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Structure of a Glucofuranosoimidazolidine

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Abstract. 3-Ethyl-1-phenyl-(3,5,6,7-tetra-*O*-acetyl-1,2-dideoxy-D-*glycero*-β-D-*talo*-heptofuranoso)[2,1-*d*]-imidazolidine-2-thione,* $C_{24}H_{30}N_2O_9S$, $M_r = 522{\cdot}6$, orthorhombic, $P2_12_12_1$, $a = 15{\cdot}347$ (5), $b = 22{\cdot}598$ (3), $c = 7{\cdot}683$ (1) Å, $V = 2665$ (1) Å^3, $Z = 4$, $D_m = 1{\cdot}285$ (5), $D_x = 1{\cdot}303$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}16$ mm^{-1}, $F(000) = 1104$, room temperature, final $wR = 0{\cdot}047$ for 1637 observed reflexions. The imidazolidine ring is planar and the sugar ring adopts the ^4E conformation. The imidazolidinefuranose bicycle shows a *cis* form of coupling and the dihedral angle between the least-squares planes through the rings is 70·1 (3)°. The phenyl ring forms a dihedral angle of 112·9 (3)° with the imidazolidine ring. Some C—H···O intermolecular contacts could be considered as weak hydrogen bonds.

Experimental. Single crystals in the form of colourless needles elongated along [001] prepared in the Organic Chemistry Department of the University of Extremadura and kindly supplied by Professor J. A. Galbis-Pérez. D_m by flotation method. Crystal 0·07 × 0·08 × 0·25 mm. Unit-cell parameters from 25 reflexions, $4 < \theta < 11°$. Enraf–Nonius CAD-4 diffractometer, graphite monochromator, $2 < \theta < 25°$ ($0 \le h \le 21$, $0 \le k \le 31$, $0 \le l \le 10$), ω–2θ scan mode. Two standard reflexions ($4\bar{2}0$, 420), variation in intensity <3% of mean value. 2665 independent reflexions measured, 1028 considered unobserved [$I < 2\sigma(I)$]. Lorentz and polarization correction, no correction for absorption or extinction. Patterson function and heavy-atom method with initial set of phases based on S-atom position. Full-matrix least-squares refinement on F, anisotropic. H atoms calculated from expected geometry (C—H = 1·08 Å); isotropic temperature factor $B = 4{\cdot}0$ Å^2 for H atoms, not refined. Further least-squares refinement including anomalous-dispersion correction for S atomic scattering factor (*International Tables for X-ray Crystallography*, 1974) reduced wR to 0·047 ($R = 0{\cdot}100$); weighting scheme based on statistical-count criterion ($w = 1/\sigma_F^2$). $(\Delta/\sigma)_{\max} = 0{\cdot}50$, $S = 0{\cdot}80$ for 325 refined parameters. Number reflexions/number parameters = 5·0. The large difference between final wR and R values should be attributed to the poor quality of the crystals. The major discrepancies between observed and calculated structure amplitudes are found, generally, for high-order reflexions with low amplitude and high standard deviation. Final difference synthesis showed $0{\cdot}40 > \Delta\rho > -0{\cdot}35$ e Å^{-3}. The enantiomorphic form of the molecule was assumed by reference to the sugar moiety (Galbis-Pérez, Palacios-Albarrán, Jiménez-Requejo & Avalos-González, 1984). Crystallographic programs of *XRAY70* system (Stewart, Kundell & Baldwin, 1970) used throughout.

Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j\cos(a_i,a_j).$$

	x	y	z	$U_{\rm eq}$
S	0·3067 (1)	0·7603 (1)	−0·1490 (3)	62 (1)
O(1)	0·3913 (3)	0·8413 (2)	0·4091 (6)	46 (2)
O(2)	0·5199 (3)	0·7129 (2)	0·3359 (7)	47 (2)
O(3)	0·5387 (3)	0·8326 (2)	0·6531 (7)	47 (2)
O(4)	0·5529 (3)	0·9171 (2)	0·2590 (7)	51 (2)
O(5)	0·7057 (3)	0·9099 (2)	0·4804 (7)	56 (2)
O(6)	0·5172 (3)	0·6506 (2)	0·5649 (8)	80 (3)
O(7)	0·6472 (3)	0·7666 (2)	0·6459 (8)	76 (3)
O(8)	0·5034 (4)	1·0099 (2)	0·2828 (10)	97 (3)
O(9)	0·7336 (3)	0·9820 (2)	0·2809 (8)	75 (3)
N(1)	0·3047 (4)	0·8127 (2)	0·1690 (8)	45 (3)
N(2)	0·3606 (4)	0·7221 (2)	0·1623 (9)	52 (3)
C(1)	0·3262 (5)	0·7651 (3)	0·0616 (12)	56 (4)
C(2)	0·3241 (4)	0·8020 (3)	0·3471 (11)	37 (3)
C(3)	0·3646 (5)	0·7393 (3)	0·3434 (11)	43 (3)
C(4)	0·4554 (4)	0·7487 (3)	0·4209 (9)	28 (3)
C(5)	0·4722 (5)	0·8124 (3)	0·3706 (11)	35 (3)
C(6)	0·5475 (5)	0·8418 (3)	0·4637 (10)	30 (3)
C(7)	0·5474 (5)	0·9110 (3)	0·4433 (1)	48 (3)
C(8)	0·6265 (6)	0·9390 (3)	0·5299 (1)	68 (4)
C(9)	0·2687 (6)	0·8670 (4)	0·1053 (12)	44 (4)
C(10)	0·3150 (7)	0·9045 (5)	0·0043 (13)	94 (6)
C(11)	0·2758 (10)	0·9585 (6)	−0·0476 (20)	114 (7)
C(12)	0·1928 (10)	0·9729 (6)	0·0010 (21)	110 (8)
C(13)	0·1503 (8)	0·9340 (6)	0·1047 (16)	83 (6)
C(14)	0·1859 (7)	0·8808 (4)	0·1541 (14)	67 (4)
C(15)	0·3742 (6)	0·6620 (4)	0·1057 (12)	61 (4)
C(16)	0·2919 (7)	0·6266 (4)	0·1135 (14)	85 (5)
C(17)	0·5476 (5)	0·6646 (3)	0·4280 (13)	46 (4)
C(18)	0·6210 (6)	0·6340 (4)	0·3333 (13)	75 (4)
C(19)	0·5932 (6)	0·7917 (4)	0·7259 (12)	58 (4)
C(20)	0·5699 (6)	0·7831 (5)	0·9177 (12)	78 (5)
C(21)	0·5293 (7)	0·9715 (4)	0·1927 (13)	67 (5)
C(22)	0·5416 (8)	0·9746 (5)	−0·0005 (15)	95 (6)
C(23)	0·7535 (5)	0·9360 (3)	0·3563 (11)	57 (3)
C(24)	0·8366 (5)	0·9030 (3)	0·3200 (14)	72 (4)

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* IUPAC name: 1-{3-acetoxy-4-ethyl-6-phenyl-5-thioxo-2,3,3a,-5,6,6a-hexahydro-4*H*-furo[2,3-*d*]imidazol-2-yl}trimethylene triacetate.

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Table 2. *Bond distances* (Å) *and angles* (°)

S–C(1)	1·648 (9)	C(2)–C(3)	1·547 (10)
C(3)–C(4)	1·530 (10)	C(4)–C(5)	1·513 (10)
C(5)–C(6)	1·512 (10)	C(6)–C(7)	1·572 (10)
C(7)–C(8)	1·522 (12)	C(9)–C(10)	1·350 (15)
C(9)–C(14)	1·361 (14)	C(10)–C(11)	1·419 (18)
C(11)–C(12)	1·367 (22)	C(12)–C(13)	1·355 (19)
C(13)–C(14)	1·374 (16)	C(15)–C(16)	1·497 (13)
C(17)–C(18)	1·508 (13)	C(19)–C(20)	1·530 (13)
C(21)–C(22)	1·497 (15)	C(23)–C(24)	1·504 (11)
O(1)–C(2)	1·442 (8)	O(1)–C(5)	1·434 (9)
O(2)–C(4)	1·434 (8)	O(2)–C(17)	1·369 (10)
O(3)–C(6)	1·477 (9)	O(3)–C(19)	1·366 (10)
O(4)–C(7)	1·424 (10)	O(4)–C(21)	1·379 (10)
O(5)–C(8)	1·434 (10)	O(5)–C(23)	1·340 (9)
O(6)–C(17)	1·193 (11)	O(7)–C(19)	1·177 (11)
O(8)–C(21)	1·178 (11)	O(9)–C(23)	1·227 (9)
N(1)–C(1)	1·394 (10)	N(1)–C(2)	1·421 (10)
N(1)–C(9)	1·432 (10)	N(2)–C(1)	1·349 (10)
N(2)–C(3)	1·445 (11)	N(2)–C(15)	1·441 (10)

C(2)–O(1)–C(5)	105·7 (5)	C(4)–O(2)–C(17)	115·4 (6)
C(6)–O(3)–C(19)	116·2 (6)	C(7)–O(4)–C(21)	116·0 (6)
C(8)–O(5)–C(23)	116·8 (6)	C(2)–N(1)–C(9)	123·7 (6)
C(1)–N(1)–C(9)	123·3 (7)	C(1)–N(1)–C(2)	112·9 (6)
C(3)–N(2)–C(15)	122·5 (7)	C(1)–N(2)–C(15)	124·2 (7)
C(1)–N(2)–C(3)	112·0 (6)	N(1)–C(1)–N(2)	107·9 (7)
S–C(1)–N(2)	125·9 (6)	S–C(1)–N(1)	126·0 (6)
O(1)–C(2)–N(1)	111·3 (5)	N(1)–C(2)–C(3)	102·8 (6)
O(1)–C(2)–C(3)	106·4 (5)	N(2)–C(3)–C(2)	104·3 (6)
C(2)–C(3)–C(4)	103·4 (6)	N(2)–C(3)–C(4)	116·8 (6)
O(2)–C(4)–C(3)	111·9 (6)	C(3)–C(4)–C(5)	100·8 (6)
O(2)–C(4)–C(5)	107·6 (5)	O(1)–C(5)–C(4)	103·5 (6)
C(4)–C(5)–C(6)	115·3 (6)	O(1)–C(5)–C(6)	111·4 (6)
O(3)–C(6)–C(5)	109·5 (6)	C(5)–C(6)–C(7)	112·8 (6)
O(3)–C(6)–C(7)	103·7 (5)	O(4)–C(7)–C(6)	101·3 (6)
C(6)–C(7)–C(8)	111·6 (6)	O(4)–C(7)–C(8)	110·3 (6)
O(5)–C(8)–C(7)	111·7 (6)	N(1)–C(9)–C(14)	117·5 (8)
N(1)–C(9)–C(10)	122·1 (8)	C(10)–C(9)–C(14)	120·4 (9)
C(9)–C(10)–C(11)	118·6 (11)	C(10)–C(11)–C(12)	121·5 (13)
C(11)–C(12)–C(13)	117·0 (13)	C(12)–C(13)–C(14)	122·7 (12)
C(9)–C(14)–C(13)	119·7 (10)	N(2)–C(15)–C(16)	111·7 (7)
O(2)–C(17)–O(6)	123·1 (7)	O(6)–C(17)–C(18)	126·5 (8)
O(2)–C(17)–C(18)	110·4 (7)	O(3)–C(19)–O(7)	123·0 (8)
O(7)–C(19)–C(20)	127·2 (8)	O(3)–C(19)–C(20)	109·7 (7)
O(4)–C(21)–O(8)	121·7 (9)	O(8)–C(21)–C(22)	126·3 (9)
O(4)–C(21)–C(22)	112·0 (8)	O(5)–C(23)–O(9)	124·9 (7)
O(9)–C(23)–C(24)	122·9 (7)	O(5)–C(23)–C(24)	112·2 (6)

Table 3. *Possible hydrogen bonds*

X–H···Y	X···Y	H···Y	X–H···Y
C(2)–H(2)···O(7^{i})	3·126 (8) Å	2·55 (8) Å	112 (5)°
C(14)–H(14)···O(6^{i})	3·445 (12)	2·55 (11)	139 (6)
C(16)–H(162)···O(5^{i})	3·488 (12)	2·45 (11)	160 (7)
C(16)–H(163)···O(9^{ii})	3·390 (10)	2·58 (9)	131 (7)
C(24)–H(242)···O(6^{iii})	3·152 (10)	2·48 (7)	119 (7)

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$.

Table 1* gives the atomic parameters for non-H atoms and Table 2 gives bond lengths and angles in the molecule, calculated by *PARST* (Nardelli, 1983). Fig. 1 indicates the labelling of the atoms and Fig. 2 shows the packing in the unit cell. Table 3 describes the possible hydrogen bonds.

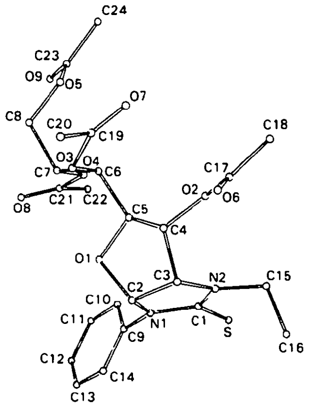


Fig. 1. Labelling of the atoms in the molecule.

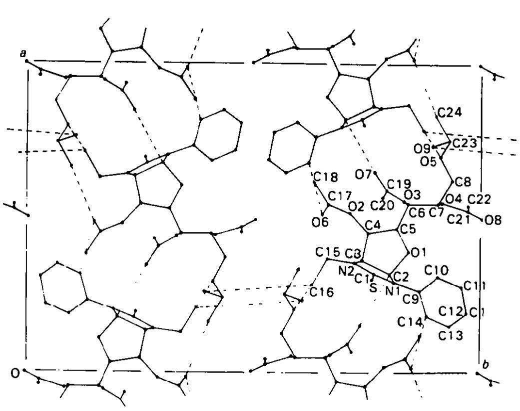
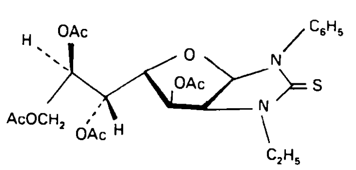


Fig. 2. A [001] projection of the unit cell. Possible hydrogen bonds are indicated by dashed lines.

Related literature. The title compound (I) is one of a series prepared by Galbis-Pérez *et al.* (1984). Previous structures of the series have been recently reported (Estrada, Conde & Márquez, 1984, 1986; Conde, Millán, Conde & Márquez, 1985*a*,*b*).



(I)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43052 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of an Iridoid-like Photolysis Product

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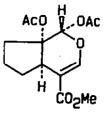
Abstract. Methyl 1α,7aα-diacetoxy-1,4aα,5,6,7,7a-hexahydrocyclopenta[*c*]pyran-4-carboxylate, $C_{14}H_{18}O_7$, $M_r = 298\cdot3$, triclinic, $P\bar{1}$, $a = 5\cdot615$ (2), $b = 8\cdot013$ (1), $c = 16\cdot673$ (4) Å, $\alpha = 89\cdot98$ (2), $\beta = 87\cdot90$ (2), $\gamma = 85\cdot07$ (2)°, $V = 747$ Å^3, $Z = 2$, $D_x = 1\cdot326$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1\cdot0$ cm^{-1}, $F(000) = 316$, $T = 298$ K, $R = 0\cdot050$ for 2069 observed reflections. The title compound is the product of a photolysis reaction. From spectroscopic data it was not clear whether the ring junction was *cis* (as established here) or *trans*. The six-membered ring adopts a half-chair conformation, whereas the five-membered ring does not correspond closely to any standard conformation. The acetyl groups lie equatorial [C(7a)] and axial [C(1)] respectively.

Experimental. (I): crystal 0·3 × 0·3 × 0·4 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and and speed (Clegg, 1981). 2628 unique reflections, $2\theta_{\max}$ 50°, $\pm h \pm k + l$, three check reflections with no intensity change. 2069 reflections with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*, Sheldrick, 1978). Index ranges $|h| \leq 6$, $|k| \leq 9$, $l \leq 19$. Cell constants refined from $\pm 2\theta$ values of 20 reflections in the range 20–25°. Absorption and extinction corrections unnecessary.



(I)

Structure solution by multisolution direct methods. Refinement on F to $R = 0\cdot050$, $wR = 0\cdot069$; all non-H atoms anisotropic, H atoms included using a riding model [C—H 0·96 Å, $U(\mathrm{H}) = 1\cdot2U_{\mathrm{eq}}(\mathrm{C})$], 199 parameters, $S = 1\cdot89$, weighting scheme $w^{-1} = \sigma^2(F) + 0\cdot00064F^2$ which gave a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0\cdot067$, max. and min. height in final $\Delta\rho$ map 0·18 and −0·17 e Å^{-3} respectively. Atomic scattering factors from *SHELXTL*.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows the atom numbering.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å$^2 \times 10^3$)

	x	y	z	U_{eq}*
C(1)	6642 (4)	6525 (3)	8154 (1)	56 (1)
O(2)	8142 (3)	7810 (2)	7949 (1)	66 (1)
C(3)	7293 (4)	8910 (3)	7391 (2)	63 (1)
C(4)	5519 (4)	8695 (2)	6910 (1)	53 (1)
C(4a)	4328 (3)	7083 (2)	6891 (1)	49 (1)
C(5)	4555 (4)	6281 (3)	6059 (1)	62 (1)
C(6)	7133 (5)	5519 (3)	6001 (1)	73 (1)
C(7)	7697 (4)	4930 (3)	6843 (1)	53 (1)
C(7a)	5717 (3)	5770 (3)	7403 (1)	48 (1)
O(8)	3932 (2)	4614 (2)	7634 (1)	53 (1)
C(9)	4578 (4)	3223 (3)	8056 (1)	55 (1)
C(10)	2503 (4)	2242 (3)	8240 (2)	71 (1)
O(11)	6586 (3)	2850 (2)	8263 (1)	68 (1)
C(12)	4723 (4)	10123 (3)	6407 (1)	59 (1)
O(13)	2504 (3)	10031 (2)	6153 (1)	66 (1)
C(14)	1571 (5)	11392 (3)	5665 (2)	82 (1)
O(15)	5864 (3)	11281 (2)	6233 (1)	94 (1)
O(16)	4607 (3)	7231 (2)	8605 (1)	64 (1)
C(17)	4773 (5)	7311 (4)	9412 (1)	76 (1)
C(18)	2479 (5)	8060 (4)	9790 (2)	100 (1)
O(19)	6554 (4)	6870 (4)	9742 (1)	118 (1)

* U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43129 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Engalnd.

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