enthusiasm. Graphite samples were kindly furnished by M. B. Carter, Advanced Materials Laboratory, Union Carbide Corporation, Lawrenceburg, Tennessee. The study was supported by the Engineering Experiment Station of the School of Engineering, Auburn University.

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The Crystal Structure of 1,4-Piperazine-γ,γ-dibutyric Acid

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1,4-Piperazine- γ , γ' -dibutyric acid crystallizes in the space group $P2_1/a$ with two molecules of

in the unit cell of dimensions $a = 15 \cdot 554 \pm 0.004$, $b = 6 \cdot 502 \pm 0.002$, $c = 6 \cdot 702 \pm 0.002$ Å; $\beta = 91 \cdot 54^{\circ} \pm 0.04^{\circ}$. A trial structure was obtained by a sign-relation method developed for three dimensions, and this has been refined by electron-density and least-squares syntheses. The only hydrogen bonds in the structure are between hydroxyl oxygen atoms and the ring nitrogen atoms in adjacent molecules; the O ---- H ··· N distance is $2 \cdot 60 \pm 0.01$ Å.

Introduction

1,4-Piperazine- γ , γ' -dibutyric acid,

$$HOOC \cdot [CH_2]_3 - N$$
 $N-[CH_2]_3 \cdot COOH$,

was prepared during investigations in polymer chemistry. The infrared spectrum does not correspond with either the dimer form



or the zwitterion form



of hydrogen bonding, although the potassium salt and the hydrochloride give the expected spectra (Stace, 1962). It appeared therefore that the hydrogen bonding should be investigated by X-ray analysis. [A preliminary report of the structure has already been published (Potter, 1962)].

Experimental

This amino acid crystallizes from anhydrous methanol in the presence of dry ether as colourless laths which cleave very readily parallel to (100). Care was necessary during all operations to keep moisture out, as the substance has a great affinity for water. The crystals last only a few hours on exposure to air; they gradually turn white owing to uptake of water. When the crystals are coated with a clear thixotropic varnish, their crystalline 'life' is increased to a few days.

Oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation indicated the Laue symmetry 2/m, with systematic absences h0l for h odd and 0k0 for k odd, thus uniquely determining the space group as $P2_1/a$. The unit cell dimensions are:

$$a = 15.554 \pm 0.004, b = 6.502 \pm 0.002, c = 6.702 \pm 0.002 \text{ Å}$$

$$\beta = 91.54^{\circ} \pm 0.04^{\circ} .$$

$$D_m = 1.27 \text{ g.cm}^{-3}; \quad D_x = 1.24 \text{ g.cm}^{-3}; \quad Z = 2 .$$

Absorption coefficient for Cu K α radiation, 9 cm⁻¹.

The space group indicates that the molecule must be centrosymmetric, thus locating the centre of the ring at a crystallographic centre of symmetry.

The intensities were recorded on multiple-film equiinclination Weissenberg photographs taken about [010] (the needle axis) with Cu $K\alpha$ radiation. All layer lines



Fig.1. Numbering of atoms adopted in the text.

having inclination angles less than 45° were recorded (k from 0 to 5 inclusive). Measurements were made by visual comparison with a calibrated wedge prepared from a strong reflexion from one of the crystals used for the intensity photographs. No attempt was made to correct the upper-layer intensities for spot distortion, measurements being made on spots on the contracted side of the film. Lorentz-polarization corrections only were applied, and intensities placed approximately on the absolute scale by the method due to Wilson (1942). The 995 reflexions measured in this way represent 78% of the possible number. Owing to the affinity for moisture, a different crystal had to be used for each layer; crystals of approximately the same dimensions were used in each case.

Structure determination

A trial structure was obtained by the sign-fixing method developed by Grant, Hine & Richards (1960) for threedimensional data. Of the 995 observed intensities, 87 terms with unitary structure factors ≥ 0.15 were included in the search for products and an iterative process yielded 71 signs for inclusion in an electron-density synthesis. This showed the structure in detail; later work indicated that all 71 signs were correct.

Initial refinement of the trial structure was carried out by electron-density syntheses. Structure factors were calculated on the basis of coordinates obtained from the first electron-density synthesis, from which 331 signs could be confidently allocated to the observed terms. Two further cycles were completed; at this stage, signs had been allocated to 913 of the 995 observed terms.



Fig. 2. Unit-cell contents. Hydrogen bonding indicated by broken lines.

The parameters were refined to the values finally adopted by three-dimensional least-squares syntheses using a program written for the Ferranti Pegasus computer (Cruickshank, 1962). Four cycles were completed before the contribution of the hydrogen atoms was considered. An F_o - F_c synthesis (where F_c does not include hydrogen contributions) revealed the hydrogen atoms as well-resolved maxima. This synthesis showed quite clearly that hydrogen bonding occurs between carboxyl oxygen atoms and the ring nitrogen atoms in adjacent molecules (Fig. 2). Hydrogen positions were also calculated from known bond distances and angles. For the hydrogen atoms bonded to carbon the normal tetrahedral arrangement was assumed with C-H distances of 1.07 Å. The calculated hydrogen atomic positions were kept fixed during the least-squares refinement. Three more cycles of least-squares were computed, layer by layer, before combining all layers together. Two further cycles yielded a final overall 'R' value of 0.147.

The final observed and calculated structure factors are listed in Table 1, and final values of the coordinates in Table 2. The final composite electron-density map is shown in Fig.3; a comparison of the final calculated hydrogen atomic positions with those measured from the final F_o-F_c synthesis is made in Table 3. The composite difference synthesis showing the hydrogen atoms is shown in Fig.4.

Discussion

Bond lengths and angles calculated on the basis of the final coordinates are shown in Fig. 5, and listed together with the estimated standard deviations in Table 4. Fig.2 shows the unit-cell contents. The short intermolecular hydrogen bonds are between hydroxyl oxygen atoms and the ring nitrogen atoms of adjacent molecules. O-H... N bonds are necessarily of infrequent occurrence (Donohue, 1952; Pimentel & McClellan, 1960), because, from energy considerations, O-H...O bonds are preferentially formed when possible. Amino-acids in general have a hydrogen bonding system involving O-H... N bonds which are rather long: for example L-glutamic acid (Hirokawa, 1955) has such bonds of length 2.86, 2.92, 2.94, 2.86 Å, DL-norleucine (Mathieson, 1953) 2.73, 2.87 Å. By comparison, the O-H ... N distance in 1,4-piperazine- γ , γ' -dibutyric acid is 2.60 ± 0.01 Å. However, there are examples in the literature of O-H... N distances of this order of magnitude; for example cysteylglycine-sodium iodide (Dyer, 1951) and guanine hydrochloride (Broomhead, 1951) have such bonds of length 2.55 and 2.62 Å respectively.

The N-H distance in 1,4-piperazine- γ , γ' -dibutyric acid is found to be 1.11 Å. This 'lengthening' of the covalent bond is a feature of very strong hydrogen bonds. (It has not been possible to locate this hydrogen atom with any precision, but it is hoped that this may be possible later by neutron diffraction.) No intermolecular distance in the structure, other than the hydrogen bonds mentioned above, is shorter than 3.27 Å. As the space group shows that the ring is disposed about a crystallographic centre of symmetry, it must therefore be chair-shaped. This is confirmed by the structure determination. The fact that the C(6)-O(2)bond is much shorter than the C(6)-O(1) bond supports the conclusion that the hydrogen atom is attached to O(1) and not to O(2).



Fig. 3. Final composite electron-density synthesis. Contours at $1 e. A^{-3}$ starting at $2 e. A^{-3}$.



Fig.4. Composite difference synthesis showing the hydrogen atoms. Contours at 0.05 e.Å⁻³ beginning at 0.1 e.Å⁻³.

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Table 1.	Observed and	calculated	structure factors

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	13 - 1 14 - 1 16 1	1.6 2 1.3 3	• •	• - 3	3•3 3' 5•5 1 3•1 4 1•6 1	- 4 - 1 7 - 7	1 2 3 -	• 8 6 • 9 0	· I I I	i - 1 3 3 -	7•1 15 7•9 7• 3•6 4		1 - 9 3 - 3	6 4 8 8	•1	, c. 2 - 3.	9 0.		6 u 7 1	• 6 • •	0 1 1	3 - 1 h 2	3 0.0 0 0.0	ŀ	7 Ь	4 2	
	ь 2 — 2	105 205 301 24	• 1	4 4 5 - 1	7.8 3 5.4 1.5	5•3 7•1 9•1	4 - 6 I 7 -	4.8 4 4.8 4 4.6 10 5.1 5		4 5 6 7	0.3 0. 0.5 0. 4.6 5.		5 - 0 6 - 1 7 - 0	· · · · ·	••	s - 0. 6 - 1.	3 3.	• • •	ь: ° - з г - та	53 •33• •113•	•	1 3 1 3 3 3	.ð 4. 1 2. .6 3.		1 2 3 4	6.8 7 1.5 2 2.9 2 6.3 6	• 1 • 0 • 0
	6 8 1 1 •	6.1 5 6.1 5	• 1	7 8 - 1 9 - 1	**9 I 2•3 2•0	3	8 - 9 10 -	3.8 4.8 3.8 4.7	•••••••••••••••••••••••••••••••••••••••	8- 9 ьа	3.0 3. 1.3 1.	::	ћ2 о б т з	й • 3 - 5 • 5 - 6	• I	7 - 3. 8 - 1. 9 - 2. 0 - 6.	3 · 3 · 9 · 1 · · · · · · · · · · · · · · · ·	•••	a = 16 3 = 0 4 = 2 5 = 9	.6 15. .8 2. .3 3.	1 0 0 1	4 - a 5 - a 7 - a	.8 3.4 •4 3.4 •2 3.4		5 - 1 6 7 8 -	3.1 3 1.9 3 0.5 0	•••
	i4 - 1	1.1		11 -	3•3 5•9	5.1		7.3 8	•• •		5.1 6 (.6 18		3 - 4	•9					6 10	5.1 13.	1	8 2	•9 4•	<u>.</u>	9	5.9 6	

<u> </u>	1 7 c	r ,	ь	r.,	r _	ь	r.,	P 0	h	Fc	r _	h	Fc	F.,	h	Fc	r ₀	h	Fc	P.,	h	r _c	P ₀	h	r _c	P _0
	h42		11	3.7	3.0		h 4 5		8	5+5	5 . 2	3 -	6.4	7 • 1	4 -	10.5	10.1	7 -	8.7	9.1	11 -	1.0	0.0	2	1.1	6.0
112	13.2	13.2	114	7.6	7 . 1	•	4+5	3.0	2	3.6	4 • I	1	6.3	5 • T	s -	10.1	10.1	8	7.2	6.1	12	2.0	3.0	3	0.2	0.0
111	0.0	1.0	13	1.6	2.0	1	0.4	0.0	10	0.0	1.0	15	5.3	5+1	6	6.2	6.1	9 -	1.7	2.0	23 -	0.5	0.0	4 -	1+3	1.0
111	3.0	3.0	122 -	1.0	2.0	1	0.5	0.0	11	2.7	3.0	1 0 -	0.0	1.0	2	3.0	3.0	10	1.6	2.0	14	0.3	0.0	5 -	1.6	1.0
1:3	- 0.1	0.0	1.2 -	0.1	0.0	3	0.4	0.0		h 4 6		2	4.7	3.0	8	1.3	1.0		1.5	1.0	1	b 5 4		6	0.9	1.0
111	- 1	1.0	1	h 4 4		11	4.9	0.1				1 8 3		0.1		1.3	1.0	122	0.2	0.0				7	1.2	1.0
1.6		0.0		0.8	1.0	13-	1.0	1.0	112	347	5.1	1.2 -	7.0	10.1	1.0 -	3 . 3	3.0	122	2.9	3.0	0 -	•• ?	6.0	8 -	3.4	1.0
1		•••	۱	0.4	8.7	15		1.0	1;-	3.7		1			1:: -	3+3		1.2		1.0	1 -	1.7	2.0	9	5.0	5+7
1	h 4 3			10.4	11.1	18-		1.0			0.0	1		1.0		0-6		l.,				3 . 2	3.0	10 -	o. '	0.C
1.	- 0.3	1.0	1.	· · · ·	· · · ·		0.0	0.0	12	1.6	1.0	1		1.0		0.3	0.0	}	h 5 3		13	1.0	c. r		0.3	0.0
1	0.6	3.0		0.6	0.0	10	1.6	3.0	6 -	1.1	1.0	114 -	0.6	0.0	114 -	1.1	6.0	۱.		-	111	1. 2	3•0		h 5 5	
1 .	15-3	16.3	1 5 -	3.0	3.0	11	0.7	1.0	7 -		3.0	15	1.1	3.0	1°,			l : _	4.	4-1	12-	1.3	2.0	1	0.1	
3	o. 1	0.0	16	5. 4	4.1	12 -	0.7	0.0	- 8 –	0. 1	0.0	16	1+3	0.0		h 5 2		1 : -	0.4	7.1	1.	1.0	0.0	1 .	4.2	4+1
4	6.6	9 . 1	1 7	3.0	3.0	13	0.5	+ 1.0	• -	0.4	1.0					1.0	0.0	1:-	y . 1	7.1	16		2.0	1	1.2	0.0
5	- 3.0	4+1	8 -	3.6	3.0	1.			10 -	0.8	1.0		h 5 1		ĩ	0.5	0.0	1 : -		4.1	1.5		4.0	4	3. 7	3.0
1 6	−. 5• ?	4 • 1	9	3.4	3.0	1			11	0.7	0.0	•	10.3	8.1	- c	1.1	1.0		4.0	2.0	110	0.8	0.0	< -	1.2	1.0
1 7	- 6,3	5.2	10 -	· 1+1	3.0	1	7.6	8.1		b b 7		1 -	2.7	3.0	3 -	10.6	10.1	6 -	5.7	5.1	11	4.0	5.0	6	1.5	I.c
1 8	- 7.8	7.1	111 -	. 1.1	3.0	3 -	4.8	6.1				1	7.6	6.1	4	10.3	9.1	1 2	2.1	3.0	12	1.4	1.0	2	1.1	1.0
1.?	- 3• ?	3+0	113 .	• 1•3	0.0	3 -	3.0	3.0	• •	1.8	3.0	3	3.6	5.0	<u>s</u> -	6.0	5+T	8	1.1	3.0	1 .	0.1	1.0	8 -	3.0	2.0
1::	- 1.4	1.0	133 -	s	3.0	1 4 -	0.8	0.0	1 -	2 • 7	3.0	4 -	3 . 2	3.0	6 -	1.3	0.0	0	1. 1	2.0	1.			9	•• •	0.0
111		3.0	114	• ••7	1.0	15-	10.0	13.7		6.6	7•I	5 -	1.7	2.0	7	2.7	3.0	10	3. 2	1.0	1	n 5 4		14	0.5	0.0
111	- 0.7	1.0	1 5	••5	1.0	<u>°</u>	1.0	2.0	3	0.1	0.0	6 -	0.6	0.0	8	0.7	1.0	11 -	0.3	0.0	1 -	1.2	0.0		n 5 6	
122	- 1.4	0-0		6 6 6		121		4+1	1.1.7	1.5	3.0	1 7 -	7 • 3	5.I	9	4+3	5•1	1.2	3 . 4	3.0	2 -		3.0	ι,	1.0	4 - 7
Ele	6	0.0	1			1 2 -		0.0	15	0.9	1.0	8	0.2	0.0	10	•• !	1.0	13	1+7	3.0	3 -	1.6	c.o	1	1.6	3.0
116	- 6.0	1.0		3.7	3.0	1.2 -	3.7	3.0	12	3.4	3.0	9 -	0.8	0.0	111	2.8	1.0	14 -	1 • 3	0.0	4	6.3	6.1	1 3	1.8	2.0
1				16.7	3.0	1	3+3	4.1	17	0.7	1.0	10	3+ 3	3.0	12	4 . 3	3+0	15 -	0.1	0.0	5	0.2	0.0	1		0.0
	ь 4 3		1.1	7.1	- 6. 7	114 -		3.0		h 4 7		111 -	0.7	1.0	13	0.7	0.0	1	h 5 3		6	4.8	5+I	5	0.3	0.0
1 7	- 7.8	7.1	12.		4.0	1		2.4	1 -	0.1	0.0	122 -	3.0	1.0	1:: 1		1.0	1.			2	1.3	3.0	6 -	0.2	0.0
1.3	8.5	8.1	16.		3.0		h46		4	0.6	3.0	1:3	4.9	1.0	- 5 -		1.0	1:-	0.9	0.0	1 *	1.7	3.0	17	3 . 7	3.0
3	5.4	5.1	17	3.6	1.0	0	0.5	0.0	3 -	r.8	3.0	1:: "		0.0				1		1.0	9	3.1	3.0	8 -	· 2. 3	1.0
14	- 5.1	5.1	18	0.7	.0.0	1	3.5	3.0	14-	0.3	0.0	1:2 -	3.0	3+0	1	n 7 Z			0.5	2.1	10 -	0.3	0.0	1	h 5 6	
l s	2.7	4.1	6	0.6	0.0	1 2 ~	2.3	3.0	5	1.3	0.0	1	. Je I	0.0	1 1 -	. 1.4	3.0	12	7.1	6.1	111	0.0	0.0	1		
6	- 0.9	1.0	10 .	4.8	6.1	3 -	3.0	4.1	6	2.6	0.0	1			1	0.0	0.0	6 -	1.6	2.0	114 -	1.4	1.0	1	- 3.8	3.0
7	- 8.0	7 . 1	11 .	0.4	0.0	14-	3.	5.1	l							11.8	10.1	1 7	4. 7	4.1				1	1.2	0.0
8	- 0.6	1.0	12.	- 3.7	3.0	5	0.5	0.0		h 5 O		1 1	7 • 4	7 . 1	14.	8.6	8.1	l é l	1.1	1.0	1	n 5 5		3.	• • • •	3.0
9	- I.Q	2.0	13	0.5	0.0	6 -	2.4	3.0	1	4.9	5+ T	1	· 9•1	10.1	Ś	9.3	9.1	9 -	1. 2	3.0	0 -	3. 7	1.0	14.		1.0
110	- 3.3	3.0	14	0.5	0.0	7	0.8	1.0	1.2	0.1	0.0	3	10.6	13+3	6 -	4.0	4 . 3	10	0.4	0.0	1 1 -	1.6	1.0	12	-	2.0

Table 1 (cont.)

The author wishes to express his thanks to Dr J.P.G. Richards for helpful discussion, to Professor A.J.C. Wilson, F.R.S., for his interest and encouragement and for use of apparatus purchased with a Royal Society grant, Imperial Chemical Industries Ltd for financial aid for computing, Dr B.C. Stace of British Nylon Spinners Ltd, Pontypool (now at Battersea College of Technology), for supplying the crystal samples and the infrared information, and the referee for his valuable comments.

Table 2. Final atomic coordinates with the corresponding temperature factor B

	x/a	у/Ь	z/c	В
O (1)	0.408	0.215	0.356	4·18 Å2
O(2)	0.282	0.239	0.186	5.01
N	0.064	0.402	0.878	3.73
C(1)	0.472	0.147	0.854	4.08
C(2)	0.084	0.467	0.088	4.21
C(3)	0.124	0.230	0.830	4.25
C(4)	0.118	0.171	0.615	4.25
C(5)	0.318	0.500	0.430	4.05
C (6)	0.334	0.303	0.310	3.68



Fig. 5. Intramolecular interatomic distances and angles.

Table 3. Comparison of the final calculated hydrogen atomic positions with those measured from the final F_o - F_c synthesis

		х	:/a	y	b/b	z/c			
		Calc.	Meas.	Calc.	Meas.	Calc.	Meas.		
[C(1)]	H(11)	0.457	0.458	0.276	0.264	0.947	0.947		
	H(12)	0.460	0.462	0.185	0.192	0.701	0.698		
[C(2)]	H(21)	0.153	0.152	0.512	0.507	0.100	0.104		
	H(22)	0.072	0.069	0.336	0.336	0.191	0.158		
[C(3)]	H(31)	0.189	0.186	0.276	0.270	0.863	0.863		
	H(32)	0.107	0.106	0.099	0.108	0.921	0.891		
[C(4)]	H(41)	0.054	0.060	0.117	0.102	0.582	0.569		
	H(42)	0.132	0.160	0.302	0.262	0.523	0.530		
[C(5)]	H(51)	0.321	0.325	0.464	0.477	0.584	0.564		
	H(52)	0.255	0.264	0.554	0.572	0.396	0.386		

Table 4(a). Bond lengths a	and other inte	eratomic distances	Table $4(b)$ (cont.)						
with their sto	andard devia	tions	Angle	Value	e.s.d.				
N(0) is the nitrogen atom i	n an adjacent	molecule bonded	C(2)-N-C(1)	110	1				
to O(1) by H(0).		C(3) - N - C(1)	114	1				
	Length	esd	N-C(2)-C(1')	110	1				
		0.01	N-C(1)-C(2')	111	1				
O(1) - C(6)	1.31 A	0.01 A	C(6)-O(1)-N(0)	111	1				
O(2) - C(6)	1.21	0.01	C(6)-O(1)-H(0)	105					
C(6) - C(5)	1.54	0.01	O(1)-H(0)-N(0)	163					
C(5) - C(4)	1.53	0.01	C(3)-N-C(2)	107	1				
C(4) - C(3)	1.50	0.01							
C(3)–N	1.51	0.01	Defen	-					
N-C(2)	1.49	0.01	Kelerences						
C(2)-C(1')	1.51	0.01	BROOMHEAD, J. M. (1951), Acta Cryst. 4, 92.						
N-C(1)	1.47	0.01	$C_{\text{RUCKSHANK}} D W I (1967)$	$\mathbf{U} = \mathbf{U} \mathbf{U} \mathbf{U} \mathbf{U}$	Vorld List of Cryst-				
C(1)-C(2')	1.51	0.01	chulceshalle, D. W. J. (1902)	Einst 1					
O(1) - N(0)	2.60	0.01	anographic Computer Frogra		Eu. p. 21.				
O(1) - H(0)	1.52		DONOHUE, J. (1952). J. Phys. C	Inem. 56, 3	502.				
H(0)-N(0)	1.11		DYER, H. B. (1951). Acta Crys	st. 4, 42.					
			GRANT, D. F., HINE, R. & RICHARDS, J. P. G. (1960). Acta						
Table $4(b)$ Bond angles	with their s	tandard deviations	Crvst. 13, 996.						
			HIROKAWA, S. (1955). Acta C	rvst. 8. 637	7.				
Angle	Value	e.s.d.	MATHIESON A MCI (1953)	Acta Cryst	6 399				
O(1)-C(6)-O(2)	125°	1°	PLATINESON, A. MCL. (1999).	$\frac{1010}{10} = 0.000$	(0, 5)				
O(2) - C(6) - C(5)	122	1	Prive London Encomon	N, A. L. (15	oo). The Hydrogen				
O(1) - C(6) - C(5)	113	1	Bona. London: Freeman.	1 100 0					
C(6) - C(5) - C(4)	113	1	POTTER, R. (1962). Nature, Lond. 193, 673.						
C(5) - C(4) - C(3)	110	1	STACE, B. C. (1962). Private communication.						
C(4) - C(3) - N	112	1	WILSON, A. J. C. (1942). Natu	re, Lond. 1	1 50 , 152.				

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The Crystal Structure of a Sodium Molybdenum Bronze

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The sodium molybdenum bronze of reported composition Na_{0.9}Mo₆O₁₇ has a monoclinic structure with space group C2/m, C2 or Cm, and cell dimensions a=9.57, b=5.50, c=12.95 Å, $\beta=90^{\circ}$; and two formula units per unit cell. Crystals are always repeatedly twinned about the normals to (310) and (310), and the composite reciprocal lattice has trigonal symmetry P3m1. The corresponding 'average structure', representing a disordered superposition of various domain arrangements, was solved by three-dimensional Patterson methods, and refined by differential Fourier techniques. It is a trigonally distorted perovskite structure, in which the sodium ions are ordered in one sixth of the voids left between the MOO₆ octahedra; and it requires either a ratio Mo:O=0:18 or randomness in the distribution of 17 oxygen atoms over 18 sites. The further distortion of 34 oxygen atoms in the unit cell. The detailed environments of the atoms are discussed.

Introduction

A recent report by Wold, Kunnmann, Arnott & Ferretti (1964) describes what is probably the first synthesis of alkali-metal molybdenum bronzes. The success of this preparation is dependent upon both the reduction temperature and the molar ratio of alkali-metal molybdate to molybdic anhydride. The reduction was carried out at a platinum cathode in a melt of the above components.

The structures of the potassium molybdenum bronzes have been described (Graham, Stephenson, Wadsley & Wold, 1965). The present work concerns a sodium molybdenum bronze reported by Wold *et al.* to be within the composition range $Na_{0.93}Mo_{5.9}O_{17}$ to $Na_{0.87}Mo_{5.95}O_{17}$ and thus to approach the composition $NaMo_6O_{17}$; a three-dimensional structure analysis has been made.

Determination of the unit cell and twin relationships

Crystals of the sodium molybdenum bronze, which were apparently single, invariably gave Weissenberg photographs indicating twinning, as shown by the fact