	•H= 6 K= 1	66 68 175 10	0 16 16 176	9 34 9 343	9 20 10 143	0 31 6 180
6 58 63 181 He a Ke an	8 67 63 29 8 55 44	3 39 20 203 3	70 76 173	•H= o K= 6+	•H= 3 K=114	•H• 5 K= 6•	3 30 9 110
ekm p Km120 \$ 177 CC CB	5 16 15 114 6 11 18 10	*** 6 K# 1	CO 48 188	•H+ 0 K+ 8+	1 33 34 04	+H= ; X= 8+	•He 7 Ke ge
0 35 36 180 6 38 30 4	7 34 33 338 *H= 4 F= 7*	1 78 78 199 9	4 38 178 10	• 14 33 333	5 24 18 26	4 10 7 184	1 26 16 68
4 10 44 151 4 04 86 174	2 88 90 179 5 10 11 15	1 3 03 00 105 •n	** ** ***	• • • • • •	1 17 17 195	1 14 10 141	3 31 5 117
6 53 38 140 3 59 47 339	4 89 101 169	7 54 40 157 4	35 25 198 2	\$ \$9 9 316	•H= # K+16*	3 26 13 327	•H= 8 ×= 0*
• 23 84 141 5 38 37 134 •He o Kesse •He o Ke se	0 00 73 174 0 53 37 0 8 46 47 170 3 67 67 77	9 33 34 135 °H		*** 1 K* 1*	0 31 0 100 eHe 1 Ke 10	5 38 15 835	6 14 20 100
0 55 58 0 6 183 110 354	•H= 3 K= 70 4 38 49 35	• 41 31 • •H	H= 8 K= 8	•H= 3 K= 3*	13 18 24 343	•He 5 K = 10*	•H+ 8 K# 2*
3 04 71 355 0 70 70 340	1 100 114 181 6 35 38 35	, 3 30 30 55 0 •He 6 Xe ce • •H	30 34 500 1	1 11 41 144	5 ··· · · · · · · · · · · · · · · · · ·	•Ha (Xa120	• • • • •
6 39 49 357 •He a Ke 5*	5 67 67 166 1 84 91 356	1 115 116 358 1	51 65 3 18	1 36 9 159	•H= 3 K= 6*	0 34 17 0	4 33 17 74
•H= 1 K= 10 1 110 137 14	7 45 61 168 3 87 91 34	3 185 180 360 3	45 51 3	•//= : K= 4*	• #3 31 15°	• • • • • • • • • •	8 11 17 111
3 48 58 348 5 78 70 339	*He 3 Ke 8* 7 44 53.35	7 68 69 346 ·H	Hu 9 Ke 10	•H= 5 K= 5*	5 29 7 87	+H= 6 X+ 0*	+H= 8 ×= 6+
5 35 80 81 7 51 45 345	0 ³ 0 79 0 9 17 34 34	9 40 43 353 1 •H= 6 K= 6+ 1	43 43 359 5	5 18 19 151	7 13 9 00	0 19 15 100	0 15 9 0 6 10 7 119
9 s8 13 342 •H= 5 K= 6•	4 58 56 359 0 36 38 184	0 33 40 180 OH	He 9 Ke 20 9	9 24 5 135	4 33 46 181	8 34 13 188	+H= 8 K= 7*
eHu : Ku := 0 76 86 150	45 53 9 8 44 44 16	9 8 31 84 883 0 •He 6 Ke 79 8	74 74 0	•He 1 Ke 70	•H= 3 K=13*	0 18 0 180	3 53 7 175
10 54 54 170 4 90 80 173	•H= 3 X= 90 •K= 4 K= 190	8 37 38 44 4	38 39 387 9	9 #4 15 345	•H= 3 ×=14*	4 20 27 314	•He 8 Xe 8=
•H= 1 K= 3= 6 63 66 195	1 38 10 337 1 50 56 58	,		•H= : K# 9*	0 34 10 100	4 30 18 70	3 31 19 310
5 31 94 903 •He a Ke 74	•He 3 Keso+ +He 4 Kes4+	eH= 6 K= 94 3	51 55 184 5	5 24 3 348	6 13 19 214	6 21 12 78	•H= 8 F=31=
7 87 86 153 1 48 58 357 eHe . Xe 48 1 18 18 18 18	a 59 58 a a 37 37	b 1 65 90 150 5 A 3 73 80 174 ●H	35 30 107 7 Han a Kan de	7 25 3 225	10 13 6 178	4 31 3 315	• P= 9 K= 4•
0 51 43 0 OHe s Ke 80	4 57 47 549 •He 5 Ke se	5 60 68 170 0	37 34 180	•H= 1 K=11*	•H= 4 K= 4*	6 31 11 266	4 10 14 101
1 100 113 345 0 89 106 180	6 57 33 336 1 519 114	1 7 40 51 171 3 He 6 Xeite 6 H	47 19 139 5 He g Xe 6	5 84 88 105	+H= 4 X= 6+	*H= 6 K= 7*	•H= 9 X= 17
6 38 88 336 4 90 79 179	1 68 61 350 5 88 87 34	1 43 50 0 0	43 28 180 9	9 35 9 146	8 24 31 8	5 28 85 875	1 16 18 176
	3 68 66 349 7 54 50 35	1 3 25 43 351 2 	33 35 154	•H= 1 K=13*	1 18 13 116	7 31 31 393	5 20 10 10 10
3 61 46 838 eHe s K- 9*	•He 3 Kasse •He 5 Ke 3*	•H• 7 X+ 10 •H	H= 9 K= 70	3 36 10 17	7 31 97	•H= 6 K= 8•	•He 9 Ke 6+
3 19 17 97 1 57 57 169	0 60 61 150 0 86 77 18	1 76 67 100 1 1 76 67 100 1	40 40 354 5	5 35 9 15	.H K. 8.	eHa 6 Ka10*	•H= 9 K= 9*
6 103 111 354 5 57 58 178	6 40 41 186 4 59 54 19	5 67 66 166 5	39 35 6	.He 1 X+14*	8 21 28 349	0 34 5 180	1 26 5 58
8 59 50 341 7 36 33 168	•Ha 3 K=130 6 39 33 17.	4 7 35 40 177 **	17 14 184 2	0 34 13 0 3 37 34 384	1 16 10 178	1 26 14 333	3 34 26 816
eH= ; K= 70 0 9; 87 0	allang Kassa allang Kanga	0 44 59 180 eH	He 10 Ke 0*	4 35 37 333	3 38 18 190	3 31 21 36	•H=10 K= 10
1 68 45 357 8 89 91 3	1 33 35 188 3 113 103 16	• • • • • • • • • • • • • • • • • • •	49 40 0 6	6 85 17 311	0 34 38 180	3 31 30 308	3 33 11 144
7 38 87 340 6 59 50 3	"He 3 X=160 9 45 47 160	5 70 74 559 4	48 48 351 1	1 23 15 6	4 20 16 137	4 23 39 113	•H+10 K= 3*
•K= 1 K= 8= 8 41 48 0	0 35 30 0 HH 5 X 40	5 81 86 349 ***	16 41 190	3 35 0 350	1 26 24 7	4 30 37 268	3 30 30 365
4 95 101 188 0 39 36 0	0 78 65 0 4 33 16 5	9 34 36 350 •H	-10 X= 40	eHe a Ke as	ette 5 Ka 24	6 23 18 8	•ו11 K• 1*
6 64 64 186 8 44 37 343	3 135 139 553 •H= 5 K= 5*	•X• 7 K• 4• 0	49 43 100 4	4 15 14 310	10 23 15 301 eHe t Ke te	7 31 20 383	.H. 11 K. 3*
10 31 18 183 6 19 31 316	6 97 80 9 3 64 51 180	3 37 39 130 4	37 37 161 10	0 23 12 559	11 22 29 168	+H= 7 F= 60	1 34 19 33
•Hu 1 Ku 90 •Hu 2 Ku 130	8 40 39 358 5 31 36 13	a Harr Karşa a H	H=10 X= 50 39 10 118 -	*H# 3 K# 3*		3 30 30 303	0 37 15 0
oHe 1 Ke10e 3 40 34 355	He 4 Ke 10 0 67 74	5 5 57 559 5	at 18 0	9 33 33 147	6 31 10 359	4 13 11 353	3 43 14 6
0 109 101 100 elle s K-140	1 108 115 337 8 38 36 3	5 17 15 33 5	31 31 1 H-11 X- 2-	•H= 3 K= 7•			
4 62 59 148 2 65 56 178	5 103 99 358 6 48 39	1 84 94 iAt 0	43 41 180				
6 60 55 157 4 50 48 178	7 64 68 333 eHe 5 Fe 7e	3 79 77 178 3	41 44 174				

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