intermolecular distances and bond angles were calculated with IBM 1620 programs by Chu & Shiono (1963).

The thermal ellipsoid parameters given in Table 3 and the illustration in Fig. 1 (Johnson, 1965) show that the molecule as a whole has no marked thermal anisotropy. As in the structure of cellobiose (Brown, 1966), all the oxygen atoms appear to have higher vibration amplitudes perpendicular to their C–O bonds than along them. The methyl group has a high thermal motion consistent with its van der Waals environment around the twofold axes (see Fig. 2).

This research has been supported by the U.S. Public Health Service, National Institutes of Health, through Grant GM 11293. We wish to thank Dr R. Bentley for providing the crystals and discussing the chemistry of maltose, Dr P. W. R. Corfield for making the convolution computation, Dr R. Shiono for his help in using his computer programs and Dr R. Rosenstein for suggestions in preparing this manuscript.

References

Bentley, R. (1959). *J. Amer. Chem. Soc.* **81**, 1952.
Berman, H. & Kim, S. H. (1967). To be published.
Beurskens, P. T. (1963). *Sign Correlation by the Sayre Equation.* Technical Report, The Crystallography Lab., Univ. of Pittsburgh.
Brown, C. J. (1966). *J. Chem. Soc.* (A) p. 927.
Brown, G. M. & Levy, H. A. (1963). *Science*, **141**, 921; and private communication.
Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *Fortran Crystallographic Least Squares Program.* ORNL-TM-305, Oak Ridge National Laboratory, Tennessee.
Chu, S. C. & Shiono, R. (1963). *Technical Reports* 43 and 45, Crystallography Lab. Univ. of Pittsburgh.
Corfield, P. W. R. (1965). *Solution of the Patterson Function by Superposition Methods.* Technical Report, Crystallography Lab. Univ. of Pittsburgh.
Cruickshank, D. W. J. (1961). In *Computing Methods and the Phase Problem in X-ray Crystal Analysis.* New York: Pergamon Press.
Hamilton, W. C., Rollett, J. S. & Sparks, R. A. (1965). *Acta Cryst.* **18**, 129.
Hauptman, H. & Karle, J. (1953). *Solution of the Phase Problem.* I. *The Centrosymmetric Crystal.* A.C.A. Monograph no. 3. Ann Arbor, Mich.: Edward Bros.
Haworth, W. N., Long, C. W. & Plant, J. H. G. (1927). *J. Chem. Soc.* p. 2809.
Hughes, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737.
Hybl, A., Rundle, R. E. & Williams, D. E. (1965). *J. Amer. Chem. Soc.* **87**, 2779.
Jacobson, R. A., Wunderlich, J. A. & Lipscomb, W. N. (1961). *Acta Cryst.* **14**, 598.
Jeffrey, G. A. & Rosenstein, R. D. (1964). *Advanc. Carbohydrate Chem.* **19**, 7.
Johnson, C. K. (1965). *A Fortran Thermal-Ellipsoid Plot Program for Crystal Structure Illustrations.* ORNL 3794. Oak Ridge National Laboratory, Tennessee.
Karle, I. L., Hauptman, H., Karle, J. & Wing, A. B. (1958). *Acta Cryst.* **11**, 257.
Kim, S. H. & Jeffrey, G. A. (1967). *Acta Cryst.* **22**, 537.
Klyne, W. & Prelog, V. (1960). *Experientia*, **16**, 521.
Mathieson, A. McL. & Poppleton, B. J. (1966). *Acta Cryst.* **21**, 72.
Robertson, J. H. & Sheldrick, B. (1965). *Acta Cryst.* **19**, 820.
Sayre, D. (1952). *Acta Cryst.* **5**, 60.
Shiono, R. (1966*a*). *Data Reduction and Scaling Fortran Program.* The Crystallography Lab., Univ. of Pittsburgh.
Shiono, R. (1966*b*). Oak Ridge Least-Squares Program Modified for the Crystallography Lab. of the Univ. of Pittsburgh.
Sundaralingam, M. (1965). Private communication.

Acta Cryst. (1967). **23**, 1049

Crystal Structure of Ethyl-1-thio-α-D-glucofuranoside

By R. Parthasarathy and Raymond E. Davis*
Center for Crystallographic Research, Roswell Park Division of Health Research, Inc., 666 *Elm Street, Buffalo, New York* 14203, *U.S.A.*

(*Received* 20 *February* 1967)

The crystal structure of ethyl-1-thio-α-D-glucofuranoside ($C_8H_{16}O_5S$) has been determined by three-dimensional Patterson and Fourier syntheses. The structure was refined by block-diagonal least squares. The final R value for the structure with the correct absolute configuration was 8·9% for the 1030 observed reflections. The absolute configuration of the molecule was determined from the anomalous scattering of Cu $K\alpha$ radiation by the sulfur atom. The geometry of the molecules is, in general, in accord with previous studies on monosaccharides. The conformation of the furanose ring seems to be slightly different from that observed in other compounds. The stacking of the molecules and the hydrogen bonding scheme are illustrated.

Introduction

The furanose ring occurs in a variety of important biological molecules, particularly in the nucleic acids and other carbohydrate derivatives. Information on the conformations of the furanose moieties is of considerable importance in nucleic acid model building. The present study on ethyl-1-thio-α-D-glucofuranoside was undertaken in order to supply additional precise information about the stereochemistry of furanoid rings.

---

* Present address: University of Texas, Chemistry Department, Austin, Texas, U.S.A.

Experimental

Crystals suitable for X-ray studies were kindly provided by Dr I. J. Goldstein of the State University of New York at Buffalo. Crystals of ethyl-1-thio-α-D-glucofuranoside (hereinafter referred to as ETGF) are monoclinic, space group $P2_1$ with two molecules in the unit cell. The cell constants are $a=11\cdot118\pm0\cdot003$, $b=5\cdot662\pm0\cdot005$, $c=8\cdot842\pm0\cdot003$ Å, $\beta=108\cdot18\pm0\cdot02°$ (Cu $K\alpha=1\cdot5418$ Å); these were measured using a General Electric XRD-5 diffractometer equipped with goniostat. The density was determined by the flotation method as 1·40 g.cm^{-3}; the calculated density on the basis of two molecules in the unit cell is 1·41 g.cm^{-3}.

The intensities were measured by the stationary-crystal stationary-counter method (Furnas & Harker, 1955). Monochromatization was obtained by means of a balanced pair of Ross filters: Ni against Co. Of the 1188 independent reflections (to the limit $2\theta=150°$ for Cu $K\alpha$ radiation) which were measured, 1030 were considered to be non-zero intensity. The crystal used for data collection was a rod of dimensions 0·11 × 0·11 × 0·38 mm, and was mounted with [010] parallel to the φ axis of the goniostat. The correction for absorption as a function of angle φ was measured for the two reflections 020 and 040. The average value of the transmission factor obtained from these measurements was used for correcting the intensities approximately for anisotropy of absorption. The usual Lorentz–polarization factors were applied and an absolute scale was approximated by Wilson's method.

Determination of the structure

The specimen under investigation was initially believed to be ethyl-β-D-glucofuranoside. The calculated and measured formula weight of the unit cell differed by 34·8 atomic mass units, which might be explained by two molecules of water of crystallization. Initial attempts to interpret the sharpened three-dimensional Patterson function were not successful. In the meantime, an elementary analysis showed the presence of one sulfur atom per molecule, leading to the revised formula $C_8H_{16}O_5S$. This fact explained the difference in the formula weight of the unit cell and the compound was assumed to be a thio derivative of a glucofuranose.

The sulfur atom was located with the aid of the Harker section (at $v=\frac{1}{2}$). A three-dimensional Fourier synthesis with the phases derived from the sulfur positions was calculated. The centrosymmetrical distribution of the heavy atoms introduced spurious mirror planes passing through them. The presence of these mirrors caused an ambiguity in the positions of the atoms derived from the Fourier synthesis; this ambiguity was readily removed by calculating a Fourier synthesis with the phases due to sulfur and one other atom, not on a mirror plane. The coordinates of this additional atom were those of a strong peak in the sulfur-phased Fourier synthesis and it was assigned the atomic scattering factor of oxygen. Three cycles of iterative Fourier and difference-Fourier calculation yielded the structure. The R index at this stage was 0·19, indicating the approximate correctness of the structure.

Refinement of the structure

The structure was refined by a series of least-squares analyses of the full three-dimensional data, employing the block-diagonal approximation. Three cycles of least-squares refinement with individual isotropic temperature factors were carried out on an IBM 7044 computer. Another three cycles of refinement with individual anisotropic temperature factors reduced the R index to 0·101. A $1/f$ weighting with f for the nitrogen atom was adopted in the final stages of the refinement. An analysis of the weighting scheme was carried out to check its appropriateness. This was done by plotting $\Sigma W(\Delta|F|)^2$ as a function of $(\sin\theta/\lambda)$ and $|F_{\rm obs}|$ and noting that $\Sigma W(\Delta|F|)^2$ is reasonably independent of $|F_{\rm obs}|$ and $(\sin\theta/\lambda)$. Reflections too weak to be observed were given zero weight during the refinement and for the R index calculation. Atomic scattering factors were those presented in *International Tables for X-ray Crystallography* (1962). An electron density difference map at this stage indicated the positions of ten hydrogen atoms. Three cycles of least-squares refinement, in which the coordinates of the hydrogen atoms were also allowed to vary, brought the R index to 0·089. A structure factor calculation omitting the hydrogen atoms yielded an R value of 0·098. A final difference electron density map was calculated, but it was not possible to fix the positions of the rest of the hydrogen atoms with certainty from this map. None of the shifts in the final stage was greater than one-sixth the corresponding standard deviation, and refinement was considered to be complete. The final positional and thermal parameters and their standard deviations are listed in Table 1. These standard deviations were obtained from the diagonal elements of the block-diagonal matrix and hence, are approximate. The observed and calculated structure factors are given in Table 2. A final electron density map is shown in Fig. 1.

Determination of the absolute configuration

No direct determination of absolute configuration of a sugar derivative seems to have been reported in the literature (Jeffrey & Rosenstein, 1964). The absolute configuration of ETGF was established by utilizing the anomalous dispersion of the sulfur atom for Cu $K\alpha$ radiation.

Two different methods were used for determining the absolute configuration. The first method, due to Bijvoet, involves the measurement of the intensities of the direct (hkl) and inverse ($\bar{h}\bar{k}\bar{l}$) reflections; the results obtained for a typical set of reflections are given in Table 3. The second method (Ibers & Hamilton, 1964), involves the calculation of the R values for the two hands of the structure. The R values for the D and L configurations were 0·085 and 0·097. Using Hamilton's (1965) notation, the R ratio is 1·14. Also, from Table 1 of Hamilton (1965), $R_{1,867,0\cdot005} < 1\cdot14$. Hence, the hypothesis that the configuration is L can be strongly rejected even at the 0·0050 significance level. The results of the 'classical' and the R-factor ratio method agree and also are in agreement with the Fischer convention.

The temperature factors

The magnitudes and direction cosines of the principal axes of thermal motion, as derived from the parameters of Table 1, are listed in Table 4. The maximum root mean square amplitudes of thermal motion of various atoms range from 0·184 Å for C(4) to 0·369 Å for C(8); the minimum amplitudes range from 0·121 Å for C(4) to 0·205 Å for C(8). In general, the minimum thermal motion is along the **b** direction, nearly the direction of hydrogen bonds between the molecules.

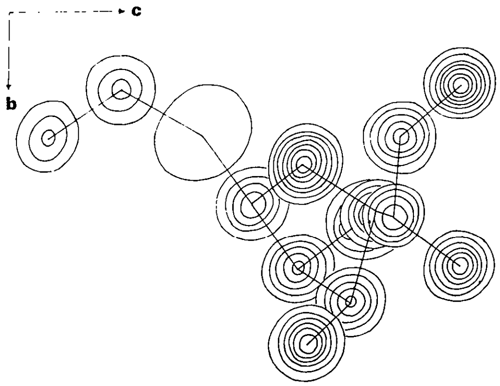

Fig. 1. A composite drawing of the final electron density map. The contours are drawn at intervals of 2 e.Å^{-3}. Only the 2 e.Å^{-3} contour is shown for S. The value of the lowest contour for other atoms is 2 e.Å^{-3}.

The geometry of the molecule

The bond distances and angles as calculated from the parameters of Table 1 are shown in Figs. 2 and 3. Their standard deviations, as calculated from the uncertainties of the final atomic parameters, are indicated in parentheses. Additional uncertainties are undoubtedly introduced owing to various effects, such as thermal libration, systematic errors in data, *etc.*, but it is difficult to evaluate their effects on the standard deviation quoted above.

All C–C bonds in the furanose ring seem to be shorter than the normal C–C single bond; their average value of 1·507 Å is considerably shorter than the C–C diamond bond of 1·54 Å. Such a shortening of the C–C bonds in the furanose ring seems to be common (see, for a review, Sundaralingam, 1965). The C(7)–C(8) bond appears to be much shorter than normal C–C bonds; however, the estimated standard deviation for this bond is 0·022 Å and this terminal bond seems to be affected considerably by thermal libration. The C(1)–S (1·769 Å) is noticeably shorter than the C(7)–S bond (1·808 Å). This is similar to the shortening of

Table 1. *Fractional coordinates and thermal parameters*

Estimated standard deviations in parentheses refer to the last decimal position reported. The expression for the temperature factor is

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

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	0·0756 (2)	0·2500	0·2201 (3)	0·0080 (2)	0·0266 (7)	0·0128 (3)	−0·0017 (10)	0·0046 (3)	−0·0028 (11)
O(1)	0·2815 (6)	0·3289 (12)	0·4741 (8)	0·0063 (5)	0·0150 (17)	0·0094 (17)	0·0046 (17)	−0·0000 (10)	−0·0061 (20)
O(2)	0·0320 (6)	0·5272 (17)	0·4734 (10)	0·0059 (4)	0·0281 (25)	0·0187 (10)	−0·0009 (19)	0·0117 (10)	−0·0021 (28)
O(3)	0·3261 (6)	0·8478 (14)	0·5031 (8)	0·0072 (5)	0·0164 (17)	0·0132 (8)	−0·0044 (18)	0·0093 (10)	−0·0006 (25)
O(4)	0·4620 (8)	0·6234 (15)	0·8415 (8)	0·0120 (8)	0·0191 (19)	0·0083 (8)	0·0003 (25)	0·0036 (12)	−0·0053 (25)
O(5)	0·4369 (7)	0·1010 (13)	0·8332 (7)	0·0086 (6)	0·0139 (18)	0·0090 (7)	0·0016 (19)	0·0041 (10)	0·0065 (21)
C(1)	0·1869 (9)	0·4471 (19)	0·3437 (11)	0·0056 (6)	0·0238 (28)	0·0087 (10)	0·0044 (24)	0·0034 (13)	−0·0037 (31)
C(2)	0·1296 (8)	0·6360 (18)	0·4200 (11)	0·0057 (6)	0·0136 (25)	0·0121 (9)	0·0008 (20)	0·0066 (11)	−0·0016 (26)
C(3)	0·2427 (8)	0·7066 (16)	0·5587 (10)	0·0062 (6)	0·0144 (25)	0·0101 (9)	0·0007 (21)	0·0080 (11)	−0·0052 (25)
C(4)	0·3034 (8)	0·4724 (15)	0·6144 (10)	0·0058 (6)	0·0108 (20)	0·0075 (10)	−0·0004 (20)	0·0038 (11)	0·0047 (26)
C(5)	0·4460 (8)	0·4829 (16)	0·7041 (10)	0·0073 (7)	0·0122 (20)	0·0066 (10)	−0·0017 (21)	0·0039 (11)	−0·0022 (26)
C(6)	0·5040 (8)	0·2433 (21)	0·7519 (10)	0·0058 (6)	0·0153 (21)	0·0106 (10)	0·0015 (24)	0·0025 (12)	0·0023 (37)
C(7)	0·1728 (14)	0·1252 (29)	0·1103 (16)	0·0127 (12)	0·0329 (40)	0·0163 (15)	0·0146 (40)	0·0074 (22)	−0·0043 (50)
C(8)	0·1855 (14)	0·2679 (46)	−0·0228 (16)	0·0148 (12)	0·0683 (80)	0·0187 (16)	−0·0167 (50)	0·0196 (21)	−0·0240 (70)

				B^*
H(1)	0·196	0·463	0·215	3·5 Å^2
H(2)	0·102	0·774	0·342	3·5
H(3)	0·215	0·822	0·632	3·5
H(4)	0·262	0·380	0·683	3·5
H(5)	0·492	0·559	0·638	3·5
H(6)	0·581	0·309	0·827	3·5
H(7)	0·262	0·071	0·188	3·5
H(8)	0·227	0·130	−0·079	3·5
H(9)	0·295	1·053	0·470	3·5
H(10)	0·482	0·814	0·807	3·5

* The B values for hydrogen atoms were arbitrarily assigned. The averages of the e.s.d. of hydrogen atoms for the x, y, and z fractional coordinates are 0·007, 0·025 and 0·015 respectively.

C(1)–O bonds reported for other carbohydrates (sucrose, Brown & Levy, 1965; β-D-glucose, Ferrier, 1963). The C–O bond lengths average to 1·435 Å; the largest deviation from the mean is for the C(1)–O(1) distance of 1·459 Å. In all the other furanoid structures which have been reported, the ring C(1)–O(1) bond is shorter than the C(4)–O(1) bond and they average to 1·427 Å and 1·450 Å respectively. In ETGF, however, this tendency seems to be reversed. Other furanoid structures [cytidylic acid, Sundaralingam & Jensen (1965);

Table 3. *Determination of the absolute configuration from the intensity measurements*

hkl	$\frac{I(\bar{h}\bar{k}\bar{l})}{I(hkl)}$ obs.	$\frac{I(\bar{h}\bar{k}\bar{l})}{I(hkl)}$ cal.*
510	0·86	0·90
51$\bar{1}$	1·18	1·18
211	1·04	1·12
32$\bar{3}$	0·84	0·91

* Calculated for coordinates corresponding to the D configuration and $\Delta f'' = 0·60$.

Table 2. *Final observed and calculated structure factors*

$$FOBS = 10·0 \times |F_o|$$
$$FCAL = 10·0 \times |F_c|$$

Reflections too weak to be observed were given zero weight in the refinement and were omitted in the R-index calculation. The unobserved reflections are given half the minimum value in that range.

H	K	L	FOBS	FCAL
0	0	1	371	441
0	0	2	57	3
0	0	3	413	466
0	0	4	146	175
0	0	5	33	51
0	0	6	104	101
0	0	7	54	57
0	1	1	[illegible]	232
0	1	2	112	110
0	1	3	48	51
0	1	4	204	211
0	1	5	73	77
0	1	7	76	72
0	1	8	121	134
0	1	10	26	19
0	2	0	199	226
0	2	1	259	285
0	2	2	156	150
0	2	3	216	206
0	2	4	265	286
0	2	5	146	149
0	2	6	83	86
0	2	7	118	126
0	2	8	47	45
0	2	9	71	69
0	2	10	21	21
0	3	1	109	116
0	3	2	114	116
0	3	3	37	46
0	3	4	149	157
0	3	5	80	84
0	3	6	48	46
0	3	7	22	19
0	3	8	34	31
0	3	9	42	41
0	4	0	65	75
0	4	1	35	22
0	4	2	157	163
0	4	3	174	172
0	4	4	132	148
0	4	5	73	72
0	4	6	98	103
0	4	7	67	72
0	5	1	48	49
0	5	2	50	43
0	5	3	53	52
0	5	4	22	21
0	5	6	66	62
0	6	0	57	69
0	6	1	92	98
0	6	2	118	123
0	6	3	20	17
0	6	4	68	68
0	6	5	66	71
0	7	1	30	33
1	0	0	67	12
1	0	-1	61	27
1	0	1	217	217
1	0	-2	260	157
1	0	2	170	106
1	0	-3	21	9
1	0	3	131	122
1	0	-4	71	76
1	0	4	148	166
1	0	-5	130	145
1	0	5	17	26
1	0	-6	143	136
1	0	6	105	110
1	0	-7	111	132
1	0	7	92	110
1	0	-8	101	116
1	0	8	99	105
1	0	-9	115	124
1	0	9	101	96
1	0	-10	49	47
1	1	0	286	296
1	1	-1	420	421
1	1	1	505	562
1	1	-2	290	313
1	1	2	195	175
1	1	-3	318	313
1	1	3	277	250
1	1	-4	97	129
1	1	4	39	38
1	1	-5	96	54
1	1	5	101	111
1	1	-6	87	87
1	1	6	116	112
1	1	-7	22	18
1	1	7	98	104
1	1	-8	91	94
1	1	8	47	41
1	1	-9	27	27
1	1	-10	26	19
1	1	10	34	36
1	2	0	90	138
1	2	-1	367	338
1	2	1	212	196
1	2	-2	280	282
1	2	2	272	257
1	2	-3	68	96
1	2	3	115	110
1	2	-4	197	177
1	2	4	69	63
1	2	-5	119	136
1	2	5	103	117
1	2	-6	70	62
1	2	6	115	113
1	2	-7	108	114
1	2	7	41	39
1	2	-8	54	56
1	2	8	26	22
1	2	9	59	54
1	2	-10	45	55
1	3	0	46	47
1	3	-1	113	108
1	3	1	167	164
1	3	-2	17	20
1	3	2	131	113
1	3	-3	76	76
1	3	3	133	145
1	3	4	21	10
1	3	-4	218	228
1	3	-5	103	100
1	3	5	125	124
1	3	-6	76	89
1	3	6	31	35
1	3	-7	63	57
1	3	7	67	62
1	3	-8	83	91
1	3	8	38	39
1	3	-9	29	33
1	3	9	21	21
1	4	0	143	150
1	4	-1	79	66
1	4	1	30	11
1	4	-2	70	53
1	4	2	112	111
1	4	-3	262	288
1	4	3	64	63
1	4	-4	49	43
1	4	4	68	82
1	4	-5	46	54
1	4	5	92	94
1	4	-6	121	127
1	4	6	35	33
1	4	-7	38	36
1	4	7	57	56
1	4	-8	25	22
1	5	0	107	123
1	5	-1	172	176
1	5	1	97	103
1	5	-2	65	54
1	5	2	31	41
1	5	-3	85	85
1	5	3	56	69
1	5	-4	49	56
1	5	4	45	47
1	5	5	160	82
1	5	-6	32	39
1	5	6	41	37
1	5	7	40	41
1	6	0	54	47
1	6	-1	27	19
1	6	1	38	43
1	6	2	39	36
1	6	-3	62	52
1	6	3	35	32
1	6	-4	44	42
1	6	4	21	22
1	6	-5	21	24
1	6	5	21	16
1	7	1	55	74
2	0	0	61	45
2	0	-1	872	912
2	0	1	275	285
2	0	-2	571	547
2	0	2	443	508
2	0	-3	235	230
2	0	3	131	119
2	0	-4	140	113
2	0	4	154	189
2	0	-5	17	30
2	0	5	121	107
2	0	-6	51	49
2	0	6	53	53
2	0	-7	25	14
2	0	7	31	36
2	0	-8	51	50
2	0	-9	42	45
2	1	0	271	283
2	1	-1	259	213
2	1	1	202	216
2	1	-2	300	284
2	1	2	102	94
2	1	-3	251	207
2	1	3	176	181
2	1	-4	157	156
2	1	4	129	139
2	1	-5	94	94
2	1	5	52	48
2	1	-6	113	124
2	1	6	127	119
2	1	-7	68	75
2	1	7	112	110
2	1	-8	28	29
2	1	8	46	38
2	1	-9	61	69
2	2	0	45	302
2	2	-1	146	170
2	2	1	254	248
2	2	-2	323	279
2	2	2	370	395
2	2	-3	111	103
2	2	3	294	290
2	2	-4	167	161
2	2	4	128	113
2	2	-5	193	200
2	2	5	60	61
2	2	-6	100	95
2	2	6	182	186
2	2	-7	125	127
2	2	7	53	47
2	2	-8	47	56
2	2	8	64	80
2	2	-9	44	50
2	2	9	50	45
2	2	-10	33	38
2	3	0	154	167
2	3	-1	64	93
2	3	1	84	94
2	3	-2	161	152
2	3	2	46	49
2	3	-3	95	76
2	3	3	106	115
2	3	-4	130	130
2	3	4	100	100
2	3	-5	116	121
2	3	5	99	97
2	3	-6	83	85
2	3	-7	59	57
2	3	-8	27	31
2	3	8	51	45
2	3	-9	29	26
2	4	0	72	95
2	4	-1	39	30
2	4	1	105	120
2	4	-2	90	80
2	4	2	71	70
2	4	-3	134	126
2	4	3	78	82
2	4	-4	157	176
2	4	4	124	127
2	4	-5	48	47
2	4	5	96	107
2	4	6	65	89
2	4	-7	77	69
2	4	7	39	31
2	4	-8	36	43
2	4	8	45	61
2	4	-9	26	33
2	5	-1	28	34
2	5	-2	98	84
2	5	2	45	53
2	5	-3	62	54
2	5	-4	60	61
2	5	4	23	19
2	5	-5	53	57
2	5	5	77	68
2	5	-6	75	65
2	6	0	64	85
2	6	-1	76	73
2	6	1	62	66
2	6	-2	36	33
2	6	2	40	53
2	6	-3	103	89
2	6	3	67	64
2	6	-4	25	19
2	6	4	69	64
2	6	-5	40	37
3	0	0	255	198
3	0	1	406	454
3	0	-2	172	175
3	0	2	415	357
3	0	-3	250	227
3	0	3	134	110
3	1	4	48	47
3	1	-5	153	134
3	0	-4	124	99
3	0	4	214	219
3	0	-5	109	118
3	0	5	159	158
3	0	-6	166	170
3	0	6	101	97
3	0	-7	158	212
3	0	-8	46	50
3	0	8	55	90
3	0	-9	42	47
3	0	-10	113	120
3	1	0	82	106
3	1	-1	35	11
3	1	1	116	118
3	1	-2	234	240
3	1	2	266	286
3	1	-3	27	26
3	1	3	103	122
3	1	-4	138	146
3	1	5	190	198
3	1	-6	151	137
3	1	6	104	100
3	1	-7	66	69
3	1	7	83	73
3	1	-8	28	19
3	1	8	31	22
3	1	-9	46	45
3	1	9	96	52
3	2	0	276	278
3	2	-1	271	278
3	2	1	243	284
3	2	2	116	137
3	2	-3	366	339
3	2	3	170	177
3	2	-4	93	99
3	2	4	37	41
3	2	-5	153	174
3	2	5	122	123
3	2	-6	148	146
3	2	-7	38	40
3	2	7	26	22
3	2	-8	99	100
3	2	8	32	25
3	2	-9	41	39
3	2	9	40	42
3	2	-10	60	60
3	3	0	167	182
3	3	-1	179	178
3	3	1	110	133
3	3	-2	103	119
3	3	2	46	61
3	3	-3	45	51
3	3	3	84	96
3	3	-4	106	98
3	3	4	104	97
3	3	-5	143	139
3	3	5	70	73
3	3	-6	100	103
3	3	6	41	32
3	3	-7	39	44
3	3	7	64	62
3	3	-8	44	44
3	3	8	21	17
3	3	-9	46	40
3	4	0	88	94
3	4	-1	202	203
3	4	1	102	119
3	4	-2	108	107
3	4	2	100	91
3	4	-3	119	122
3	4	-4	257	282
3	4	4	29	23
3	4	-5	113	107
3	4	5	37	38
3	4	-6	66	63
3	4	6	49	48
3	4	-7	52	50
3	4	-8	53	48
3	5	0	89	98
3	5	-1	54	48
3	5	1	29	47
3	5	-2	139	144
3	5	2	64	67
3	5	3	106	118
3	5	-4	39	32
3	5	4	53	63
3	5	-5	54	50
3	5	5	36	33
3	5	-6	55	48
3	5	6	15	17
3	5	-7	30	28
3	6	0	85	84
3	6	-1	87	82
3	6	1	40	37
3	6	-2	44	36
3	6	2	25	28
3	6	-3	43	41
3	6	3	67	73
3	6	-4	46	30
3	6	-5	77	67
4	0	0	249	204
4	0	-1	130	163
4	0	1	366	385
4	0	-2	283	282
4	0	2	120	118
4	0	-3	254	255
4	0	3	56	66
4	0	-4	161	167
4	0	4	200	203
4	0	-5	57	54
4	0	5	124	122
4	0	-6	35	34
4	0	6	34	34
4	0	-7	170	162
4	0	7	56	52
4	0	-10	35	35
4	1	0	67	86
4	1	-1	58	61
4	1	1	71	87
4	1	-2	190	155
4	1	2	241	265
4	1	-3	367	331
4	1	3	111	109
4	1	-4	198	166
4	1	4	28	19
4	1	-5	145	144
4	1	5	105	104
4	1	-6	25	25
4	1	6	115	105
4	1	-7	121	121
4	1	-8	80	84
4	1	8	29	17
4	1	-9	70	69
4	2	0	30	29
4	2	-1	227	225
4	2	1	155	149
4	2	-2	48	29
4	2	2	83	71
4	2	-3	66	66
4	2	3	39	44
4	2	-4	112	100
4	2	4	88	87
4	2	-5	124	112
4	2	5	77	90
4	2	-6	156	147
4	2	6	123	124
4	2	-7	40	39
4	2	7	56	53
4	2	-8	77	84
4	2	8	73	66
4	2	-9	30	30
4	2	-10	30	38
4	3	0	159	202
4	3	-1	103	105
4	3	1	84	92
4	3	-2	139	145
4	3	2	151	162
4	3	-3	241	227
4	3	3	33	23
4	3	-4	101	95
4	3	4	89	95
4	3	5	34	37
4	3	-5	49	35
4	3	-6	71	57
4	3	6	35	29
4	3	-7	112	118
4	3	7	32	33
4	3	-8	27	19
4	3	8	15	38
4	3	-9	50	47
4	3	-10	15	24
4	4	0	45	46
4	4	-1	50	42
4	4	1	116	131
4	4	-2	44	50
4	4	2	67	53
4	4	-3	83	76
4	4	3	91	99
4	4	-4	47	39
4	4	4	37	35
4	4	-5	96	90
4	4	5	87	84
4	4	-6	43	47
4	4	6	48	41
4	4	7	40	41
4	4	-8	53	49
4	4	-9	21	26
4	5	0	45	39
4	5	-1	84	93
4	5	-2	58	58
4	5	2	45	48
4	5	-3	58	52
4	5	3	33	33
4	5	4	48	60
4	5	4	72	60
4	5	-5	92	78
4	5	-6	50	41
4	5	-7	67	67
4	6	0	32	30
4	6	-1	57	63
4	6	1	44	45
4	6	-2	43	39
4	6	2	68	78
4	6	-3	29	32
4	6	3	30	41
4	6	-4	56	45
4	6	-5	26	23
5	0	0	220	207
5	0	1	250	237
5	0	-1	45	69
5	0	-2	35	61
5	0	2	73	74
5	0	-3	33	28
5	0	3	191	202
5	0	-4	142	164
5	0	4	80	75
5	0	-5	121	115
5	0	5	149	153
5	0	-6	176	175
5	0	-7	61	54
5	0	7	78	67
5	0	-8	168	177
5	0	8	45	38
5	0	-9	58	62
5	1	0	174	184
5	1	-1	227	210
5	1	1	105	117
5	1	-2	182	155
5	1	2	122	134
5	1	-3	201	196
5	1	3	51	49
5	1	-4	135	116
5	1	4	164	167
5	1	5	133	131
5	1	-5	107	93
5	1	-6	59	55
5	1	6	64	60
5	1	-7	33	23
5	1	7	104	88
5	1	-8	31	26
5	1	8	42	34
5	1	-9	30	29
5	1	-10	48	54
5	2	0	158	197
5	2	-1	274	278
5	2	1	80	96
5	2	-2	214	195
5	2	2	170	168
5	2	-3	254	214
5	2	3	176	195
5	2	-4	250	228
5	2	4	116	122
5	2	-5	53	48
5	2	5	68	54
5	2	-6	100	93
5	2	6	32	28
5	2	-7	88	93
5	2	7	52	48
5	2	8	37	34
5	2	-9	65	61
5	2	-10	39	42
5	3	0	47	51
5	3	-1	83	88
5	3	1	61	70
5	3	-2	90	82
5	3	2	49	107
5	3	-3	57	48
5	3	3	116	121
5	3	-4	49	39
5	3	4	34	44
5	3	-5	42	37
5	3	5	35	27
5	3	-6	74	67
5	3	6	66	59
5	3	-7	87	91
5	3	7	40	42
5	3	-9	29	30
5	4	0	38	44
5	4	-1	72	72
5	4	1	149	157
5	4	-2	246	234
5	4	2	40	47
5	4	-3	58	50
5	4	-4	89	101
5	4	4	55	68
5	4	-5	136	124
5	4	5	38	35
5	4	-6	135	123
5	4	-7	66	62
5	4	-8	36	35
5	5	0	61	69
5	5	-1	90	99
5	5	1	90	92
5	5	-2	86	78
5	5	2	143	126
5	5	-3	43	45
5	5	3	24	22
5	5	-4	42	31
5	5	4	29	33
5	5	-5	73	60
5	5	-7	45	43
5	6	-1	25	18
5	6	1	73	81
5	6	-2	75	70
5	6	2	54	67
5	6	-3	47	39
5	6	-4	37	27
6	0	0	21	33
6	0	-1	131	132
6	0	1	215	212
6	0	-2	275	263
6	0	2	79	83
6	0	-3	27	27
6	0	3	116	123
6	0	-4	35	55
6	0	4	40	35
6	0	-5	246	246
6	0	-6	68	58
6	0	-8	101	100
6	0	-9	42	45
6	0	-10	29	27
6	1	0	54	56
6	1	-1	209	216
6	1	1	55	54
6	1	-2	180	157
6	1	2	53	65
6	1	-3	85	66
6	1	3	102	113
6	1	-4	135	125
6	1	4	82	85
6	1	-5	262	251
6	1	5	67	67
6	1	-6	52	53
6	1	-7	83	84
6	1	7	29	25
6	1	-8	56	55
6	1	-9	44	45
6	1	-10	64	63
6	2	0	132	133
6	2	-1	91	80
6	2	1	111	109
6	2	-2	180	175
6	2	2	103	111
6	2	-3	57	42
6	2	3	100	95
6	2	-4	104	90
6	2	4	26	26
6	2	-5	112	101
6	2	5	63	57
6	2	-6	115	115
6	2	-7	88	84
6	2	7	69	68
6	2	-9	101	105
6	2	-10	37	42
6	3	0	71	60
6	3	-1	201	209
6	3	1	146	151
6	3	-2	53	47
6	3	2	100	114
6	3	-3	131	117
6	3	3	74	81
6	3	4	66	63
6	3	-4	35	23
6	3	-5	72	70
6	3	5	33	26
6	3	-6	62	59
6	3	6	36	29
6	3	-7	39	32
6	3	-8	39	42
6	4	0	76	69
6	4	-1	67	60
6	4	-2	97	110
6	4	2	91	97
6	4	-3	76	79
6	4	3	37	30
6	4	4	54	45
6	4	-5	66	61
6	4	5	42	49
6	4	-6	32	26
6	4	-7	37	27
6	4	-8	26	23
6	5	-1	54	52
6	5	1	56	57
6	5	-3	40	40
6	5	3	42	42
6	5	-4	38	29
6	5	4	55	55
6	5	-5	55	46
6	5	-6	47	41
6	5	-7	52	55
6	6	0	37	36
6	6	-1	33	23
6	6	1	39	40
6	6	-2	21	29
6	6	-3	50	54
6	6	-4	50	56
7	0	0	21	9
7	0	-1	152	135
7	0	1	25	22
7	0	-2	100	97
7	0	2	170	172
7	0	-3	21	22
7	0	3	44	36
7	0	-4	107	106
7	0	4	102	102
7	0	-5	30	26
7	0	5	70	69
7	0	6	67	64
7	0	-7	113	110
7	0	7	64	54
7	0	-8	125	128
7	0	-9	72	76
7	0	-10	29	30
7	1	0	60	61
7	1	-1	142	152
7	1	1	90	95
7	1	-2	99	86
7	1	2	26	12
7	1	-3	66	45
7	1	3	116	108
7	1	-4	184	175
7	1	4	75	66
7	1	-5	53	52
7	1	5	23	23
7	1	-6	64	67
7	1	6	51	74
7	1	-7	40	69
7	1	7	30	28
7	1	-8	52	56
7	1	-10	30	25
7	2	0	164	168
7	2	-1	35	27
7	2	1	72	70
7	2	-2	121	135
7	2	2	129	131
7	2	-3	37	38
7	2	3	34	36
7	2	-4	76	79
7	2	4	75	72
7	2	-5	135	142
7	2	5	60	51
7	2	-6	48	37
7	2	6	57	52
7	2	-8	46	51
7	2	-9	56	53
7	2	-10	26	26
7	3	0	126	131
7	3	-1	66	76
7	3	1	152	151
7	3	-2	109	110
7	3	2	97	97
7	3	-3	90	90
7	3	3	34	46
7	3	4	57	48
7	3	5	51	50
7	3	-5	42	46
7	3	-6	61	66
7	3	6	21	23
7	3	-8	75	70
7	4	0	119	124
7	4	-1	22	20
7	4	-2	66	70
7	4	2	46	47
7	4	-3	121	116
7	4	-4	109	115
7	4	-5	62	58
7	4	5	37	42
7	4	-7	71	63
7	5	0	37	33
7	5	-1	39	42
7	5	1	69	69
7	5	3	30	35
7	5	-4	35	28
7	5	-5	36	32
7	5	-6	70	71
7	6	0	65	72
7	6	-1	40	46
7	6	-3	37	35
8	0	0	173	182
8	0	-1	51	50
8	0	1	25	21
8	0	-2	55	55
8	0	2	61	82
8	0	-3	176	174
8	0	3	50	46
8	0	-4	63	68
8	0	-6	243	245
8	0	-7	150	160
8	0	-8	32	32
8	0	-9	37	47
8	0	-10	69	75
8	1	0	58	60
8	1	-1	125	111
8	1	1	153	166
8	1	-2	173	173
8	1	2	70	78
8	1	-3	141	136
8	1	3	45	43
8	1	-4	67	63
8	1	4	37	37
8	1	-5	67	68
8	1	5	79	74
8	1	-6	107	107
8	1	-7	39	39
8	1	-8	48	49
8	1	-9	40	40
8	2	-1	159	155
8	2	1	111	112
8	2	-2	97	93
8	2	2	42	38
8	2	-3	120	122
8	2	-4	49	48
8	2	4	56	47
8	2	-5	90	85
8	2	5	29	23
8	2	-6	31	28
8	2	6	21	25
8	2	-7	55	54
8	2	-8	33	30
8	2	-9	33	27
8	2	-10	30	39
8	3	0	48	48
8	3	-1	42	40
8	3	1	104	112
8	3	-2	119	115
8	3	-3	68	75
8	3	3	33	52
8	3	-4	57	51
8	3	4	43	46
8	3	-5	34	37
8	3	5	43	45
8	3	-6	55	56
8	3	-6	22	26
8	4	0	27	31
8	4	-1	51	47
8	4	1	55	64
8	4	-2	34	34
8	4	2	60	55
8	4	-3	74	80
8	4	3	51	51
8	4	-4	53	57
8	4	4	26	30
8	4	-5	54	52
8	4	-7	36	33
8	4	-8	21	26
8	5	0	68	69
8	5	-1	67	60
8	5	1	26	18
8	5	2	26	31
8	5	-4	49	50
8	5	-5	26	29
9	0	0	28	24
9	0	-1	52	51
9	0	1	63	71
9	0	-2	40	31
9	0	2	56	53
9	0	-3	64	81
9	0	3	62	59
9	0	-4	28	30
9	0	4	50	50
9	0	-7	50	53
9	0	-8	35	30
9	0	-9	43	42
9	0	-10	26	26
9	1	0	159	154
9	1	-1	38	39
9	1	1	34	30
9	1	-2	63	59
9	1	2	61	64
9	1	-3	131	127
9	1	3	51	50
9	1	-4	104	101
9	1	-5	93	98
9	1	5	54	48
9	1	-6	43	46
9	1	-7	55	56
9	1	-8	33	30
9	1	-9	66	61
9	2	0	63	67
9	2	-1	96	99
9	2	1	48	45
9	2	-2	84	79
9	2	2	67	64
9	2	3	27	20
9	2	-4	56	57
9	2	4	103	96
9	2	-5	89	90
9	2	5	30	30
9	2	-7	46	46
9	2	-8	55	63
9	3	0	61	58
9	3	-1	65	60
9	3	-2	50	50
9	3	2	31	31
9	3	3	38	26
9	3	-4	108	118
9	3	4	43	43
9	3	-5	58	51
9	3	-7	28	29
9	3	-8	30	28
9	4	0	51	50
9	4	-1	50	47
9	4	1	38	32
9	4	2	26	29
9	4	-3	82	89
9	4	-5	72	69
9	4	-6	29	29
9	4	-7	34	29
9	5	0	52	53
9	5	-1	30	29
9	5	-2	33	35
9	5	-4	26	24
9	5	-5	30	39
10	0	-1	94	97
10	0	1	78	82
10	0	-2	54	50
10	0	-3	127	133
10	0	-4	31	28
10	0	4	21	20
10	0	-5	29	23
10	0	-6	57	61
10	0	-7	40	44
10	1	0	127	131
10	1	1	34	33
10	1	-2	28	26
10	1	-3	68	67
10	1	3	45	47
10	1	-4	90	59
10	1	4	50	44
10	1	-6	29	27
10	1	-7	40	49
10	1	-9	37	39
10	2	0	29	54
10	2	-1	37	38
10	2	1	70	67
10	2	2	28	25
10	2	-3	64	63
10	2	3	42	38
10	2	-4	126	127
10	2	4	26	27
10	2	-5	109	107
10	2	-8	63	65
10	3	0	50	55
10	3	-1	38	40
10	3	1	59	64
10	3	-2	55	50
10	3	2	65	65
10	3	-3	30	32
10	3	3	61	70
10	3	-4	40	41
10	3	-5	52	42
10	3	-6	54	60
10	3	-7	49	51
10	4	0	25	29
10	4	-1	33	29
10	4	1	61	59
10	4	-2	25	20
10	4	-3	38	41
10	4	-4	25	26
10	4	-6	43	42
11	0	-1	41	38
11	0	1	63	58
11	0	-2	105	105
11	0	-3	71	74
11	0	3	30	28
11	0	-6	50	49
11	0	-7	53	55
11	0	-9	21	28
11	1	0	78	71
11	1	-1	100	99
11	1	1	34	30
11	1	-2	82	87
11	1	2	67	65
11	1	-3	65	58
11	1	-4	32	32
11	1	-5	76	76
11	1	-7	52	50
11	1	-8	36	41
11	2	-1	54	58
11	2	1	50	44
11	2	-2	27	19
11	2	2	104	42
11	2	-3	30	31
11	2	-4	35	43
11	2	-6	35	32
11	2	-7	47	46
11	2	-8	53	61
11	3	-1	32	30
11	3	-3	50	47
11	3	-4	32	29
11	3	-5	64	67
11	3	-6	45	48
11	3	-7	59	66
11	4	-1	34	35
11	4	-2	21	18
11	4	-3	21	22
11	4	-4	48	48
12	0	0	25	18
12	0	1	45	42
12	0	-2	98	99
12	0	-3	30	39
12	0	-4	46	45
12	0	-6	43	42
12	0	-7	25	30
12	1	0	29	28
12	1	-1	59	56
12	1	1	21	18
12	1	-2	42	45
12	1	2	21	23
12	1	-4	34	36
12	1	-5	47	50
12	1	-7	30	27
12	1	-8	21	30
12	2	0	78	75
12	2	-1	53	50
12	2	1	21	19
12	2	-2	25	21
12	2	-4	60	62
12	2	-6	[illegible]	66
12	3	-1	21	21
12	3	-2	21	18
12	3	-3	33	37
12	3	-4	21	26
12	3	-5	21	21
13	0	-1	33	35
13	0	-2	30	22
13	0	-3	33	30
13	0	-4	29	23
13	0	-6	26	30
13	1	0	30	31
13	1	-1	56	52
13	1	-2	42	40
13	1	-3	63	71
13	1	-4	26	26
13	1	-5	30	22
13	1	-6	34	38
13	2	-1	21	22
13	2	-2	15	22
13	2	-3	40	45
13	2	-4	30	35

Unobserved Reflections

H	K	L	FOBS	FCAL
0	0	8	23	20
0	0	9	21	14
0	0	10	16	11
0	1	6	21	22
0	1	9	21	25
0	4	8	16	20
0	5	5	22	23
0	5	7	15	20
1	0	10	14	15
1	1	9	19	22
1	2	-9	20	21
1	4	8	15	19
1	5	-5	22	27
1	5	-7	16	21
1	6	-2	21	26
1	7	0	13	19
1	7	-1	13	25
2	0	8	22	11
2	0	9	19	12
2	0	-10	19	17
2	1	9	18	29
2	1	-10	18	12
2	3	6	23	26
2	3	7	22	18
2	4	-6	23	29
2	5	0	23	25
2	5	1	23	25
2	5	3	23	20
2	5	6	16	29
2	5	-7	16	18
3	0	7	23	17
3	0	9	16	10
3	1	-10	19	20
3	2	6	23	22
3	4	3	23	36
3	4	7	16	17
3	4	-9	13	11
3	5	-3	23	24
3	6	4	14	3
4	0	-8	23	19
4	0	8	19	29
4	0	9	13	11
4	0	-9	22	4
4	1	7	22	25
4	1	-10	19	24
4	4	-7	21	17
4	5	-4	22	9
4	5	5	16	20
5	0	6	23	7
5	0	-10	19	19
5	2	-8	23	20
5	3	-8	22	13
5	4	3	23	16
5	4	6	16	18
5	4	-9	13	10
5	5	5	13	21
5	5	-6	19	11
5	6	0	18	11
5	6	-5	14	13
6	0	5	23	28
6	0	6	22	26
6	0	-7	23	23
6	0	7	19	3
6	1	6	22	43
6	2	6	20	19
6	2	-8	23	18
6	3	-9	17	12
6	4	1	23	19
6	4	-4	23	29
6	5	0	22	18
6	5	2	20	30
6	5	-2	22	21
7	0	-6	22	13
7	1	-9	21	8
7	2	-7	23	6
7	3	-4	23	28
7	3	-7	22	23
7	3	-9	16	4
7	4	-1	23	29
7	4	1	23	32
7	4	3	20	27
7	4	4	18	20
7	4	-6	21	21
7	4	-8	16	8
7	5	-2	21	25
7	5	2	17	5
7	5	-3	20	24
7	6	-2	14	15
8	0	4	22	24
8	0	5	20	18
8	0	-5	22	22
8	0	6	16	17
8	1	6	16	25
8	1	-10	16	28
8	2	0	23	14
8	2	3	23	19
8	3	2	22	24
8	3	-7	21	21
8	3	-9	15	12
8	4	-6	20	22
8	5	-2	19	20
8	5	-3	19	19
8	5	-6	14	9
9	0	-5	23	25
9	0	5	17	11
9	0	-6	23	20
9	1	4	20	13
9	2	-3	23	21
9	2	-6	23	17
9	2	-9	16	18
9	3	1	22	19
9	3	-3	23	22
9	3	-6	22	26
9	4	-2	21	16
9	4	3	13	14
9	4	-4	20	32
9	5	-3	16	18
10	0	0	23	20
10	0	2	22	22
10	0	3	20	5
10	0	-8	20	44
10	0	-9	16	29
10	1	-1	23	35
10	1	2	22	16
10	1	-5	23	18
10	1	-8	19	17
10	2	-2	23	28
10	2	-6	22	25
10	2	-7	20	14
10	2	-9	13	19
10	3	-8	14	17
10	4	-5	17	16
11	0	0	22	32
11	0	2	19	26
11	0	-4	23	19
11	0	-5	22	30
11	0	-8	17	6
11	1	3	15	10
11	1	-6	21	11
11	2	0	20	22
11	2	-5	21	13
11	3	0	17	25
11	3	1	16	23
11	3	-2	19	16
11	4	-5	13	15
12	0	-1	20	3
12	0	2	14	4
12	0	-5	20	25
12	0	-8	14	22
12	1	-3	21	14
12	1	-6	18	17
12	2	-3	19	20
12	2	-5	18	20
12	2	-7	14	24
13	0	0	14	10
13	0	-5	16	11
13	2	-2	14	21
13	2	-5	13	16

5-fluoro-2-deoxy-β-uridine, Harris & Macintyre (1964); adenylic acid, Kraut & Jensen (1963); calcium thymidilate, Trueblood, Horn & Luzzati (1961); deoxyadenosine, Watson, Sutor & Tollin, quoted in Sundaralingam (1965); sucrose, Brown & Levy (1963)] where C(1)–O(1) is shorter than C(4)–O(1) all have the β configuration. It may be that the reversal of this tendency is due to the α configuration.

The C–C–O angles within the furanoside ring and outside it average respectively to 105·5° and 110·0°. The internal C–C–C angles of the furanoside ring average to 101·9°. These internal angles are considerably smaller than the tetrahedral value of 109·5°. The ring C–O–C angle is 108·0°, in agreement with the results obtained in other furanoid structures (see, for a review, Jeffrey & Rosenstein, 1964).

As observed in other furanoid saccharides, the furanose ring in ETGF is non-planar and this puckering is due to the nonbonded interactions of the substituent atoms and results in an approximately staggered configuration (Spencer, 1959). As a result, one of the carbon atoms is out of the plane containing the other

Table 4. *Description of thermal ellipsoids*

$B_i = 8\pi^2 U_i^2$ where U_i is the root-mean square displacement corresponding to the i axis of the ellipsoid. C_{ia}, C_{ib}, C_{ic}* are the direction cosines of the i axis with respect to the crystal axes a, b, c*.

Atom	Axis i	B_i	U_i	C_{ia}	C_{ib}	C_{ic}*
S	1	4·17	0·23	−0·7511	−0·1230	0·6486
	2	3·48	0·21	−0·5137	0·7259	−0·4573
	3	3·15	0·20	−0·4146	−0·6767	−0·6085
O(1)	1	4·17	0·23	0·7709	0·3679	−0·5199
	2	2·02	0·16	0·5869	−0·0931	0·8043
	3	1·54	0·14	0·2475	−0·9252	−0·2877
O(2)	1	5·33	0·26	−0·1359	0·1153	0·9840
	2	3·48	0·21	−0·0033	−0·9933	0·1157
	3	2·02	0·16	−0·9907	−0·0129	0·1353
O(3)	1	4·17	0·23	0·4523	−0·1540	0·8785
	2	2·85	0·19	0·7368	−0·4905	−0·4653
	3	1·77	0·15	0·5026	0·8577	−0·1084
O(4)	1	5·75	0·27	0·9970	0·0670	−0·0383
	2	2·85	0·19	0·0753	−0·7379	0·6707
	3	1·77	0·15	0·0168	−0·6716	−0·7408
O(5)	1	4·17	0·23	0·9985	−0·0151	−0·0523
	2	2·85	0·19	0·0530	0·4889	0·8707
	3	1·33	0·13	0·0124	−0·8722	0·4890
C(1)	1	3·48	0·21	0·5486	0·7784	0·3052
	2	2·28	0·17	0·4017	0·0747	0·9127
	3	2·02	0·16	0·7332	−0·6233	−0·2717
C(2)	1	3·48	0·21	−0·0925	0·0809	−0·9924
	2	2·28	0·17	−0·9689	−0·2370	0·0709
	3	1·77	0·15	−0·2295	0·9687	0·1003
C(3)	1	3·15	0·20	−0·4750	0·2112	−0·8542
	2	2·28	0·17	−0·7546	−0·5971	0·2720
	3	1·33	0·13	−0·4526	0·7739	0·4430
C(4)	1	2·55	0·18	−0·9715	0·1958	0·1334
	2	2·28	0·17	−0·2018	−0·3895	−0·8986
	3	1·13	0·12	−0·1240	−0·9000	0·4180
C(5)	1	3·48	0·21	0·9917	−0·0909	0·0904
	2	2·56	0·16	0·1213	0·4378	−0·8908
	3	1·55	0·14	0·0415	0·8944	0·4452
C(5)	1	3·48	0·21	0·6582	−0·0582	−0·7506
	2	2·56	0·18	0·7089	0·3833	0·5920
	3	1·78	0·15	0·2532	−0·9218	0·2935
C(7)	1	7·10	0·30	0·8212	0·5563	−0·1271
	2	4·54	0·24	0·1917	−0·0592	0·9797
	3	2·85	0·19	0·5375	−0·8288	−0·1553
C(8)	1	10·80	0·37	−0·3776	0·7991	−0·4678
	2	5·33	0·26	−0·7174	−0·5719	−0·3978
	3	3·16	0·20	−0·5855	0·1854	0·7892

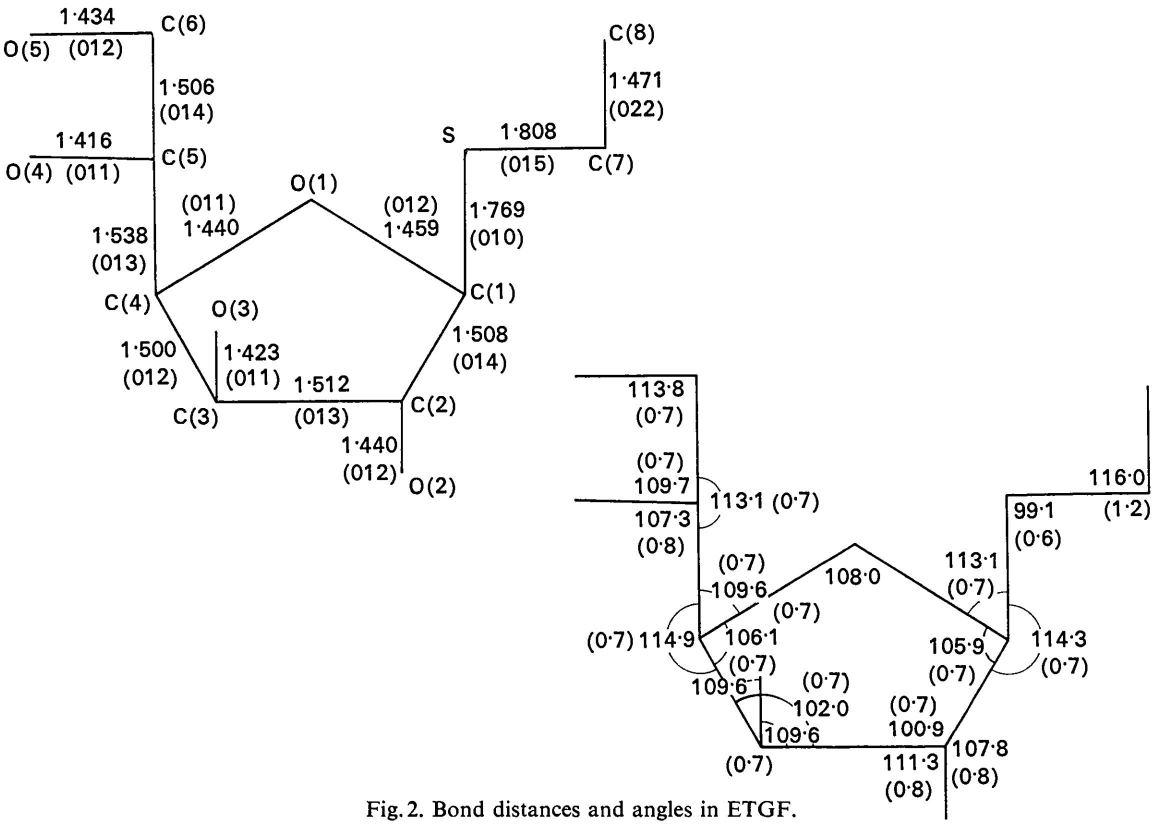

Fig. 2. Bond distances and angles in ETGF.

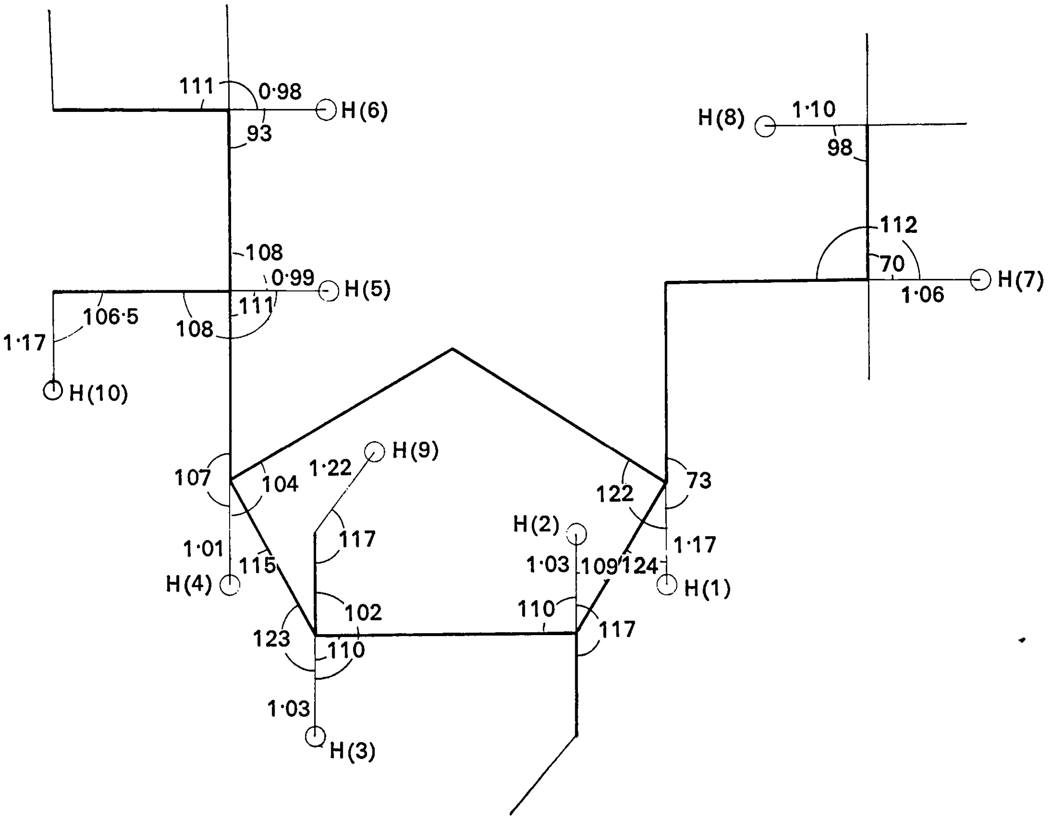

Fig. 3. Bond distances and angles involving hydrogen atoms in ETGF. The hydrogen atoms whose coordinates were derived from the difference electron density map are shown by open circles.

three carbon atoms and the ring-oxygen atom; the choice in other structures for the out of plane atom seems to be either C(2) or C(3). In ETGF, C(3) is 0·589 Å from the plane $0{\cdot}7491X+0{\cdot}6209Y-0{\cdot}2309Z=1{\cdot}678$ passing through the atoms O(1), C(1), C(2) and C(4); the coefficients in the above equation are the direction cosines of the normal to the plane relative to **a**, **b** and **c***. The deviations of the individual atoms from this plane are: −0·077, 0·074, −0·045 and 0·048 Å for O(1), C(1), C(2) and C(4) respectively. In addition, the atoms O(1), C(1), C(3) and C(4) are closely coplanar and lie at distances of 0·071, −0·044, 0·042, −0·070 Å respectively from the plane $0{\cdot}9025X+0{\cdot}3501Y-0{\cdot}2506Z=1{\cdot}226$. In this case, the major displacement occurs for C(2), which is −0·595 Å out of this plane. The description of the planarity of the ring in terms of either one of these planes seems to be equally satisfactory. These two planes are inclined to one another at 17·95°. This is also clear from the fact that C(2) and C(3) are at distances of −0·341 and 0·318 Å respectively from the plane $0{\cdot}8413X+0{\cdot}4876Y-0{\cdot}2334Z=1{\cdot}511$ containing C(1), O(1) and C(4).

In view of the puckering observed in the furanosides, the hydroxyls on C(2) and C(3) cannot exhibit a 'true' *cis* or 'true' *trans* relation. In other words, the projected angles of the hydroxyls when viewed along C(2)–C(3) can no longer be 0° or 120°. The actual value of this projected angle is 171°, close to the value of 160°, which has been suggested by Sundaralingam (1965).

The conformation of the furanose ring may be more conveniently described in terms of an angle of twist about each ring bond (Brown & Levy, 1963). These angles may be defined as follows: Consider the four successive atoms C(1), C(2), C(3) and C(4) around the ring. Then the conformation angle of the directed bond C(3)→C(2) is the angle, measured anticlockwise, that the projection of C(3)→C(4) makes relative to the projection of bond C(2)→C(1) when viewed in the direction of the bond C(3)→C(2). In ETGF, the conformation angles for the various bonds are: C(3)→C(2), 40·6°; C(4)→C(3), −33·8°; O(1)→C(4), 12·8°; C(1)→O(1), 13·6°; C(2)→C(1), −33·9°. The conformation of the furanose ring in ETGF is closely similar to that found in sucrose and adenylic acid (AMP) and

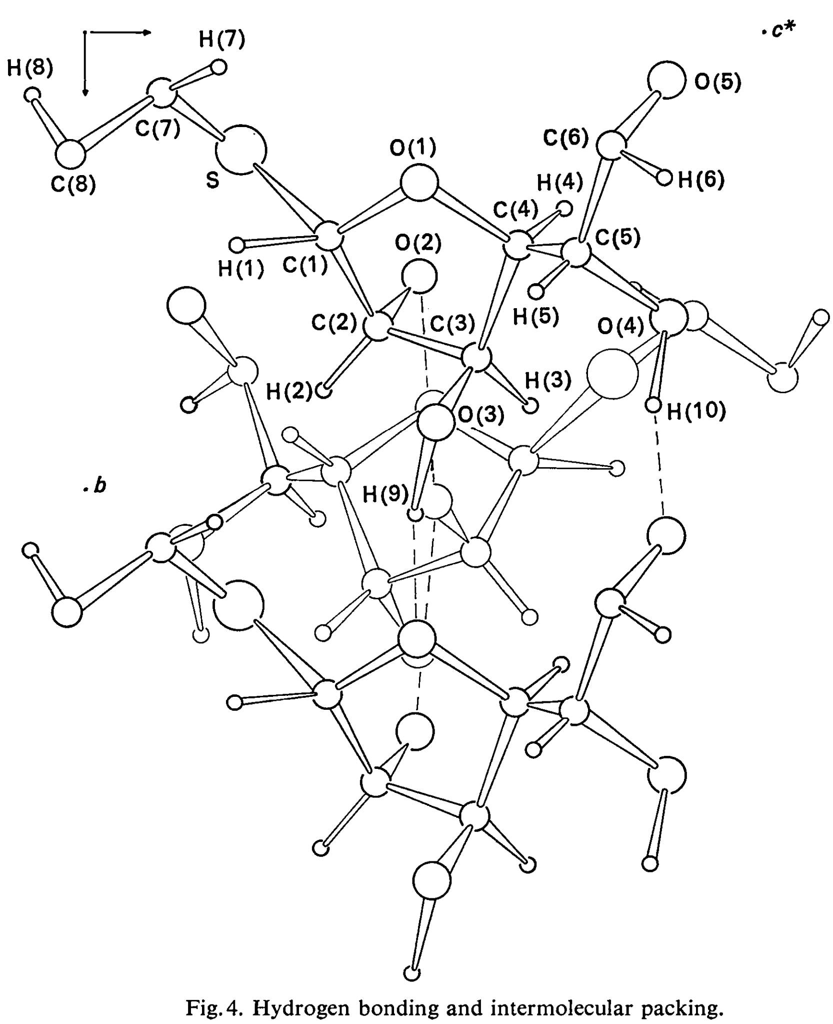

Fig. 4. Hydrogen bonding and intermolecular packing.

significantly different from that found in cytidylic acid (CMP), 5-fluoro-deoxy-β-uridine (FUDR), calcium thymidylate (TMP) and deoxyadenosine (DA). (For the conformation angles of these compounds, see Table 6 of Sundaralingam (1965). The abbreviations AMP *etc.* are those used in this reference).

The bond opposite the out-of-plane atom has the smallest value for the conformation angle φ. Thus, in CMP, FUDR and sucrose, the smallest conformation angle is around O(1)–C(4) whereas for AMP, TMP and DA, C(1)–O(1) has the smallest conformational angle. In ETGF, however, both O(1)–C(4) and C(1)–O(1) have nearly the same conformational angle.

In nucleic acid components, the preferred conformation about the C(4)–C(5) bond (of the furanose) is *gauche–gauche* (*gg*). This conformation can be more conveniently described in terms of the angles φ_{OO} and φ_{OC} which in the case of ETGF are the projected angles made by C(5)–C(6) [C(5)–O(5) for a nucleic acid component] with O(1)–C(4) and C(3)–C(4). (See Shefter & Trueblood (1965) for a definition of the angles $\varphi_{OO}, \varphi_{OC}$. The primes on the numerals in the conventional numbering of the atoms in the sugar have been dropped here for easy comparison with ETGF). The values for φ_{OO} and φ_{OC} are 58·2° and 177·6°, indicating that the conformation about C(4)–C(5) is *gauche–trans* (*gt*). It is interesting to note that α-D-glucose has the same conformation.

Hydrogen bonding and intermolecular packing

The distances and angles involving the covalently bound hydrogen atoms are shown in Figs. 2 and 3. The average distances for CH and OH are 1·05 ± 0·10 Å and 1·20 ± 0·10 Å respectively.

Three pairs of atoms have separations which are of the right order for hydrogen bond formation: O(1)–O(3′), 2·767 Å; O(4)–O(5′), 2·718 Å; O(2)–O(2″), 2·99 Å. They are shown in Fig. 4 and the corresponding distances and angles are listed in Table 5. The ring oxygen O(1) accepts the hydrogen H(9′) from the hydroxyl group O(3′) and this results in an almost linear hydrogen bond (O(3′)–H(9′)---O(1) = 165·0). The hydrogen bond between O(4) and O(5′) through H(10) is not linear, the angle O(4)–H(10)–O(5′) being 138·2°. Such hydrogen bonds have been observed in other structures (Baur, 1965). Another hydrogen bond, between O(2) and O(2″), is rather weak, (O(2″)---O(2), 2·999 Å). In addition, there is an interesting short contact (3·139 Å) between O(3) and O(5′). Such a separation of 3·2 Å between two oxygen atoms which could form a shorter hydrogen bond has been observed in other sugars (Beevers, McDonald, Robertson & Stern, 1952, Ferrier, 1960). Most of the hydrogen bonds are nearly parallel to the *b* axis, consistent with the form of the crystal as needles elongated along [010]. All intermolecular short contacts less than 3·6 Å between heavy atoms and 3·0 Å between a hydrogen and a heavy atom are listed in Table 6.

Table 6. *Intermolecular contacts*

Coordinates: (x,y,z) unprimed, $(x,y,z)+(0,1,0)$ single prime;
$(\bar{x},\frac{1}{2}+y,\bar{z})+(0,\bar{1},1)$ double prime;
$(\bar{x},\frac{1}{2}+y,\bar{z})+(1,0,1)$ triple prime.

O(2)	C(2″)	3·182 Å
O(2)	C(3″)	3·497
O(3)	O(5′)	3·139
O(3)	C(6′)	3·325
O(3)	C(6‴)	3·415
C(3)	O(5′)	3·503

S′	H(2)	2·881 Å
O(2)	H(2″)	2·912
O(2)	H(3″)	2·872
O(3)	H(7′)	2·933
O(3)	H(5‴)	2·942
C(1′)	H(9)	2·618
C(4′)	H(9)	2·680
C(6′)	H(10)	2·502

We are grateful to Dr David Harker for many interesting discussions and his keen interest, to Dr I. J. Goldstein for the crystals and to Dr M. Sundaralingam for many discussions on the stereochemistry of furanosides. Computations were carried out on an IBM 7040 machine at the Roswell Park Memorial Institute or on an IBM 7044 machine at the Computing Center of the State University of New York at Buffalo. We are indebted to Dr D. R. Harris for making available the following programs: BDLSQ, ERFR3, Distance-Angle and Best Plane. Financial assistance by N.I.H. (AM-3942) and N.S.F. (GB-4056) is gratefully acknowledged.

Table 5. *Hydrogen bond lengths and angles with estimated standard deviations* (*in parentheses*)

The e.s.d.'s of distances and angles involving hydrogen atoms are less than 0·2 Å and 5° respectively.

Coordinates: x,y,z unprimed
$(x,y,z)+(0,1,0)$ single prime
$(\bar{x},\ \frac{1}{2}+y,\ \bar{z})+(0,\bar{1},1)$ double prime

I	
O(3)–H(9)---O(1′)	
O(1′)---H(9)	1·570 Å
O(3)---O(1′)	2·767 (010) Å
O(3)–H(9)---O(1′)	165·0°
C(3)–O(3)---O(1′)	117·9 (5)°

II	
O(4)–H(10)---O(5′)	
O(5′)---H(10)	1·734 Å
O(5′)---O(4)	2·718 (011) Å
O(4)–H(10)---O(5′)	138·2°
C(5)–O(4)---O(5′)	123·5 (6)°

III	
O(2)---O(2″)	
O(2″)---O(2)	2·999 (013) Å
C(2″)–O(2″)---O(2)	134·4 (6)°

References

BAUR, E. H. (1965). *Acta Cryst.* **19**, 909.

BEEVERS, C. A., MCDONALD, R., ROBERTSON, J. H. & STERN, F. (1952). *Acta Cryst.* **5**, 689.

BROWN, G. M. & LEVY, H. A. (1965). *Science,* **141**, 921.

FERRIER, G. (1960). *Acta Cryst.* **13**, 678.

FERRIER, G. (1963). *Acta Cryst.* **16**, 1023.

FURNAS, T. F. & HARKER, D. (1955). *Rev. Sci. Instrum.* **26**, 449.

HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502.

HARRIS, D. R. & MACINTYRE, W. M. (1964). *Biophys. J.* p. 203.

IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 789.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

JEFFREY, G. A. & ROSENSTEIN, R. D. (1964). *Advanc. Carbohydrate Chem.* **19**, 7.

KRAUT, J. & JENSEN, L. H. (1963). *Acta Cryst.* **16**, 79.

SHEFTER, E. & TRUEBLOOD, K. N. (1965). *Acta Cryst.* **18**, 1067.

SPENCER, M. (1959). *Acta Cryst.* **12**, 59.

SUNDARALINGAM, M. (1965). *J. Amer. Chem. Soc.* **87**, 599.

SUNDARALINGAM, M. & JENSEN, L. H. (1965). **13**, 914, 930.

TRUEBLOOD, K. N., HORN, P. & LUZZATI, V. (1961). *Acta Cryst.* **14**, 965.

Acta Cryst. (1967). **23**, 1057

The Crystal Structures of 9-Dicyanomethylenefluorene Derivatives. I. 9-Dicyanomethylene-2,4,7-trinitrofluorene

BY J. SILVERMAN, A. PAULINE KRUKONIS AND N. F. YANNONI

U.S. Air Force Cambridge Research Laboratories, L. G. Hanscom Field, Bedford, Massachusetts 01730, *U.S.A.*

(*Received* 3 *January* 1967)

The crystal structure of 9-dicyanomethylene-2,4,7-trinitrofluorene is orthorhombic (space group *Pbcn*) with four molecules in a unit cell of dimensions $a = 13{\cdot}12$, $b = 11{\cdot}03$, $c = 10{\cdot}40$ Å. Molecular centers lie on twofold special positions 4(*c*) so that the unpaired nitro group occurs randomly at one of two possible locations. 748 reflections observed on X-ray photographs were used to refine the structure by the method of least squares to an *R* index of 12·5 %. A slight twist of the fluorene framework into a propeller shape leaves outer rings out of parallelism by 3·2°. The unpaired nitro group is rotated out of the molecular plane by 19·3°. The force tending to maximize the resonance energy of this group is believed responsible for: (1) the intramolecular overcrowding of the observed configuration, (2) the slight deviation from planarity of the fluorene framework, and (3) the angular distortion in C–C–N bond angles. There is a herringbone packing arrangement with the overall molecular plane deviating by 0·5° from the intensely reflecting (302) plane. Limiting intermolecular contacts occur largely through the nitro groups.

Introduction

9-Dicyanomethylene-2,4,7-trinitrofluorene (DTF) is an electron acceptor synthesized recently by Mukherjee & Levasseur (1965). This compound forms stronger charge-transfer complexes than its precursor, 2,4,7-trinitrofluorenone. The crystallography of DTF and related nitrofluorenes form part of a program underway in our laboratories on the synthesis and structural characterization of new organic semiconductors and photoconductors (Mukherjee, 1966).

Experimental

The specimen selected for data collection was a well-developed prismatic plate with a long dimension of 0·8 mm (mounted coaxially in a glass capillary), an average width of 0·33 mm, and a thickness of 0·12 mm.

Crystal data

9-Dicyanomethylene-2,4,7-trinitrofluorene (DTF), $C_{16}H_5N_5O_6$; m.p. 266–268 °C.

Orthorhombic, $a = 13{\cdot}12$, $b = 11{\cdot}03$, $c = 10{\cdot}40$ [radiation Mo $K\alpha$, $\lambda = 0{\cdot}7017$ Å. These parameters, derived from measurements made on precession photographs (without film shrinkage corrections), are reproducible to $\pm 0{\cdot}1\%$ but are assigned e.s.d.'s of 0·3% on the basis of previous experience (Buerger, 1964)].

Absent reflections: $0kl$ when k is odd; $h0l$ when l is odd; $hk0$ when $h+k$ is odd. Space group is *Pbcn* (no. 60).

Absorption coefficients for X-rays: Cu $K\alpha$, 12·67 cm^{-1}; Mo $K\alpha$, 1·57 cm^{-1}.

Density data: D_x $(Z=4) = 1{\cdot}602$ g.cm^{-3}; D_m (flotation in $ZnCl_2$–H_2O solution) $= 1{\cdot}58$ g.cm^{-3}.

Levels $hk0$–$hk8$ were taken with unfiltered copper radiation on an integrating Weissenberg camera used in the equi-inclination geometry. Levels were recorded on packets of five films, and the spots were measured photometrically with a densitometer. Faint spots were