Table 6. *Predicted unit cell dimensions of 'homologues'* $Nb_{3n+1}O_{8n-2}$

System: monoclinic. Space group: *P*2 (or *Pm*)

n in formula $Nb_{3n+1}O_{8n-2}$	Possible compound	*a*	*b*	*c*	*β*
9	$Nb_{28}O_{70}$ (=Nb_2O_5)	21·16 Å	3·822 Å	19·35 Å	119·8°
7	$Nb_{22}O_{54}$ (=$NbO_{2·454}$)	21·2	3·82	15·6	124·5
5	$Nb_{16}O_{38}$ (=$NbO_{2·375}$)	21·2	3·82	12·0	132·2

$$a=18·86,\ b=3·822,\ c=15·75\ \text{Å},\ \beta=102°\ 11'.$$

This can be changed by the axial transform $101/010/00\bar{1}$ to

$$a=21·86,\ b=3·822,\ c=15·75\ \text{Å},\ \beta=122°\ 30',$$

and by comparison with Table 6 it appears that this phase could indeed be a homologue of Nb_2O_5 with a structure closely related to Fig. 10, if not identical with it. As yet there is no evidence that $Nb_{16}O_{38}$ exists. Homologues $Nb_{3n+1}O_{8n-2}$ based upon this principle will arise only when *n* is odd, as significant structural changes are present when *n* is even (Roth & Wadsley, 1964).

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The Crystal Structure of 3,3,4,4-Tetrahydrofurantetrol (An Application of Vector Verification)

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(*Received* 15 *July* 1963 *and in revised form* 30 *January* 1964)

A three-dimensional crystal structure analysis of 3,3,4,4-tetrahydrofurantetrol has determined that this molecule exists in a twisted configuration (44° ring twist), that the space group is $P\bar{4}2_1c$ with eight molecules per unit cell, and that the molecules lie in layers which are held together and interconnected by hydrogen bonds.

The trial model was obtained by a new method called vector verification. The method systematically checks, by applying space group symmetry and other conditions, each point in a three-dimensional grid in electron density space. At each grid point, a series of vectors is computed and compared with the observed Patterson function, and only if the vectors exist in the Patterson map can the grid point be considered as a possibility for an atomic position. The trial model was refined using full matrix least squares to a final model having an agreement index of 0·11.

Introduction

Spectrograms in the ultraviolet, infrared and nuclear magnetic resonance reveal stabilization of the heterocycle, tetrahydro-3,4-furandione, in the dione form with no evidence of enolization. The dione rapidly adds water or alcohol and tends to exist with one or both ketone groups doubly substituted as a tetrol, hydroxydioxane, hydroxydioxolane or hemiketal (Kendall & Hajos, 1960). The subject of this investigation is the tetrol (3,3,4,4-tetrahydrofurantetrol). A small well-formed crystal was carefully selected and, because of instability in contact with air, was sealed in a glass capillary tube.

Crystal data

The unit cell was determined as tetragonal from an analysis of precession films with dimensions:

$$a=b=10\cdot66\pm0\cdot02,\ c=10\cdot01\pm0\cdot02\ \text{Å}\ .$$

Systematic extinctions (*hhl* absent for *l* odd and *h*00 absent for *h* odd) established the space group as $P\bar{4}2_1c$. The calculated density of 1·58 g.cm^{-3}, assuming 8 molecules per unit cell, compares favorably with the observed density of 1·57 g.cm^{-3} as determined by flotation.

The data were collected with a General Electric Goniostat using Zr-filtered molybdenum radiation and then corrected for Lorentz and polarization factors (Gorres & Jacobson, 1963). A 2θ scan technique was used in which the reflections were scanned at a rate of 2°.min^{-1} over a range of 4°. A 20 sec background count was measured at each end of the scan. Although this method should be modified if intensities of high accuracy are desired, it allowed us to obtain reasonable accuracy in a minimum amount of time (800 reflections in 50 hours). Since the longest dimension of the crystal was approximately 0·5 mm and a glass capillary had to be used, no absorption correction was applied.

Determination of structure

A three-dimensional sharpened Patterson function was calculated as follows:

$$P(u,v,w)=\frac{1}{V}\sum\sum\sum_{hkl}|F^m_{hkl}|^2\cos 2\pi(hu+kv+lw)$$

where

$$F^m_{hkl}=(\sin^2\theta/\lambda^2+0\cdot16)\,((F_{hkl})^2/\hat{f}^2)\exp\,(B'\sin^2\theta/\lambda^2)$$

and

$$\hat{f}=\Sigma f_j/\Sigma Z_j\ .$$

The expression $(\sin^2\theta/\lambda^2+0\cdot16)$ is specific to derivative sharpening (Jacobson, Wunderlich & Lipscomb, 1961). The approximate value of the temperature factor, *B*, was determined from a Wilson plot and *B'* was set at somewhat less than twice the value of *B*.

Over a period of several months, superposition techniques were used in unsuccessful attempts to determine a good trial structure. Most of the difficulty was due to the high density of the peaks in the Patterson map, approximately 4·5 peaks.Å^{-3}. No single peaks were clearly discernible and thus it was difficult to find a satisfactory point on which to initiate superpositions. Also the results of the superpositions gave a great many spurious peaks in addition to the correct ones. This coupled with the fact that there was an unexpected ring twist made it difficult to recognize fragments of the molecule in the results of the superpositions.

Therefore, it was decided to attempt to develop a new method which uses space group symmetry in a systematic way to analyze the Patterson map. This method, called vector verification, was first tested on a known compound (Mighell & Jacobson, 1963) and was successful. The method was then applied to the 3,3,4,4-tetrahydrofurantetrol, and it enabled us to determine a good trial structure. The vector verification method consists in testing each point on a three-dimensional grid in electron density space to determine if it can represent a possible atomic position. First, a grid point, in order to represent an actual atomic position, must, as a minimum, satisfy certain symmetry requirements. These requirements are simply that the vectors between the grid point and the related positions lie in the Patterson map. Second, if the position of one or more atoms is known or assumed, vectors between the grid point and those atoms must also lie in the Patterson map. Thus in the program for the CDC 1604 computer, three major steps are involved: the storage of the appropriate three-dimensional Patterson map, the symmetry check, and the check against a tentative atomic position.

Storage of the Patterson map

The 80 × 80 × 80 grid points of the three-dimensional Patterson map for this compound were read into the memory of the computer. Each point of the map was read into the computer from magnetic tape and stored either as a one, if the value of the Patterson function was greater than a threshold value, or as a zero if below the threshold value. Because of the 48-bit word size in this computer, 48 points were stored per word. The threshold value chosen was three-fourths of the carbon–carbon single peak height.

Symmetry check

With the Patterson map stored in the memory of the computer, the second part of the vector verification method, the symmetry check, was performed. Fig. 1 is a schematic diagram of the symmetry check. A grid of 80 × 80 × 80 points in electron density space was scanned and each point tested to determine if it could be a possible atomic position. First, at each test (grid) point the program generated the remaining seven equivalent points in the set of eight required by the space group symmetry. Next the seven vectors were calculated between the test point and the equivalent points, and finally the Patterson map was referenced to determine if the calculated vectors were present. Only if all seven vectors were found in the Patterson map was a given test point considered a possible atomic position.

Fig. 2 shows one section (80 × 80) after the symmetry check had been applied at each grid point in this investigation. The *x*'s represent those points which survived the symmetry test. In the space group $P\bar{4}2_1c$, for every given point which survived the symmetry check, then, owing to directional ambiguities, there

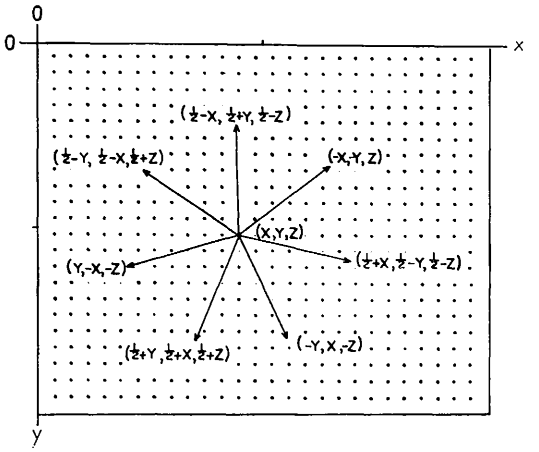

Fig. 1. Symmetry check: A schematic diagram illustrating the equivalent points and the seven vectors generated from a typical point.

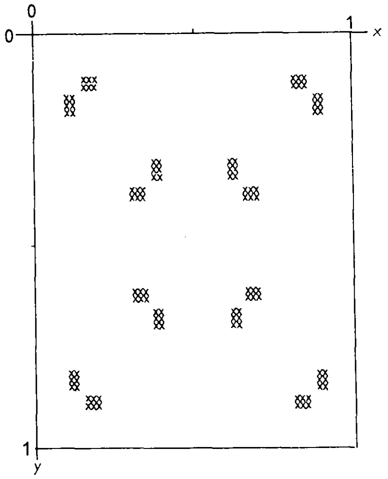

Fig. 2. A typical section (80 × 80) illustrating the results of the symmetry check on 3,3,4,4-tetrahydrofurantetrol. The crosses indicate the points that passed the symmetry check and thus represent possible atomic positions in x, y, z space.

must be 63 other points related to the first by mirror planes that will also satisfy the symmetry check. For example, Fig. 2 shows 16 such clusters, and there are also three other sections in the unit cell that are identical. However, of the set of 64 positions, assuming it is a correct set, only eight positions will usually be retained in the final structure. In this case, by imposing space group symmetry, 90% of the grid points were eliminated as possible atomic positions. In order to find the correct sets of eight and to eliminate the extraneous sets, further vector conditions must be evolved and checked for consistency in the Patterson map.

Tentative atom procedure

By careful analysis of the results of the symmetry check, an atomic position was tentatively selected. The group of crosses in the upper left hand corner of Fig. 2 was selected as the tentative atom, though any of the clusters in the set would have worked equally well. As the tentative atom's position was not known exactly, a small cubical region of 3 × 3 × 3 points was chosen instead of a single grid point. With this choice, further conditions are imposed on any other possible atomic positions, namely, that the vectors from any possible atomic position to the symmetry set of the tentative atom must lie in the Patterson map. At each grid point eight new vectors were calculated and checked for their presence in the Patterson map. These new vectors were those between the test grid point and the tentative atom set of 8 equivalent positions. Fig. 3 shows diagrammatically the total of 15 vectors (symmetry plus tentative atom vectors) which must lie in the Patterson map for the test point to be considered a possible atomic position.

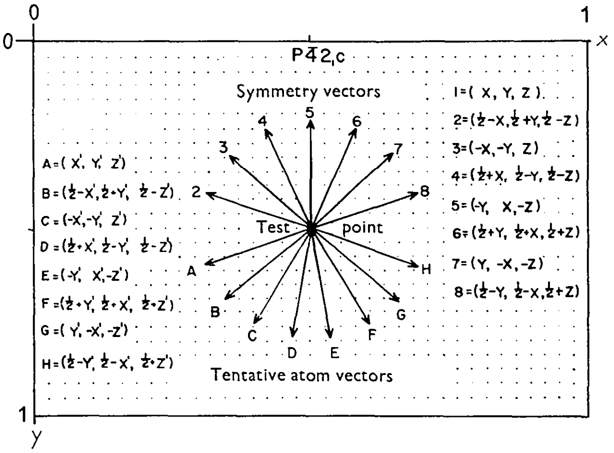

Fig. 3. Tentative atom procedure: A schematic diagram illustrating the 15 vectors generated at a typical test point. The vectors with the numerals are the symmetry check vectors and those with the letters are the tentative atom vectors.

With the application of the tentative atom procedure, many of the spurious peaks were now eliminated. It should be noted at this point that if the atomic position tentatively chosen does not correspond to a true position, no meaningful results will be forthcoming. As a means of confirmation, a peak was selected from the results of the above tentative atom procedure and was used as a second tentative atom and the method repeated. The two maps resulting from the two different tentative atoms were then compared and each found to have the same set of six well-formed peaks, plus a few others. It was further demonstrated that if any one of these six peaks were used as a tentative atom the other five appeared in the output map, and that if a wrong set were used, the resulting map contained few well-formed peaks. Although further tests of this type were not necessary, the results provided added confirmation as to the validity of these atomic positions.

Table 1. *The final atomic coordinates and temperature parameters*

	x/a	y/b	z/c	B		x/a	y/b	z/c	B
O(1)	0·1721	0·2240	0·4102	—	H(10)	0·1578	0·0420	0·3698	—
C(2)	0·1013	0·3066	0·3308	1·74	H(11)	0·2920	0·0835	0·3583	—
C(3)	0·0611	0·2317	0·2094	1·28	H(12)	0·0284	0·3220	0·3637	—
C(4)	0·1772	0·1440	0·1928	0·98	H(13)	0·1602	0·3727	0·3162	—
C(5)	0·2052	0·1146	0·3358	1·51	H(14)	0·1334	−0·0183	0·1343	—
O(6)	0·2749	0·2127	0·1436	—	H(15)	0·2692	0·2529	0·0841	—
O(7)	0·1580	0·0423	0·1108	—	H(16)	−0·0917	0·1696	0·2460	—
O(8)	0·0402	0·3097	0·1017	—	H(17)	−0·0149	0·2754	0·0792	—
O(9)	−0·0395	0·1509	0·2355	—					

The anisotropic temperature factors for oxygen are expressed in the form

$$T = \exp\left(-B_{11}h^2 - B_{22}k^2 - B_{33}l^2 - B_{12}hk - B_{23}kl - B_{31}hl\right)$$

	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{31}
O(1)	0·0129	0·0072	0·0021	0·0038	−0·0036	−0·0047
O(6)	0·0044	0·0036	0·0032	0·0011	0·0025	−0·0026
O(7)	0·0042	0·0023	0·0028	−0·0005	0·0022	0·0035
O(8)	0·0043	0·0041	0·0036	−0·0030	−0·0025	0·0009
O(9)	0·0027	0·0066	0·0038	−0·0015	−0·0044	0·0002

With these six positions in the asymmetric unit, an electron density map was calculated. The remaining three atomic positions were found by an inspection of the electron density map and the maps from the tentative atom procedure. These three peaks were all present in the results of the symmetry check and the tentative atom procedure, but were not as well-formed as the first six. The trial model led to a structure having an agreement index of 11%.

Refinement of the structure

The structure was refined with a full matrix least squares program (Muller & Jacobson, 1963) on F_o^2 using 449 independent reflections. Data were taken slightly beyond the copper sphere and reflections having a measured F_o below 3 were considered as unobserved because of the highly unfavorable ratio of background to the net intensity. Though the compound of this study is in the tetragonal space group $P\bar{4}2_1c$, it was refined in the orthorhombic space group $P2_12_12$. The asymmetric unit in the lower space group contained the two molecules related by the fourfold inversion axis in the higher space group. The data were essentially doubled by introducing the alterant *hk* combination. Various types of weighting factor schemes were investigated. But the film weighting scheme (Hughes, 1941) was found the most satisfactory since the plots of $\omega\Delta^2$, where $\Delta = [F_o^2 - F_c^2]$, as a function of the magnitude of F_o and $\sin^2\theta$ were the most constant. As a further test, the model was also subjected to refinement on F_o and no significant discrepancies were noted.

From a difference map, four well-formed peaks of approximately $\frac{3}{5}$ e.Å^{-3} were readily assigned to the four hydrogen atoms attached to carbon atoms 2 and 5 (Fig. 6) and these positions gave reasonable bond distances and angles (Table 3). The location of the hydroxyl hydrogens was more difficult. Six peaks were left in the difference map and on the basis of bond distances and angles four of these were chosen as the hydrogen positions. These four positions placed the hydrogen atoms between different close intermolecular oxygen–oxygen distances ranging between 2·65 and 2·84 Å (Fig. 5). All of the hydrogen to oxygen distances, however, were short and even on refinement these bonds showed no tendency to lengthen.

Analysis of a difference map indicated some anisotropic character in the oxygen atoms but none was noted for the carbons. Therefore, in the final series of refinements only the oxygen atoms were refined anisotropically. The anisotropic temperature factors and their ellipsoid analysis are given in Tables 1 and 2. Although the values of the thermal parameters are only approximate, this analysis indicates that the ring oxygen may be the most anisotropic.

Table 2. *Analysis of anisotropic temperature factors*

	Axis vector	Direction cosines $\times 10^3$ (relative to the *a*, *b* and *c* axes respectively)			Amplitude (Å $\times 10^2$)
Oxygen 1	1	−87	−29	21	28
	2	33	−86	16	20
	3	14	21	96	8
Oxygen 6	1	−81	5	50	17
	2	26	80	43	16
	3	38	−48	75	9
Oxygen 7	1	77	12	55	17
	2	−37	77	41	13
	3	−37	−53	72	7
Oxygen 8	1	60	−63	37	19
	2	−64	−25	67	14
	3	34	65	63	11
Oxygen 9	1	14	−84	43	21
	2	−79	11	52	13
	3	49	41	73	11

The final atomic coordinates are given in Table 1 and the bond angles and distances in Table 3. The final agreement factor, $R = \Sigma\,||F_o| - |F_c|| / |F_o|$, was 0·11.

Agreement factors were computed for special classes of reflections which depended on the nature of the indices, the magnitude of the F_o and the value of $\sin \theta/\lambda$, and no significant discrepancies were noted. The average standard deviations, as determined by diagonal matrix approximation, are 0·004 Å for oxygen, 0·005 Å for carbon and 0·030 Å for hydrogen. The standard deviations in the bond distances are reported in Table 3. Even though the standard deviation on the O–H bond lengths from least squares is 0·03 Å, it is quite likely that the actual standard deviation is larger. This discrepancy along with the short O–H distances probably reflects errors, both in the data and in the scattering factor curves, not taken into account in the least squares treatment.

Table 3. *Intermolecular bond distances, angles, and errors*

Bond distances

Ring distances	
C(3)–C(4)	1·56 Å
C(2)–C(3)	1·52
C(4)–C(5)	1·50
C(5)–O(1)	1·43
C(2)–O(1)	1·41

Carbon–hydroxyl	
C(4)–O(6)	1·37
C(4)–O(7)	1·38
C(3)–O(8)	1·39
C(3)–O(9)	1·40

Hydrogen–carbon	
H(11)–C(5)	1·01 Å
H(10)–C(5)	0·99
H(13)–C(2)	0·96
H(12)–C(2)	0·86

Hydrogen–oxygen	
H(14)–O(7)	0·74
H(15)–O(6)	0·74
H(16)–O(9)	0·60
H(17)–O(8)	0·73

Bond angles

Ring angles	
C(2)–O(1)–C(5)	110·1°
O(1)–C(2)–C(3)	106·2
O(1)–C(5)–C(4)	106·5
(C(5)–C(4)–C(3)	100·4
C(4)–C(3)–C(2)	100·2

Hydroxyl carbon	
O(9)–C(3)–O(8)	113·1
O(9)–C(3)–C(2)	112·8
O(9)–C(3)–C(4)	105·0
O(8)–C(3)–C(2)	111·1
O(8)–C(3)–C(4)	113·8
O(7)–C(4)–O(6)	108·5
O(7)–C(4)–C(5)	116·1
O(7)–C(4)–C(3)	114·7
O(6)–C(4)–C(5)	107·9
O(6)–C(4)–C(3)	108·8

Hydrogen–carbon	
H(13)–C(2)–H(12)	120·8°
H(12)–C(2)–C(3)	99·0
H(12)–C(2)–O(1)	112·6
H(13)–C(2)–C(3)	116·6
H(13)–C(2)–O(1)	101·3
H(11)–C(5)–H(10)	97·7
H(11)–C(5)–C(4)	117·8
H(11)–C(5)–O(1)	112·1
H(10)–C(5)–C(4)	113·2
H(10)–C(5)–O(1)	109·4

Hydrogen-oxygen	
H(14)–O(7)–C(4)	123·4
H(15)–O(6)–C(4)	122·9
H(16)–O(9)–C(3)	122·6
H(17)–O(8)–C(3)	94·2

Standard deviations

C–C ± 0·01 Å
C–O ± 0·01
C–H ± 0·03

Discussion of the structure

The structure of this compound is characterized by two interesting features, namely the twist in the ring and the amount of hydrogen bonding used in the

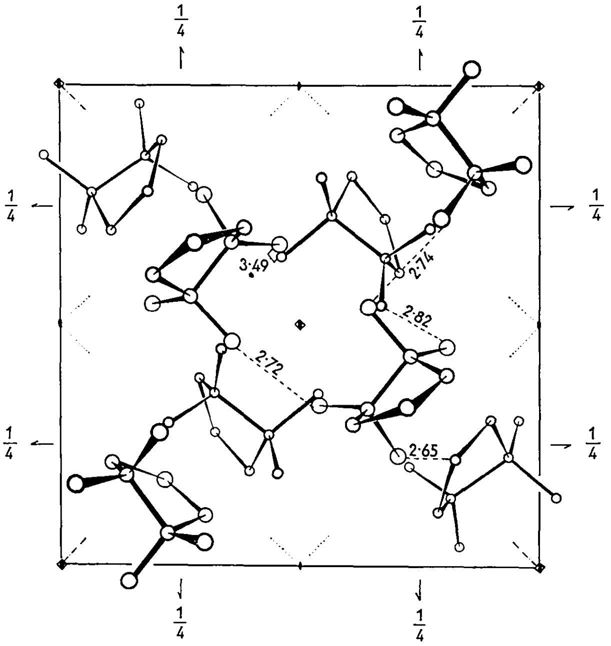

Fig. 4. Projection down the *c* axis illustrating the molecular packing and the twist in the ring.

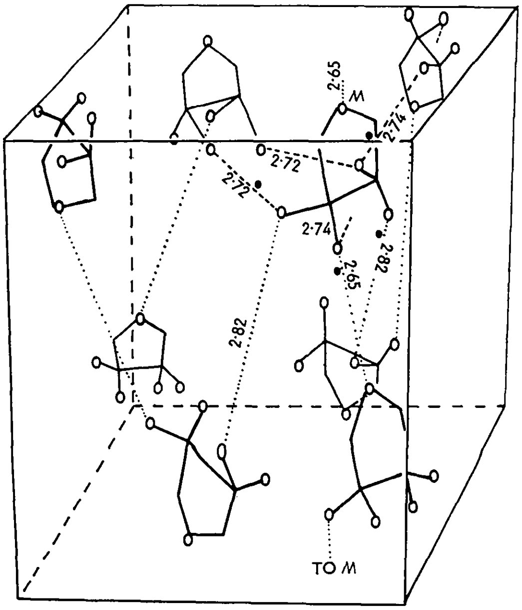

Fig. 5. A three-dimensional view showing the two layers in the structure, the proposed hydrogen bonding about one molecule, and all the closest intermolecular oxygen–oxygen approaches. The oxygen atoms are indicated by the open circles and the hydrogen atoms by the filled circles.

Table 4. *Final structure factors*

H	K	L	F(OBS)	F(CALC)
0	0	0		576
0	0	2	147.7	132.9
0	0	4	142.3	129.8
0	0	6	26.6	30.2
0	0	8	18.6	18.0
0	0	10	25.8	20.5
0	0	12	14.7	13.6
0	1	1	43.5	36.9
0	1	3	38.6	35.2
0	1	4	11.8	13.9
0	1	7	26.3	22.2
0	1	8	19.7	19.6
0	1	9	36.6	35.3
0	2	0	113.8	113.8
0	2	1	72.7	69.0
0	2	2	9.7	8.5
0	2	3	27.8	26.0
0	2	4	58.3	56.3
0	2	5	43.4	40.0
0	2	6	21.8	19.8
0	2	7	16.9	16.2
0	2	8	12.8	11.3
0	2	9	12.8	12.2
0	3	1	46.7	49.7
0	3	2	24.6	25.9
0	3	4	46.9	46.4
0	3	5	58.6	57.4
0	3	6	24.5	25.9
0	3	9	10.8	6.5
0	3	12	15.4	16.0
0	4	0	27.3	27.7
0	4	2	20.1	19.2
0	4	3	47.6	48.2
0	4	4	29.3	29.6
0	4	9	12.4	14.9
0	4	11	9.6	12.4
0	5	2	22.2	22.4
0	5	3	27.6	28.7
0	5	4	33.8	33.2
0	5	5	31.9	30.9
0	5	7	14.2	12.5
0	5	8	25.2	26.2
0	5	9	12.0	9.0
0	5	11	10.3	9.2
0	5	12	13.4	16.8
0	6	0	28.6	28.2
0	6	3	29.4	28.6
0	6	6	19.0	18.1
0	6	7	9.7	7.2
0	6	9	13.5	12.8
0	6	10	13.1	11.5
0	7	1	19.0	17.9
0	7	2	17 7	18.3
0	7	4	13.7	13.4
0	7	5	16.0	15.8
0	7	6	23.3	23.5
0	7	8	27.4	26.8
0	8	0	29.7	29.0
0	8	1	20.8	19.5
0	8	3	24.9	24.2
0	8	4	13.3	13.5
0	8	5	10.5	9.8
0	8	9	9.7	13.1
0	8	10	12.7	8.6
0	9	1	25.0	25.3
0	9	2	17.0	18.5
0	9	3	26.4	27.8
0	9	4	20.6	22.7
0	9	9	15.4	8.0
0	10	0	13.9	14.6
0	10	3	11.5	12.0
0	10	4	15.0	13.0
0	10	6	24.5	25.1
0	10	8	11.1	6.7
0	11	1	9.9	9.1
0	11	3	9.6	10.3
0	11	4	13.2	10.2
0	11	6	11.8	.9
0	11	7	10.7	6.9
0	12	0	19.7	20.9
0	12	3	12.5	11.7
0	12	5	11.6	10.8
0	13	1	10.0	3.8
0	13	4	11.5	10.0
1	1	0	70.0	64.2
1	1	2	45.7	41.3
1	1	4	35.1	31.6
1	1	6	18.0	19.3
1	1	8	15.2	14.0
1	1	10	20.2	18.9
1	1	12	11.4	4.3
1	2	0	23.9	21.6
1	2	1	88.2	87.0
1	2	2	17.2	15.1
1	2	3	46.2	43.8
1	2	4	19.1	17.4
1	2	5	28.9	30.0
1	2	6	26.7	25.1
1	2	7	13.4	14.1
1	2	8	16.0	13.1
1	2	9	26.6	25.3
1	2	10	13.8	14.0
1	2	11	10.8	8.0
1	3	0	32.9	32.7
1	3	1	20.2	21.7
1	3	2	22.4	24.6
1	3	3	77.9	80.4
1	3	4	40.1	39.7
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1	6	0	21.1	20.9
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1	7	0	23.0	23.5
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2	8	9	12.6	13.9
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2	9	1	13.6	13.2
2	9	2	12.9	11.6
2	9	3	15.3	14.6
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2	11	0	15.0	12.6
2	11	1	10.9	.3
2	12	0	16.9	15.0
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2	13	1	12.2	11.1
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4	11	6	9.6	5.9
5	5	0	21.1	20.3
5	5	2	11.5	10.0
5	5	4	29.1	32.2
5	5	6	29.0	30.8
5	6	0	11.9	4.6
5	6	2	27.6	27.9
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5	6	4	13.4	13.1
5	6	5	14.5	13.6
5	6	6	17.3	19.1
5	6	9	12.0	10.2
5	6	10	10.1	12.3
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7	11	4	15.5	8.6
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8	8	2	10.6	14.2
8	8	4	11.0	10.0
8	9	4	10.4	6.2
8	9	5	12.1	9.2
8	9	6	10.3	6.6
8	10	2	11.1	8.5
8	10	3	16.5	15.3
8	10	4	13.3	76
9	10	0	11.8	9.5
9	10	1	13.7	12.2

packing. The twist in the ring may be seen in Fig. 4, which shows a projection down the *c* axis, and in Fig. 6 which shows an end view of the molecule. The twist of 44° permits a lengthening of the distance between oxygen atoms on adjacent carbons from approximately 2·5 Å in the undistorted configuration to 2·71 Å in this structure.

The molecular packing is illustrated in Figs. 4 and 5. The hydrogen bonds connect the molecules in layers parallel to the *ab* plane and also interconnect the layers (Fig. 5). Each molecule is attached to other molecules by eight hydrogen bonds in which the oxygen to oxygen distances are between 2·65 Å and 2·83 Å. The hydrogen bonds are indicated by dotted lines between the layers and dashed lines within one layer.

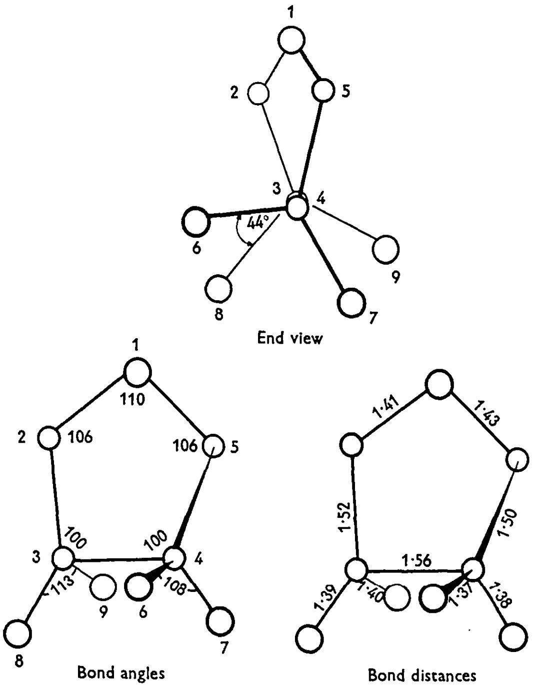

Fig. 6. The bond angles, distances and the end view of the molecule illustrating the twist of the ring.

Conclusion

For the compound of this investigation, 3,3,4,4-tetrahydrofurantetrol, the method of vector verification led to a good trial structure. In general this method should be applicable to the determination of many light-atom-type structures, or the determination of the positions of the heavier atoms where this is not obvious from the Patterson function. It should be especially useful for compounds that have four or more equivalent positions in the unit cell. Since the basic question concerns the existence of a given set of vectors in the Patterson map, this method or some variation of it is relatively easy to program on a large scale computer, and once programmed, may be readily run on a variety of compounds.

Logical extensions of the method would include running two or more tentative atoms at the same time, further processing of the results of the tentative atom procedure within the computer, and using procedures involving the selection of certain vectors from the Patterson map. Preliminary investigation indicates that the selected vector approach is particularly promising. The selected vector is allowed to scan through a grid in electron density space. At each position of the vector, all symmetry related points to the head and the tail are generated and all unique vectors are then checked. The grid points which pass this test should have a high probability of being true atomic positions, and then both of these points can be used in the tentative atom approach. For the compound of this investigation, a total of (7+7+8) vectors would be checked at each grid point.

We are indebted to Dr E. C. Kendall for synthesizing and recrystallizing the compound and for many helpful discussions in connection with the physical and chemical properties of the molecule. Our thanks are due to R. Olcott for the bond distance and ellipsoid programs, to the Army Research Office (Durham) for financial support, to the Public Health Service for support of one of us (A. M.) under Grant GPM-14, 776-R1, Division of General Medical Sciences, and to the Princeton University Computing Center for the necessary time to develop and run the vector verification program.

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