

The Crystal and Molecular Structure of 1-Methyl-4-(β -D-erythrofuranosyl)-4-imidazoline-2-thione*

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The crystal structure of title compound, $C_8H_{13}N_2SO_3$, has been determined by single-crystal X-ray analysis from 1040 diffractometer data. The unit cell is monoclinic with $a = 10.674$ (6), $b = 10.349$ (5) and $c = 4.688$ (3) Å, $\beta = 102.00$ (4)°. The space group is $P2_1$ and there are two molecules in the unit cell. The structure has been solved by direct methods and refined by a full-matrix least-squares method to a final R value of 0.055. The glycosidic torsion angle defined for the sequence of atoms O(1)–C(5)–C(3)–C(4) is 44.2° . The sugar ring puckering is in the twist conformation 2T_3 . The molecules are linked together by hydrogen bonds one of which, involving the sugar O(1) atom, is unusually short.

Introduction

The crystal structure of 1-methyl-4-(β -D-erythrofuranosyl)-4-imidazoline-2-thione has been determined as part of a continuing investigation of the conformational aspects of glucimidazoles and imidazole C-nucleosides in this laboratory. This program has so far included 4-(β -D-erythrofuranosyl)imidazoline-2-thione (Pérez-Garrido, López-Castro & Márquez, 1973), 1-methyl-4,5-(D-glucofurano)imidazolidine-2-thione (Jiménez-Garay, López-Castro & Márquez, 1974) and 1-*p*-chlorophenyl-4-(α -D-erythrofuranosyl)-4-imidazoline-2-thione (Pérez-Garrido, Conde & Márquez, 1974).

The preparation and properties of the title compound have been reported (Fernández-Bolaños, Fuentes & Pradera de Fuentes, 1973). The formula was established by oxidation analysis and n.m.r. spectroscopy. Its molecular conformation, however, could not be uniquely assigned, and X-ray analysis was suggested by Professor J. Fernández-Bolaños.

Experimental

Single crystals of this compound were prepared and kindly supplied by Professor J. Fernández-Bolaños (Departamento de Química Orgánica of this University). The crystals are colourless prisms elongated along the c axis.

Preliminary rotation and Weissenberg photographs indicated monoclinic symmetry. The condition for reflexion was observed to be: $0k0$ $k = 2n$ and the possible space groups consistent with this observation are $P2_1$ and $P2_1/m$. The former was chosen on the basis of the E statistics.

The unit-cell parameters were determined by least-squares methods from 25 accurate 2θ values measured

on an automatic four-circle diffractometer. The crystal data are as follows: $a = 10.674$ (6), $b = 10.349$ (5), $c = 4.688$ (3) Å, $\beta = 102.00$ (4)°, $V = 506.52$ Å³, $Z = 2$, $D_m = 1.41$, $D_x = 1.41$ g cm⁻³, $F(000) = 230$, $\mu(\text{Mo } K\alpha) = 2.94$ cm⁻¹.

Intensity data were collected by the θ - 2θ scan method on a Philips automated four-circle diffractometer with graphite monochromated Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å). Reflexions were collected to a maximum value of $\sin \theta/\lambda = 0.60$. Three reference reflexions were measured after every 50 reflexions during the data collection. The variation in intensity was observed to be less than 2% of its mean value.

The intensities of 1040 independent reflexions were measured. 98 of these reflexions with $I < 2\sigma(I)$ were considered as unobserved and were assigned a zero weight throughout the refinement process. The data were corrected for Lorentz-polarization effects but not for absorption ($\mu R < 0.1$).

Solution and refinement of the structure

The data were placed on an approximately absolute scale by Wilson's (1942) method. The normalized structure factors E (Karle & Karle, 1966) were calculated with an overall temperature parameter of 2.8 Å². A statistical test of the distribution of normalized structure factors (Table 1) gave a strong indication for the absence of a centre of symmetry and the space group $P2_1$ was definitively assigned.

Table 1. E statistics

	Theoretical		
	Experimental	Centric	Acentric
$\langle E \rangle$	0.877	0.798	0.886
$\langle E ^2 - 1 \rangle$	0.702	0.968	0.736
$ E > 3$	0.00	0.003	0.000
$ E > 2$	0.02	0.010	0.018
$ E > 1$	0.39	0.320	0.368

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The structure was solved by multiresolution tangent formula refinement (Germain, Main & Woolfson, 1971) of 180 reflexions with $E \geq 1.30$. From the convergence mapping a successful starting set (Table 2) was obtained. The first three reflexions were used to fix the origin. 10,0,0 and 802 had a high probability ($P > 0.85$) and, therefore, were considered as known phases. From this information and with two appropriate symbolic phases, of which 831 was used to fix the enantiomorph, eight phase sets were generated.

Table 2. Starting set of phases

<i>h k l</i>	φ	<i>h k l</i>	φ
5 0 0	2π	8 0 2	2π
8 0 3	2π	8 3 1	$+ \pi/4$; $+ 3\pi/4$
9 1 0	2π	8 5 0	$\pm \pi/4$; $\pm 3\pi/4$
10 0 0	2π		

An *E* map based on the phases generated for the set with the highest 'figure of merit' revealed seven atomic positions of the molecule. The structure (excluding the H atoms) was completed from a three-dimensional electron-density synthesis in which the phase angles were determined from the known atomic positions. Isotropic temperature factors $\exp(-B \sin^2 \theta/\lambda^2)$ were

Table 3. Observed and calculated structure factors

The columns are in the order *h, k, l, F_o* and *F_c*.

10 0 2	1.1076	1.1076	10 0 2	10.796	10.796
11 0 0	1.0781	1.0781	11 0 0	10.796	10.796
11 0 1	1.0781	1.0781	11 0 1	10.796	10.796
11 0 2	1.0781	1.0781	11 0 2	10.796	10.796
11 0 3	1.0781	1.0781	11 0 3	10.796	10.796
11 0 4	1.0781	1.0781	11 0 4	10.796	10.796
11 0 5	1.0781	1.0781	11 0 5	10.796	10.796
11 0 6	1.0781	1.0781	11 0 6	10.796	10.796
11 0 7	1.0781	1.0781	11 0 7	10.796	10.796
11 0 8	1.0781	1.0781	11 0 8	10.796	10.796
11 0 9	1.0781	1.0781	11 0 9	10.796	10.796
11 0 10	1.0781	1.0781	11 0 10	10.796	10.796
11 0 11	1.0781	1.0781	11 0 11	10.796	10.796
11 0 12	1.0781	1.0781	11 0 12	10.796	10.796
11 0 13	1.0781	1.0781	11 0 13	10.796	10.796
11 0 14	1.0781	1.0781	11 0 14	10.796	10.796
11 0 15	1.0781	1.0781	11 0 15	10.796	10.796
11 0 16	1.0781	1.0781	11 0 16	10.796	10.796
11 0 17	1.0781	1.0781	11 0 17	10.796	10.796
11 0 18	1.0781	1.0781	11 0 18	10.796	10.796
11 0 19	1.0781	1.0781	11 0 19	10.796	10.796
11 0 20	1.0781	1.0781	11 0 20	10.796	10.796
11 0 21	1.0781	1.0781	11 0 21	10.796	10.796
11 0 22	1.0781	1.0781	11 0 22	10.796	10.796
11 0 23	1.0781	1.0781	11 0 23	10.796	10.796
11 0 24	1.0781	1.0781	11 0 24	10.796	10.796
11 0 25	1.0781	1.0781	11 0 25	10.796	10.796
11 0 26	1.0781	1.0781	11 0 26	10.796	10.796
11 0 27	1.0781	1.0781	11 0 27	10.796	10.796
11 0 28	1.0781	1.0781	11 0 28	10.796	10.796
11 0 29	1.0781	1.0781	11 0 29	10.796	10.796
11 0 30	1.0781	1.0781	11 0 30	10.796	10.796
11 0 31	1.0781	1.0781	11 0 31	10.796	10.796
11 0 32	1.0781	1.0781	11 0 32	10.796	10.796
11 0 33	1.0781	1.0781	11 0 33	10.796	10.796
11 0 34	1.0781	1.0781	11 0 34	10.796	10.796
11 0 35	1.0781	1.0781	11 0 35	10.796	10.796
11 0 36	1.0781	1.0781	11 0 36	10.796	10.796
11 0 37	1.0781	1.0781	11 0 37	10.796	10.796
11 0 38	1.0781	1.0781	11 0 38	10.796	10.796
11 0 39	1.0781	1.0781	11 0 39	10.796	10.796
11 0 40	1.0781	1.0781	11 0 40	10.796	10.796
11 0 41	1.0781	1.0781	11 0 41	10.796	10.796
11 0 42	1.0781	1.0781	11 0 42	10.796	10.796
11 0 43	1.0781	1.0781	11 0 43	10.796	10.796
11 0 44	1.0781	1.0781	11 0 44	10.796	10.796
11 0 45	1.0781	1.0781	11 0 45	10.796	10.796
11 0 46	1.0781	1.0781	11 0 46	10.796	10.796
11 0 47	1.0781	1.0781	11 0 47	10.796	10.796
11 0 48	1.0781	1.0781	11 0 48	10.796	10.796
11 0 49	1.0781	1.0781	11 0 49	10.796	10.796
11 0 50	1.0781	1.0781	11 0 50	10.796	10.796
11 0 51	1.0781	1.0781	11 0 51	10.796	10.796
11 0 52	1.0781	1.0781	11 0 52	10.796	10.796
11 0 53	1.0781	1.0781	11 0 53	10.796	10.796
11 0 54	1.0781	1.0781	11 0 54	10.796	10.796
11 0 55	1.0781	1.0781	11 0 55	10.796	10.796
11 0 56	1.0781	1.0781	11 0 56	10.796	10.796
11 0 57	1.0781	1.0781	11 0 57	10.796	10.796
11 0 58	1.0781	1.0781	11 0 58	10.796	10.796
11 0 59	1.0781	1.0781	11 0 59	10.796	10.796
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11 0 68	1.0781	1.0781	11 0 68	10.796	10.796
11 0 69	1.0781	1.0781	11 0 69	10.796	10.796
11 0 70	1.0781	1.0781	11 0 70	10.796	10.796
11 0 71	1.0781	1.0781	11 0 71	10.796	10.796
11 0 72	1.0781	1.0781	11 0 72	10.796	10.796
11 0 73	1.0781	1.0781	11 0 73	10.796	10.796
11 0 74	1.0781	1.0781	11 0 74	10.796	10.796
11 0 75	1.0781	1.0781	11 0 75	10.796	10.796
11 0 76	1.0781	1.0781	11 0 76	10.796	10.796
11 0 77	1.0781	1.0781	11 0 77	10.796	10.796
11 0 78	1.0781	1.0781	11 0 78	10.796	10.796
11 0 79	1.0781	1.0781	11 0 79	10.796	10.796
11 0 80	1.0781	1.0781	11 0 80	10.796	10.796
11 0 81	1.0781	1.0781	11 0 81	10.796	10.796
11 0 82	1.0781	1.0781	11 0 82	10.796	10.796
11 0 83	1.0781	1.0781	11 0 83	10.796	10.796
11 0 84	1.0781	1.0781	11 0 84	10.796	10.796
11 0 85	1.0781	1.0781	11 0 85	10.796	10.796
11 0 86	1.0781	1.0781	11 0 86	10.796	10.796
11 0 87	1.0781	1.0781	11 0 87	10.796	10.796
11 0 88	1.0781	1.0781	11 0 88	10.796	10.796
11 0 89	1.0781	1.0781	11 0 89	10.796	10.796
11 0 90	1.0781	1.0781	11 0 90	10.796	10.796
11 0 91	1.0781	1.0781	11 0 91	10.796	10.796
11 0 92	1.0781	1.0781	11 0 92	10.796	10.796
11 0 93	1.0781	1.0781	11 0 93	10.796	10.796
11 0 94	1.0781	1.0781	11 0 94	10.796	10.796
11 0 95	1.0781	1.0781	11 0 95	10.796	10.796
11 0 96	1.0781	1.0781	11 0 96	10.796	10.796
11 0 97	1.0781	1.0781	11 0 97	10.796	10.796
11 0 98	1.0781	1.0781	11 0 98	10.796	10.796
11 0 99	1.0781	1.0781	11 0 99	10.796	10.796
11 0 100	1.0781	1.0781	11 0 100	10.796	10.796
11 0 101	1.0781	1.0781	11 0 101	10.796	10.796
11 0 102	1.0781	1.0781	11 0 102	10.796	10.796
11 0 103	1.0781	1.0781	11 0 103	10.796	10.796
11 0 104	1.0781	1.0781	11 0 104	10.796	10.796
11 0 105	1.0781	1.0781	11 0 105	10.796	10.796
11 0 106	1.0781	1.0781	11 0 106	10.796	10.796
11 0 107	1.0781	1.0781	11 0 107	10.796	10.796
11 0 108	1.0781	1.0781	11 0 108	10.796	10.796
11 0 109	1.0781	1.0781	11 0 109	10.796	10.796
11 0 110	1.0781	1.0781	11 0 110	10.796	10.796
11 0 111	1.0781	1.0781	11 0 111	10.796	10.796
11 0 112	1.0781	1.0781	11 0 112	10.796	10.796
11 0 113	1.0781	1.0781	11 0 113	10.796	10.796
11 0 114	1.0781	1.0781	11 0 114	10.796	10.796
11 0 115	1.0781	1.0781	11 0 115	10.796	10.796
11 0 116	1.0781	1.0781	11 0 116	10.796	10.796
11 0 117	1.0781	1.0781	11 0 117	10.796	10.796
11 0 118	1.0781	1.0781	11 0 118	10.796	10.796
11 0 119	1.0781	1.0781	11 0 119	10.796	10.796
11 0 120	1.0781	1.0781	11 0 120	10.796	10.796
11 0 121	1.0781	1.0781	11 0 121	10.796	10.796
11 0 122	1.0781	1.0781	11 0 122	10.796	10.796
11 0 123	1.0781	1.0781	11 0 123	10.796	10.796
11 0 124	1.0781	1.0781	11 0 124	10.796	10.796
11 0 125	1.0781	1.0781	11 0 125	10.796	10.796
11 0 126	1.0781	1.0781	11 0 126	10.796	10.796
11 0 127	1.0781	1.0781	11 0 127	10.796	10.796
11 0 128	1.0781	1.0781	11 0 128	10.796	10.796
11 0 129	1.0781	1.0781	11 0 129	10.796	10.796
11 0 130	1.0781	1.0781	11 0 130	10.796	10.796
11 0 131	1.0781	1.0781	11 0 131	10.796	10.796
11 0 132	1.0781	1.0781	11 0 132	10.796	10.796
11 0 133	1.0781	1.0781	11 0 133	10.796	10.796
11 0 134	1.0781	1.0781	11 0 134	10.796	10.796
11 0 135	1.0781	1.0781	11 0 135	10.796	10.796
11 0 136	1.0781	1.0781	11 0 136	10.796	10.796
11 0 137	1.0781	1.0781	11 0 137	10.796	10.796
11 0 138	1.0781	1.0781	11 0 138	10.796	10.796
11 0 139	1.0781	1.0781	11 0 139	10.796	10.796
11 0 140	1.0781	1.0781	11 0 140	10.796	10.796
11 0 141	1.0781	1.0781	11 0 141	10.796	10.796
11 0 142	1.0781	1.0781	11 0 142	10.796	10.796
11 0 143	1.0781	1.0781	11 0 143	10.796	10.796
11 0 144	1.0781	1.0781	11 0 144	10.796	10.796
11 0 145	1.0781	1.0781	11 0 145	10.796	10.796
11 0 146	1.0781	1.0781	11 0 146	10.796	10.796
11 0 147	1.0781	1.0781	11 0 147	10.796	10.796
11 0 148	1.0781	1.0781	11 0 148	10.796	10.796
11 0 149	1.0781	1.0781	11 0 149	10.796	10.796
11 0 150	1.0781	1.0781	11 0 150	10.796	10.796

Table 3 (cont.)

11 0 151	1.0781	1.0781	11 0 151	10.796	10.796
11 0 152	1.0781	1.0781	11 0 152	10.796	10.796
11 0 153	1.0781	1.0781	11 0 153	10.796	10.796

Table 4. Atomic parameters

(a) Fractional coordinates and their standard deviations

	x	y	z
S	0.4132 (1)	0.0*	0.9046 (4)
O(1)	-0.0028 (4)	-0.1229 (5)	0.0165 (12)
O(2)	-0.0134 (4)	-0.4299 (4)	0.3735 (11)
O(3)	-0.1904 (4)	-0.3447 (5)	-0.1116 (10)
N(1)	0.3944 (4)	-0.2003 (6)	0.5254 (13)
N(2)	0.2122 (4)	-0.1064 (5)	0.5140 (12)
C(1)	0.5305 (5)	-0.2279 (8)	0.5945 (20)
C(2)	0.3382 (5)	-0.1025 (6)	0.6419 (14)
C(3)	0.1906 (6)	-0.2045 (6)	0.3091 (15)
C(4)	0.3037 (6)	-0.2650 (7)	0.3154 (16)
C(5)	0.0595 (5)	-0.2387 (6)	0.1432 (14)
C(6)	-0.0321 (6)	-0.2932 (6)	0.3241 (14)
C(7)	-0.1627 (5)	-0.2615 (6)	0.1356 (15)
C(8)	-0.1368 (6)	-0.1238 (8)	0.0359 (19)

(b) Anisotropic thermal parameters. The expression used is:

$$T = \exp[-10^{-4}(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
S	5701 (1)	70 (2)	478 (10)	-3 (1)	-1 (3)	-42 (4)
O(1)	69 (4)	59 (4)	501 (28)	-3 (3)	-8 (9)	62 (10)
O(2)	68 (5)	53 (4)	439 (27)	-5 (3)	30 (9)	41 (9)
O(3)	54 (4)	85 (5)	364 (26)	-11 (4)	7 (8)	-35 (10)
N(1)	47 (4)	63 (6)	389 (31)	9 (4)	4 (10)	-17 (11)
N(2)	39 (4)	52 (4)	391 (28)	-1 (4)	12 (9)	0 (11)
C(1)	28 (6)	126 (10)	766 (59)	25 (6)	-8 (14)	-43 (21)
C(2)	52 (6)	54 (6)	345 (33)	4 (5)	12 (11)	3 (13)
C(3)	57 (6)	58 (6)	331 (32)	-6 (5)	17 (11)	2 (13)
C(4)	43 (5)	77 (7)	459 (40)	4 (5)	15 (12)	7 (15)
C(5)	48 (5)	56 (6)	308 (33)	1 (4)	4 (11)	2 (12)
C(6)	63 (5)	48 (6)	320 (33)	-6 (5)	21 (12)	9 (12)
C(7)	46 (5)	63 (6)	370 (35)	8 (5)	32 (11)	-4 (13)
C(8)	49 (6)	77 (7)	709 (55)	3 (5)	22 (15)	32 (19)

* This coordinate was used to fix the origin.

Results and discussion

The molecular conformation of 1-methyl-4-(β -D-erythrofuranosyl)-4-imidazoline-2-thione is given in Fig. 1. The bond lengths and angles with the estimated standard deviations are given in Table 5.

Table 5. Bond lengths and angles in the molecule

(a) Bond lengths (Å) (standard deviations in parentheses)

S—C(2)	1.695 (6)	N(1)—C(4)	1.400 (9)
O(1)—C(5)	1.438 (8)	N(2)—C(2)	1.355 (7)
O(1)—C(8)	1.452 (8)	N(2)—C(3)	1.384 (8)
O(2)—C(6)	1.440 (7)	C(3)—C(4)	1.355 (9)
O(3)—C(7)	1.425 (8)	C(3)—C(5)	1.495 (9)
N(1)—C(1)	1.449 (7)	C(5)—C(6)	1.529 (9)
N(1)—C(2)	1.348 (8)	C(6)—C(7)	1.523 (9)
		C(7)—C(8)	1.542 (9)

(b) Bond angles (°) (standard deviations in the range 0.5–0.6°)

S—C(2)—N(2)	128.0	C(5)—O(1)—C(8)	110.2
S—C(2)—N(1)	125.5	C(3)—C(5)—O(1)	108.7
N(2)—C(2)—N(1)	106.4	O(1)—C(5)—C(6)	104.0
C(1)—N(1)—C(2)	124.5	C(3)—C(5)—C(6)	115.9
C(1)—N(1)—C(4)	125.2	C(5)—C(6)—C(7)	102.3
C(2)—N(1)—C(4)	110.1	C(7)—C(6)—O(2)	112.6
C(2)—N(2)—C(3)	109.6	C(5)—C(6)—O(2)	111.6
N(2)—C(3)—C(4)	107.8	C(6)—C(7)—O(3)	110.6
N(1)—C(4)—C(3)	106.1	C(8)—C(7)—O(3)	109.4
C(4)—C(3)—C(4)	129.4	C(6)—C(7)—C(8)	100.1
N(2)—C(3)—C(4)	122.5	C(7)—C(8)—O(1)	105.4

(i) Imidazoline ring

The bond lengths and angles in the imidazoline moiety agree well with the corresponding parameters from the other analogous compounds. The S—C(2), C(2)—N(1) and C(2)—N(2) distances indicate the effect of the thiourea resonance system (Valle, Cojazzi, Bus-

Table 6. Least-squares planes in the molecule

(a) Equations of various planes in the standard orthogonal system as: $Ax + By + Cz = D$

	Plane	A	B	C	D
I	Imidazoline ring	-0.3356	-0.6246	0.7052	1.7982
II	Five-atom furano ring	-0.0357	0.5452	0.8375	-0.6446
III	O(1)—C(5)—C(6)—C(7)	-0.0217	0.3646	0.9309	-0.3351
IV	O(1)—C(5)—C(6)—C(8)	0.1486	0.6003	0.7859	-0.8042
V	O(1)—C(5)—C(8)	0.0514	0.4544	0.8893	-0.5128

(l) Deviations in Å from the planes

	I	II	III	IV	V
S	-0.006 (*)				
O(1)		0.017	0.057	0.094	0.0
O(2)		-1.660 (*)	-1.400 (*)	-2.025 (*)	-1.662 (*)
O(3)		-0.328 (*)	0.319 (*)	-0.595 (*)	-0.011 (*)
N(1)	0.006				
N(2)	0.011				
C(1)	-0.057 (*)				
C(2)	-0.010				
C(3)	-0.007	0.616 (*)	0.846 (*)	0.905 (*)	0.901 (*)
C(4)	0.001				
C(5)	0.093 (*)	-0.170	0.035	-0.089	0.0
C(6)		0.137	0.054	-0.058	0.0
C(7)		-0.243	-0.032	-0.609 (*)	-0.260 (*)
C(8)		0.259	0.627 (*)	0.053	0.422 (*)

* Atom not included when calculating the plane.

setti & Mammi, 1970; Pérez-Garrido, López-Castro & Márquez, 1973; Jiménez-Garay, López-Castro & Márquez, 1974; Pérez-Garrido, Conde & Márquez, 1974). The N(1)–C(1) distance of 1.45 Å agrees well with the N–C (1.458 Å) distance given by Singh & Vijayan (1973) and the N–C bond length of 1.456 Å reported by Jiménez-Garay, López-Castro & Márquez (1974) for the 1-methyl-4,5-(D-glucofuran)imidazolidine-2-thione.

The imidazoline ring is planar as expected. The least-squares plane for the atoms of the ring and the corresponding deviations are shown in Table 6. The S and C(1) atoms (not included when calculating the plane) lie almost in the least-squares plane of the ring, at 0.01 and 0.06 Å below the plane, respectively. The torsion angles S–C(1)–N(1)–N(2) and C(2)–N(2)–C(1)–C(4) are 1.8 and 3.9°.

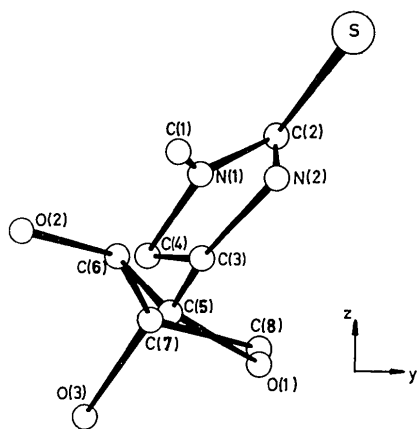


Fig. 1. A [100] view of the molecular conformation.

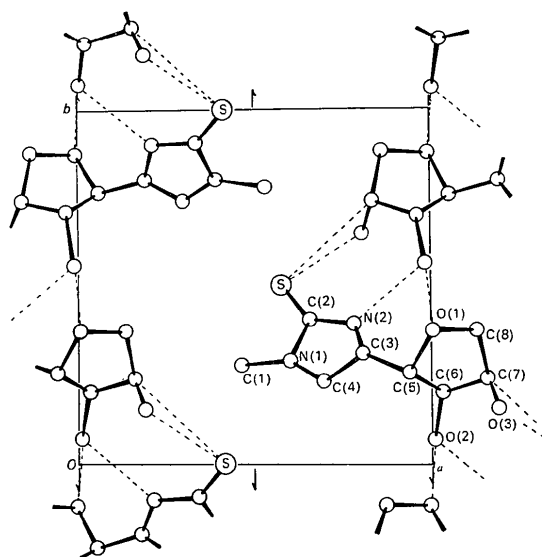


Fig. 2. Packing of the molecules as viewed along [001].

(ii) Sugar ring

The dimensions of the glucofuran ring are similar to those found in other nucleosides and nucleotides (Sundaralingam, 1969; Arnott, 1970; James & Matsushima, 1973; Sprang & Sundaralingam, 1973; Jiménez-Garay, López-Castro & Márquez, 1974; Pérez-Garrido, Conde & Márquez, 1974). As found in these other structures there is a discrepancy between the bond length C(5)–O(1), 1.44 Å, and C(8)–O(1), 1.45 Å. This distinction between the two ring C–O distances most likely has its source in the anomeric effect (Berman, Chu & Jeffrey, 1967).

The exocyclic–oxygen hydroxyl bond lengths are C(7)–O(3), 1.42 Å, and C(6)–O(2), 1.44 Å. These values agree well with the C–O bond distances in 1-methyl-4,5-(D-glucofuran)imidazolidine-2-thione (Jiménez-Garay, López-Castro & Márquez, 1974) and 1-*p*-chlorophenyl-4-(α -D-glucofuran)imidazoline-2-thione (Pérez-Garrido, Conde & Márquez, 1974) but they are longer than those found in ribose-containing nucleosides (Lai & Marsh, 1972; Viswamitra, Swaminatha Reddy, James & Williams, 1972; James & Matsushima, 1973).

The sugar ring adopts the C(6)-*cis* mode of puckering and is in the twist conformation 2T_3 in which C(6) and C(7) are displaced 0.26 and 0.24 Å respectively, on opposite sides of the furanose five-atom least-squares plane. Some least-squares planes for the sugar ring are given in Table 6.

Values for the important torsion angles are given in Table 7 and are similar to those in other nucleosides with analogous puckering of the sugar ring. Estimated standard deviations in torsion angles are in the range 0.3–0.5°.

Table 7. Intramolecular torsion angles (°)

S—C(2)–N(1)–N(2)	1.8
C(1)–N(1)–C(4)–C(2)	3.9
C(5)–C(3)–C(4)–N(2)	6.2
C(3)–C(5)–C(6)–O(1)	60.8
O(2)–C(6)–C(7)–C(5)	60.1
O(3)–C(7)–C(6)–C(8)	64.7
C(8)–O(1)–C(5)–C(6)	16.5
C(3)–C(5)–C(6)–C(7)	24.1
O(1)–C(5)–C(6)–C(7)	36.7
C(5)–C(6)–C(7)–C(8)	41.4
C(6)–C(7)–C(8)–O(1)	32.1
C(7)–C(8)–O(1)–C(5)	10.1
O(1)–C(5)–C(3)–C(4)	44.2
C(6)–C(5)–C(3)–N(2)	65.4

(iii) Conformation of the molecule

The dihedral angle between the mean planes of the imidazoline and the sugar ring is 74.2°. The conformation of the sugar ring relative of the imidazoline can be described in terms of a torsion angle about the glycosidic bond. This angle, defined for the sequence of atoms O(1)–C(5)–C(3)–C(4) (Sundaralingam, 1969) is 44.2°. Arnott (1970) uses the sugar atom C(6) as the reference point for his definition of this torsion angle. The χ value in Arnott's notation is 65.4°.

(iv) *Molecular packing*

Fig. 2 shows a view of the arrangement of the molecules in the unit cell as viewed along [001]. The molecular packing is clearly dominated by the formation of hydrogen bonds. Each molecule of 1-methyl-4-(β -D-erythrofuranosyl)-4-imidazoline-2-thione is connected to three neighbouring molecules. The details of molecular interactions are given in Table 8, which contains those intermolecular contacts which are less than the sum of the van der Waals radii (Pauling, 1966). One of the more interesting aspects of this packing scheme is the involvement of the sugar ring O(1) in the moderately strong hydrogen bond to O(2). Participation of O(1) in hydrogen bonding is relatively uncommon and is known to be generally weak in the cases in which it occurs (Sprang & Sundaralingam, 1973).

Table 8. *Intermolecular contacts* (\AA) *less than the sum of the van der Waals radii*

S-O(1 ⁱ)	3.184 (5)	O(1)-O(2 ⁱⁱ)	2.738 (7)
S-C(7 ⁱ)	3.615 (6)	O(2)-N(2 ⁱⁱⁱ)	2.930 (6)

Key to symmetry relations

- (i) $-x, \frac{1}{2}+y, -z$
- (ii) $-x, \frac{1}{2}+y, 1-z$
- (iii) $-x, -\frac{1}{2}+y, -z$

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All the calculations were performed in the Calcul Center of this University by means of a DCT 2000 terminal connected to a Univac 1108.

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