

Fig. 2. A stereoscopic view of the unit cell along [100].

Table 4. Bond distances (Å) and bond angles (°)

N(1)—N(2)	1.310 (3)	C(3a)—C(4)	1.395 (3)
N(1)—C(7a)	1.391 (3)	C(3a)—C(7a)	1.399 (3)
N(2)—C(3)	1.376 (3)	C(4)—C(5)	1.374 (3)
N(3)—N(4)	1.110 (3)	C(5)—C(6)	1.399 (3)
C(3)—N(3)	1.338 (3)	C(6)—C(7)	1.374 (3)
C(3)—C(3a)	1.418 (3)	C(7)—C(7a)	1.402 (3)
C(4)—H(4)	0.93 (3)	C(6)—H(6)	0.95 (3)
C(5)—H(5)	0.98 (3)	C(7)—H(7)	1.00 (3)
N(2)—N(1)—C(7a)	108.9 (2)	C(4)—C(3a)—C(7a)	121.5 (2)
N(1)—N(2)—C(3)	107.3 (2)	C(3a)—C(4)—C(5)	117.1 (2)
N(4)—N(3)—C(3)	179.6 (2)	C(4)—C(5)—C(6)	121.8 (2)
N(2)—C(3)—N(3)	119.5 (2)	C(5)—C(6)—C(7)	121.6 (2)
N(2)—C(3)—C(3a)	111.9 (2)	C(6)—C(7)—C(7a)	117.4 (2)
N(3)—C(3)—C(3a)	128.4 (2)	N(1)—C(7a)—C(3a)	110.9 (2)
C(3)—C(3a)—C(4)	137.6 (2)	N(1)—C(7a)—C(7)	128.5 (2)
C(3)—C(3a)—C(7a)	100.9 (2)	C(3a)—C(7a)—C(7)	120.6 (2)

planar to within 0.011 Å and the dihedral angle between the two rings is 2.0°. These effects are presumably due to molecular packing.

A stereoview of the molecular packing is given in Fig. 2. The molecules are arranged in layers nearly parallel to the *ac* plane, the interlayer separation being approximately 3.4 Å; the molecules are held together by van der Waals forces.

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1-*p*-Tolyl-4-(β-D-erythrofuransyl)imidazoline-2-thione

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Abstract. $\text{SN}_2\text{O}_3\text{C}_{14}\text{H}_{16}$, orthorhombic, space group $P2_12_12_1$, $a = 26.215$ (14), $b = 7.608$ (5), $c = 7.142$ (4) Å; $Z = 4$; $D_c = 1.36$, $D_m = 1.37$ g cm⁻³. The structure was solved by direct methods and refined by full-matrix

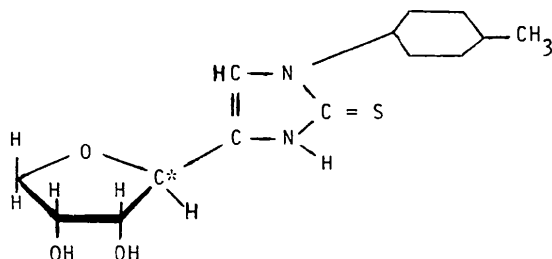
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least-squares procedures to a final R of 0.040. The sugar-ring puckering has the twist conformation ${}_3T$. The molecules are linked by hydrogen bonds and van der Waals forces.

Introduction. The structure determination of 1-*p*-tolyl-4-(β -D-erythrofuranosyl)imidazoline-2-thione



was undertaken as part of a series of structural studies of glucimidazoles and imidazole C-nucleosides. A structural study of 1-*p*-tolyl-4-(α -D-erythrofuranosyl)imidazoline-2-thione has already been made (Barragán, López-Castro & Márquez, 1977).

The title compound was obtained by heating under pressure an aqueous solution of 1-*p*-tolyl-4-(D-arabinotetrahydroxybutyl)imidazoline-2-thione. The reaction takes place with inversion at C*.

This structure determination completes the conformational study of the two anomers obtained by dehydration of 1-*p*-tolyl-4-(D-arabinotetrahydroxybutyl)imidazoline-2-thione.

Crystals were grown by slow evaporation of an ethanol solution and were yellow prisms elongated along *c*. Weissenberg and rotation photographs established the orthorhombic space group $P2_12_12_1$. The cell dimensions were obtained by least-squares analysis of diffractometer measurements with Mo $K\alpha$ radiation ($\lambda = 0.7107 \text{ \AA}$).

Intensity data were collected by the ω - 2θ scan method on a Philips automated four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radi-

ation. Reflections were collected to a maximum $\sin \theta/\lambda$ of 0.60. Three reference reflections were used during the data collection. The variation in intensity was observed to be less than 2% of the mean value.

The intensities of 2431 independent reflections were measured. Of these, 890 with $I < 2\sigma(I)$ were considered unobserved and were assigned zero weight through the refinement process. Data were corrected for Lorentz and polarization effects. No absorption correction was applied ($\mu R < 0.1$).

The structure was solved by the multisolution tangent formula method (Germain, Main & Woolfson, 1971). The data were placed on an absolute scale (Wilson, 1942) and normalized structure factors calculated. The first *E* map revealed nine non-hydrogen atoms. The structure was completed from a three-dimensional electron-density synthesis in which the phase angles were determined from the known atomic positions. The whole structure was then refined by full-matrix least-squares methods, varying all positional and thermal parameters. Three cycles with isotropic thermal refinement followed by three anisotropic cycles lowered *R* to 0.060. A subsequent difference synthesis revealed the 16 H atoms. The positional parameters of the H atoms, with isotropic thermal parameters equal to those of the carrier atoms, were kept constant in the refinement process. The quantity minimized was $\sum w(F_o - F_c)^2$ with weights $w = \sigma^{-2}(F_o)$. Standard tabulations of scattering factors were used for S, O, N and C (Cromer & Mann, 1968) and H (*International Tables for X-ray Crystallography*, 1962). The refinement was terminated at $R = 0.040$. The positional parameters from the final least-squares cycle are listed in Tables 1 and 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33003 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Atomic coordinates ($\times 10^4$) for non-hydrogen atoms

	<i>x</i>	<i>y</i>	<i>z</i>
O(1)	521 (1)	8698 (4)	-1917 (4)
O(2)	-82	11779 (4)	-333 (4)
O(3)	478 (1)	10598 (4)	2664 (4)
N(1)	1389 (1)	4969 (5)	1022 (5)
N(2)	742 (1)	6217 (5)	2383 (5)
C(1)	3196 (2)	988 (7)	-802 (10)
C(2)	1828 (2)	3873 (6)	640 (8)
C(3)	2196 (2)	3663 (7)	1957 (8)
C(4)	2638 (2)	2665 (7)	1463 (11)
C(5)	2694 (2)	1985 (7)	-328 (12)
C(6)	2309 (2)	2216 (7)	-1622 (9)
C(7)	1863 (2)	3156 (7)	-1127 (7)
C(8)	1093 (2)	4983 (6)	2624 (6)
C(9)	1207 (2)	6273 (6)	-193 (6)
C(10)	802 (2)	7009 (6)	654 (7)
C(11)	477 (2)	8545 (7)	62 (6)
C(12)	664 (2)	10345 (6)	815 (7)
C(13)	450 (2)	11610 (6)	-652 (7)
C(14)	558 (2)	10565 (7)	-2406 (7)
S	1140 (1)	3598 (2)	4492 (2)

Table 2. Atomic coordinates of H ($\times 10^3$) and isotropic temperature factors

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (\AA^2)
H1(C1)	307	-25	-85	2.3
H2(C1)	333	100	-200	2.3
H3(C1)	327	40	20	2.3
H(C3)	212	400	340	4.0
H(C4)	285	230	260	5.1
H(C6)	232	145	-295	4.7
H(C7)	163	340	-218	3.8
H(C9)	135	630	-150	3.0
H(C11)	10	840	59	2.8
H(C12)	108	1030	70	2.0
H(C13)	58	1282	-60	2.8
H1(C14)	29	1085	-35	3.9
H2(C14)	90	1090	-30	3.9
H(N2)	48	660	350	1.6
H(O3)	15	1015	275	3.4
H(O2)	-22	1240	-140	4.0

Discussion. A perspective view of the molecule with the numbering scheme is shown in Fig. 1. The bond lengths and angles are given in Tables 3 and 4. Most have reasonable values and there are no significant deviations from the values found in analogous compounds. The C(1)–C(5) distance of 1.56 Å, although rather long, is in good agreement with the C–C single bond (1.56 Å) found by Hoge & Nordman (1974).

The computed least-squares planes in the molecule are: $0.439x + 0.843y - 0.312z = 4.431$, $0.610x + 0.675y + 0.416z = 5.082$ and $0.998x - 0.011y - 0.067z = 1.352$ for the phenyl, imidazoline and furanosyl rings respectively. Both the phenyl and the imidazoline rings are planar with displacements in the range of the standard deviations. The dihedral angle between the phenyl and imidazoline rings is 45° . The furanosyl ring is not planar, as expected. The dihedral angle between the mean planes of the imidazoline and furanosyl rings is 55° . For the α anomer (Barragán, López-Castro & Márquez, 1977) the dihedral angles were 53° and 87° respectively.

As found in other compounds the C–O endocyclic bonds are practically the same for the α anomer and different for the β anomer. In fact O(1)–C(14) = 1.47

and O(1)–C(11) = 1.42 Å. This may be due to the anomeric effect (Berman, Chu & Jeffrey, 1967; Conde, Moreno & Márquez, 1975).

The conformation of the sugar ring can be described (Sundaralingam, 1973) by ring torsion angles and the least-squares planes. In the title compound, the β anomer, C(12) has a maximum deviation of 0.26 Å on the same side as C(10), and C(13) has the next largest deviation of 0.24 Å on the opposite side of the mean

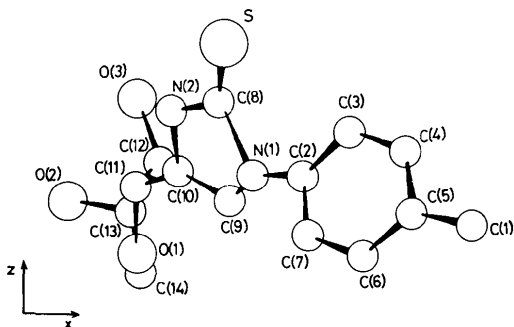


Fig. 1. A perspective view of the molecule.

Table 3. Bond lengths (Å)

O(1)–C(11)	1.42 (0)	C(2)–C(7)	1.38 (1)
O(1)–C(14)	1.47 (1)	C(3)–C(4)	1.43 (1)
O(2)–C(13)	1.42 (1)	C(4)–C(5)	1.39 (1)
O(3)–C(12)	1.42 (1)	C(5)–C(6)	1.38 (1)
N(1)–C(2)	1.45 (1)	C(6)–C(7)	1.41 (1)
N(1)–C(8)	1.38 (1)	C(8)–S	1.70 (0)
N(1)–C(9)	1.40 (1)	C(9)–C(10)	1.34 (1)
N(2)–C(8)	1.33 (1)	C(10)–C(11)	1.51 (1)
N(2)–C(10)	1.38 (1)	C(11)–C(12)	1.55 (1)
C(1)–C(5)	1.56 (1)	C(12)–C(13)	1.53 (1)
C(2)–C(3)	1.36 (1)	C(13)–C(14)	1.51 (1)

Table 4. Bond angles ($^\circ$)

C(11)–O(1)–C(14)	108.8 (3)	N(2)–C(8)–S	126.1 (4)
C(2)–N(1)–C(8)	127.3 (4)	N(1)–C(8)–N(2)	106.7 (4)
C(2)–N(1)–C(9)	124.2 (4)	N(1)–C(9)–C(10)	106.6 (4)
C(8)–N(1)–C(9)	108.4 (4)	N(2)–C(10)–C(11)	121.1 (4)
C(8)–N(2)–C(10)	110.2 (4)	C(9)–C(10)–C(11)	130.0 (4)
N(1)–C(2)–C(3)	120.2 (4)	N(2)–C(10)–C(9)	108.0 (4)
N(1)–C(2)–C(7)	117.0 (4)	O(1)–C(11)–C(10)	107.3 (4)
C(3)–C(2)–C(7)	122.7 (5)	O(1)–C(11)–C(12)	104.3 (4)
C(2)–C(3)–C(4)	117.9 (5)	C(10)–C(11)–C(12)	114.2 (4)
C(3)–C(4)–C(5)	120.7 (6)	O(3)–C(12)–C(13)	115.2 (4)
C(1)–C(5)–C(4)	118.1 (6)	O(3)–C(12)–C(11)	109.5 (4)
C(1)–C(5)–C(6)	122.4 (6)	C(11)–C(12)–C(13)	101.7 (4)
C(4)–C(5)–C(6)	119.5 (6)	O(2)–C(13)–C(12)	107.9 (4)
C(5)–C(6)–C(7)	120.1 (5)	C(12)–C(13)–C(14)	99.7 (4)
C(2)–C(7)–C(6)	119.0 (5)	O(2)–C(13)–C(14)	111.4 (4)
N(1)–C(8)–S	127.1 (4)	O(1)–C(14)–C(13)	107.5 (4)

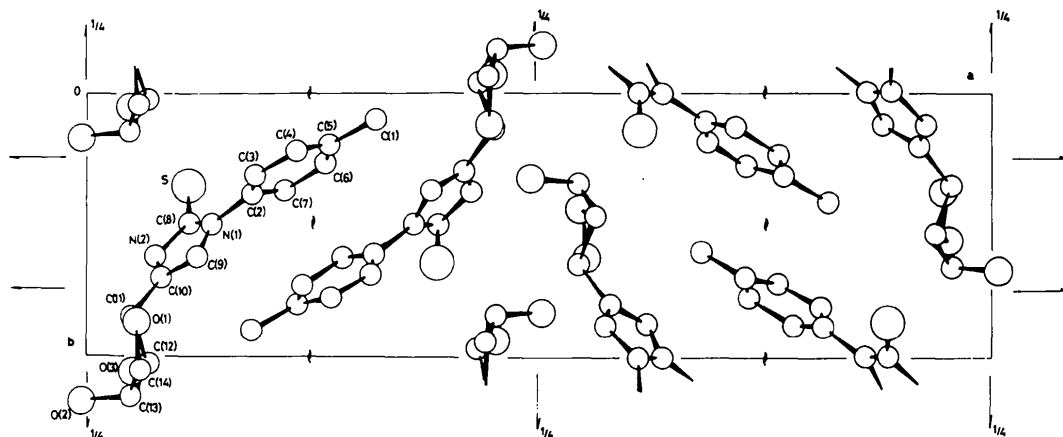


Fig. 2. Projection of the structure along the *c* axis.

Table 5. *Torsion angles* ($^{\circ}$)

C(14)–O(1)–C(11)–C(12)	–17.9 (5)
O(1)–C(11)–C(12)–C(13)	37.7 (5)
C(11)–C(12)–C(13)–C(14)	–41.2 (4)
C(12)–C(13)–C(14)–O(1)	31.9 (5)
C(13)–C(14)–O(1)–C(11)	–9.1 (5)
O(1)–C(11)–C(10)–C(9)	24.1 (7)
O(2)–C(13)–C(14)–O(1)	–81.7 (4)
O(3)–C(12)–C(11)–O(1)	159.8 (3)
O(3)–C(12)–C(13)–C(14)	–159.4 (4)
O(2)–C(13)–C(12)–C(11)	75.1 (13)

plane so that the sugar pucker is C(12)-*endo*, C(13)-*exo*. From the torsion angles in Table 5 it can be deduced that the furanose ring is in the 3T conformation.

The anomeric configuration can be defined by the atoms bonded to the furanose ring. In the β anomer C(10) is on the opposite side of the mean plane to O(2) and O(3), and these three atoms have deviations of 0.66, –1.65 and –0.32 Å respectively, while the α anomer has C(10), O(2) and O(3) on the same side of the least-squares plane.

Fig. 2 shows the arrangement of the molecules in the unit cell as viewed along [010]. The molecules are linked by hydrogen bonds and van der Waals forces. Each molecule is involved in two strong hydrogen bonds, O(1)···H–O(2) of 2.70 Å and N(2)–H···O(2) of 2.76 Å. Both contacts are between molecules related by twofold screw axes and translated, for the first, along the *bc* diagonal, and, for the second, along the *b* axis. There is a short S···C(14) distance of 3.55 Å between molecules translated along the *bc* diagonal that may indicate some van der Waals

interaction. The effect is to produce a three-dimensional network of molecules.

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1,9-Diacetoxy-5-chloro-12,12-dimethyltricyclo[6.2.2.0^{2,7}]dodeca-4,9-diene-3,6-dione

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Abstract. C₁₈H₁₉O₆Cl, triclinic, $P\bar{1}$, $a = 13.280$ (6), $b = 8.638$ (5), $c = 8.164$ (5) Å, $\alpha = 107.6$ (2), $\beta = 82.4$ (2), $\gamma = 97.7$ (2) $^{\circ}$, $V = 880.7$ Å³, $M_r = 366.5$, $D_m = 1.36$, $D_c = 1.38$ g cm⁻³ for $Z = 2$. The structure

was solved by direct methods to a final R of 0.073 for 1724 unique X-ray diffractometer data. There are no unusual bond lengths.

Introduction. 1-Methoxycyclohexa-1,3-diene has been shown to undergo regiospecific Diels–Alder addition to

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