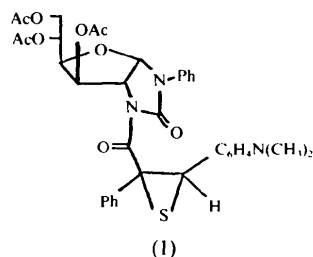


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3-[(2*R*,3*R*)-3-(4-Dimethylaminophenyl)-2-phenyl-2,3-epithiopropanoyl]-1-phenyl-(3,5,6-tri-*O*-acetyl-1,2-dideoxy- α -D-glucofuranoso)[1,2-*d*]imidazolidin-2-one

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Abstract

This work unambiguously establishes the overall conformation of the title molecule, C₃₆H₃₇N₃O₉S, which includes a thiirane ring. The configuration is α -D-glucofuranose with the furanosyl ring *T*₄³; the imidazolidine-2-one group assumes a form in which the fused C2' atom deviates by 0.14 Å from the best plane.

Comment

3-[(2*R*,3*R*)-3-(4-Dimethylaminophenyl)-2-phenyl-2,3-epithiopropanoyl]-1-phenyl-(3,5,6-tri-*O*-acetyl-1,2-dideoxy- α -D-glucofuranoso)[1,2-*d*]imidazolidine-2-one (1) was synthesized (Arecas, Avalos, Babiano, González, Jiménez & Palacios, 1992) by the reaction of 2,5-diphenyl-(3,5,6-tri-*O*-acetyl-1,2-dideoxy- α -D-glucofuranoso)[1,2:4,5]-4*aH*,4*bH*-imidazo[2,1-*b*]thiazolium-3-olate (Arecas, Avalos, Babiano, González, Jiménez, Palacios & Pilo, 1991) with 4-(*N,N*-dimethylamino)benzaldehyde. Compound (1) and related compounds are of interest as intermediates in the synthesis of chiral synthons and also because of the molecular overcrowding and the effect it has on geometrical parameters.

In order to elucidate the molecular conformation and the orientations of the substituents at the bond-forming sites, the structure of the product was determined by a single-crystal X-ray analysis.

An ORTEP drawing (Johnson, 1976) of the molecule is shown in Fig. 1. In the thiirane ring, the C—C distance of 1.510 (5) Å is longer than that found (1.468 Å) for the analogous bond in (2*S*,3*S*)-1-cyano-2-hydroxy-3,4-epithiobutane- α -naphthylurethane (Bates, Grady & Sneath, 1972). In thiirane itself (Cunningham, Boyd, Myers & Gwinn, 1951), the corresponding bond length is 1.492 Å. The C—S bonds have more normal lengths of 1.832 (5) and 1.804 (4) Å. In the above 3,4-epithiobutane- α -naphthylurethane (Bates *et al.*, 1972), the two C—S distances are 1.802 and 1.815 Å. In 1,3,5,7-tetrathiocane (Frank & Degen, 1973), the average C—S length is 1.817 Å, and in thiirane the experimental (Cunningham *et al.*, 1951) and calculated value (Rohmer & Roos, 1975) is 1.819 Å. However, C—S distances as long as 1.84–1.85 Å have been found, *e.g.* in dibenzyl disulfide (Lee & Bryant, 1969) and in *L*- α -(*p*-chlorobenzenesulphonamido)- β -propiionothiolactone (Milinovic & Bezjak, 1973).

Bond lengths and angles in the furanosyl ring agree with the mean values reported for these compounds. The asymmetry of the endocyclic bonds [O—C1' = 1.415 (5) and O—C4' = 1.442 (5) Å] clearly shows the anomeric effect. The furanosyl ring is not planar and in terms of ring-puckering coordinates (Cremer & Pople, 1975) the amplitude–phase magnitudes are $Q = 0.38$ (1) Å and $\varphi = -59.4$ (6)° for the sequence O—C1'—C2'—C3'—C4', and the resulting conformation is twist *T*₄³ with C₂ symmetry through the C1' atom [Nardelli (1983) asymmetry parameter $\Delta C_2(C1') = 0.018$ (1)].

Bond lengths and angles in the imidazolidine ring are normal. The bond lengths N1—C1' 1.454 (5) and N3—C2' 1.455 (4) Å, and the O2—C2 length of 1.203 (5) Å clearly indicate double-bond character as found in analogous compounds (Conde, Bernier & Márquez, 1980). This partial double-bond character reflects the resonance of the urea system. The remaining bond lengths and angles have expected values within the limits of experimental error. The imidazolidine-2-one group is corrugated (maximum deviation from the best plane is 0.140 Å). The imidazolidine substituents C71 and C1'' are at deviations of 0.123 and -0.067 Å, respectively.

Evidence for steric hindrance is seen in the short intramolecular contacts (Table 3). These may be

responsible for some deviation from planarity of the imidazolidine and phenyl rings. Atoms of the phenyl ring C71–C76 are coplanar within 0.004 Å; those of the C81–C86 and C91–C96 rings are coplanar within 0.007 and 0.016 Å, respectively.

The dihedral angles between the best planes through different rings are: thiirane–phenyl (C81–C86) 62.3 (2), thiirane–phenyl (C91–C96) 78.4 (2), thiirane–imidazolidine 41.6 (2) and furanosyl ring–imidazolidine 71.6 (1)°.

Intermolecular contacts less than 3.5 Å between non-H atoms include C21...O61' (at $-x, \frac{1}{2} + y, -z$) 3.25, C76...O1'' (at $-x, -\frac{1}{2} + y, -z$) 3.30, C93...O51' (at $-x, y + \frac{1}{2}, -z + 1$) 3.49, C32'...O2 (at $x + 1, y, z$) 3.43 and C91...O1'' (at $x + 1, y, z + 1$) 3.24 Å.

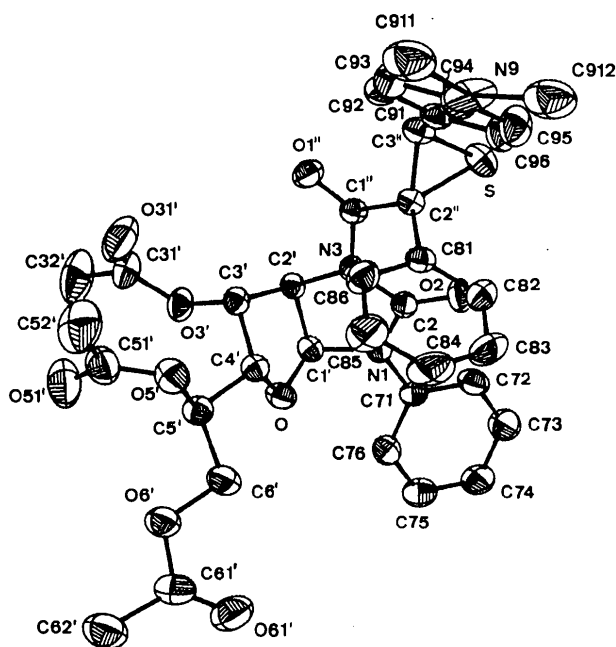


Fig. 1. An ORTEP drawing of the title molecule.

Experimental

Crystal data

C₃₆H₃₇N₃O₉S

M_r = 687.76

Monoclinic

*P*2₁

a = 11.441 (4) Å

b = 16.666 (1) Å

c = 10.251 (3) Å

β = 115.34 (1)°

V = 1766.6 (8) Å³

Z = 2

D_x = 1.293 Mg m⁻³

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 5–12.5°

μ = 0.141 mm⁻¹

T = 293 K

0.48 × 0.32 × 0.25 mm

Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer

ω–2θ scans

Absorption correction: empirical (*DIFABS*;

Walker & Stuart, 1983)

*T*_{min} = 0.65, *T*_{max} = 1.32

5067 measured reflections

4830 independent reflections

Refinement

Refinement on *F*

R = 0.04

wR = 0.04

S = 1.16

3259 reflections

442 parameters

H-atom parameters not refined

3259 observed reflections

[*I* > 2σ(*I*)]

*R*_{int} = 0.08

θ_{max} = 26°

h = 0 → 14

k = 0 → 20

l = -14 → 14

3 standard reflections

frequency: 60 min

intensity variation: none

w = 1/σ²(*F*)

(Δ/σ)_{max} = 0.09

Δρ_{max} = 0.35 e Å⁻³

Δρ_{min} = -0.03 e Å⁻³

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
S	0.3469 (1)	0.4367	0.0671 (1)	0.0445 (7)
N1	0.0731 (3)	0.1899 (2)	-0.1465 (3)	0.0321 (13)
C2	0.1514 (4)	0.2527 (2)	-0.0720 (4)	0.0331 (15)
O2	0.2612 (3)	0.2653 (2)	-0.0526 (3)	0.0462 (14)
N3	0.0775 (3)	0.3005 (2)	-0.0213 (3)	0.0302 (12)
O	-0.0589 (3)	0.1358 (2)	-0.0394 (3)	0.0386 (13)
C1'	-0.0516 (3)	0.1911 (2)	-0.1405 (4)	0.0310 (14)
C2'	-0.0576 (3)	0.2761 (2)	-0.0847 (4)	0.0301 (14)
C3'	-0.1059 (3)	0.2603 (2)	0.0307 (4)	0.0317 (14)
C4'	-0.0453 (3)	0.1791 (2)	0.0881 (4)	0.0325 (15)
C5'	-0.1069 (4)	0.1311 (3)	0.1676 (4)	0.0363 (16)
C6'	-0.0356 (4)	0.0529 (3)	0.2238 (5)	0.0448 (19)
O3'	-0.2443 (2)	0.2521 (2)	-0.0457 (3)	0.0430 (13)
C31'	-0.3181 (4)	0.2968 (3)	0.0010 (6)	0.055 (2)
O31'	-0.2749 (4)	0.3383 (3)	0.1036 (5)	0.080 (2)
C32'	-0.4588 (5)	0.2852 (5)	-0.0952 (8)	0.098 (4)
O5'	-0.0896 (3)	0.1803 (2)	0.2912 (3)	0.0481 (14)
C51'	-0.1941 (5)	0.1939 (3)	0.3150 (6)	0.056 (2)
O51'	-0.2996 (4)	0.1682 (3)	0.2403 (4)	0.080 (2)
C52'	-0.1630 (6)	0.2461 (4)	0.4433 (6)	0.083 (3)
O6'	-0.1093 (3)	0.0084 (2)	0.2832 (3)	0.0474 (15)
C61'	-0.0731 (5)	-0.0689 (3)	0.3163 (4)	0.0466 (19)
O61'	0.0165 (4)	-0.0985 (2)	0.3039 (4)	0.0643 (19)
C62'	-0.1643 (6)	-0.1118 (3)	0.3636 (6)	0.065 (3)
C1''	0.1140 (3)	0.3730 (2)	0.0526 (4)	0.0309 (14)
O1''	0.0313 (3)	0.4193 (2)	0.0478 (3)	0.0430 (13)
C2''	0.2559 (3)	0.3861 (2)	0.1492 (4)	0.0303 (14)
C3''	0.2904 (4)	0.4716 (2)	0.1998 (4)	0.0347 (15)
C71	0.1092 (4)	0.1273 (2)	-0.2164 (4)	0.0321 (15)
C72	0.2006 (5)	0.1402 (3)	-0.2699 (5)	0.050 (2)
C73	0.2333 (5)	0.0780 (3)	-0.3377 (6)	0.058 (3)
C74	0.1774 (5)	0.0039 (3)	-0.3521 (5)	0.055 (2)
C75	0.0870 (5)	-0.0085 (3)	-0.2979 (5)	0.050 (2)
C76	0.0532 (4)	0.0525 (3)	-0.2299 (4)	0.0395 (17)
C81	0.3155 (3)	0.3193 (2)	0.2559 (4)	0.0314 (15)
C82	0.4244 (4)	0.2779 (3)	0.2712 (5)	0.0448 (18)
C83	0.4746 (5)	0.2185 (3)	0.3742 (6)	0.060 (2)
C84	0.4160 (5)	0.2001 (3)	0.4638 (6)	0.064 (3)
C85	0.3068 (6)	0.2406 (3)	0.4472 (6)	0.062 (3)
C86	0.2560 (4)	0.3009 (3)	0.3452 (5)	0.0451 (19)
C91	0.3826 (4)	0.4871 (2)	0.3527 (4)	0.0339 (15)
C92	0.3318 (4)	0.5154 (3)	0.4455 (4)	0.0412 (17)
C93	0.4073 (4)	0.5225 (3)	0.5919 (4)	0.0436 (17)

C94	0.5378 (4)	0.5002 (3)	0.6529 (4)	0.0421 (16)	S—C3''—C91—C92	178.4 (4)	C71—N1—C2—O2	-0.4 (6)
C95	0.5895 (4)	0.4741 (3)	0.5585 (5)	0.0485 (18)	S—C2''—C81—C82	-17.8 (5)	C2'—C3'—O3'—C31'	-129.2 (4)
C96	0.5127 (4)	0.4666 (3)	0.4132 (5)	0.0442 (17)	S—C2''—C1''—N3	93.3 (4)	C1''—C2''—C3''—C91	138.0 (4)
N9	0.6132 (4)	0.5036 (3)	0.8007 (4)	0.0657 (19)	O—C4'—C5'—O5'	-179.1 (3)	C81—C2''—C3''—C91	0.4 (5)
C911	0.7506 (5)	0.4988 (6)	0.8575 (6)	0.107 (4)	O—C4'—C5'—C6'	65.0 (4)	C81—C2''—C1''—N3	-52.5 (5)
C912	0.5629 (5)	0.5362 (4)	0.8960 (5)	0.082 (3)	O1''—C1''—C2''—C3''	-19.2 (5)	C1''—C2''—C3''—HC3''	-41.9 (6)
					O2—C2—N3—C1''	3.9 (6)	C81—C2''—C3''—HC3''	-179.5 (4)

Table 2. Bond lengths (Å), bond angles (°) and selected torsion angles (°)

S—C2''	1.804 (4)	O5'—C51'	1.338 (7)
S—C3''	1.832 (5)	C1''—O1''	1.205 (5)
N1—C2	1.376 (4)	C1''—C2''	1.512 (4)
N1—C1'	1.454 (5)	C2''—C3''	1.510 (5)
N1—C71	1.424 (5)	C2''—C81	1.503 (5)
C2—O2	1.203 (5)	C3''—C91	1.490 (4)
C2—N3	1.412 (5)	C71—C72	1.389 (8)
N3—C2'	1.455 (4)	C71—C76	1.380 (6)
N3—C1''	1.391 (4)	C72—C73	1.385 (8)
O—C1'	1.415 (5)	C73—C74	1.369 (7)
O—C4'	1.442 (5)	C74—C75	1.382 (9)
C1'—C2'	1.539 (5)	C75—C76	1.378 (7)
C2'—C3'	1.528 (6)	C81—C82	1.374 (6)
C3'—C4'	1.519 (5)	C81—C86	1.389 (7)
C3'—O3'	1.441 (4)	C82—C83	1.380 (6)
C4'—C5'	1.513 (6)	C83—C84	1.384 (9)
C5'—C6'	1.513 (6)	C84—C85	1.365 (8)
C5'—O5'	1.450 (5)	C85—C86	1.385 (6)
C6'—O6'	1.439 (6)	C91—C92	1.392 (7)
C31'—O31'	1.175 (7)	C91—C96	1.388 (5)
C31'—C32'	1.497 (6)	C92—C93	1.379 (5)
C51'—O51'	1.198 (6)	C93—C94	1.399 (5)
C51'—C52'	1.487 (8)	C94—C95	1.401 (7)
O6'—C61'	1.350 (5)	C94—N9	1.387 (5)
C61'—O61'	1.192 (7)	C95—C96	1.373 (5)
C61'—C62'	1.506 (9)	N9—C911	1.425 (6)
O3'—C31'	1.357 (7)	N9—C912	1.436 (8)
C2''—S—C3''	49.0 (1)	N3—C1''—O1''	119.0 (3)
C1'—N1—C71	122.1 (3)	O1''—C1''—C2''	122.5 (3)
C2—N1—C71	125.2 (3)	S—C2''—C1''	115.7 (2)
C2—N1—C1'	112.5 (3)	C1''—C2''—C81	113.2 (3)
N1—C2—N3	106.4 (3)	C1''—C2''—C3''	114.2 (3)
N1—C2—O2	127.5 (3)	S—C2''—C81	120.8 (3)
O2—C2—N3	125.9 (3)	S—C2''—C3''	66.4 (2)
C2—N3—C1''	127.0 (3)	C3''—C2''—C81	118.5 (3)
C2—N3—C2'	111.4 (3)	S—C3''—C2''	64.5 (2)
C2'—N3—C1''	120.0 (3)	C2''—C3''—C91	119.2 (3)
C1'—O—C4'	108.6 (2)	S—C3''—C91	121.3 (3)
N1—C1'—O	112.8 (3)	N1—C71—C76	119.3 (3)
O—C1'—C2'	107.5 (2)	N1—C71—C72	120.9 (3)
N1—C1'—C2'	103.1 (3)	C72—C71—C76	119.6 (4)
N3—C2'—C1'	102.4 (3)	C71—C72—C73	119.5 (4)
C1'—C2'—C3'	102.5 (3)	C72—C73—C74	120.9 (5)
N3—C2'—C3'	111.6 (3)	C73—C74—C75	119.1 (4)
C2'—C3'—O3'	105.7 (3)	C74—C75—C76	120.8 (4)
C2'—C3'—C4'	101.3 (3)	C71—C76—C75	119.8 (4)
C4'—C3'—O3'	109.8 (3)	C2''—C81—C86	116.4 (3)
O—C4'—C3'	103.9 (2)	C2''—C81—C82	124.2 (3)
C3'—C4'—C5'	116.0 (3)	C82—C81—C86	119.3 (3)
O—C4'—C5'	109.1 (3)	C81—C82—C83	120.5 (4)
C4'—C5'—O5'	104.4 (3)	C82—C83—C84	120.3 (5)
C4'—C5'—C6'	111.1 (3)	C83—C84—C85	119.0 (5)
C6'—C5'—O5'	107.7 (3)	C84—C85—C86	121.2 (5)
C5'—C6'—O6'	106.6 (3)	C81—C86—C85	119.4 (4)
C3'—O3'—C31'	117.4 (3)	C3''—C91—C96	124.6 (3)
O3'—C31'—C32'	110.6 (4)	C3''—C91—C92	117.6 (3)
O3'—C31'—O31'	123.4 (5)	C92—C91—C96	117.3 (3)
O31'—C31'—C32'	125.9 (5)	C91—C92—C93	121.5 (4)
C5'—O5'—C51'	117.1 (3)	C92—C93—C94	120.9 (4)
O5'—C51'—C52'	111.4 (5)	C93—C94—N9	121.5 (4)
O5'—C51'—O51'	123.8 (5)	C93—C94—C95	117.3 (3)
O51'—C51'—C52'	124.6 (6)	C95—C94—N9	120.9 (4)
C6'—O6'—C61'	115.2 (4)	C94—C95—C96	121.1 (4)
O6'—C61'—C62'	110.7 (4)	C91—C96—C95	120.9 (4)
O6'—C61'—O61'	123.5 (4)	C94—N9—C912	121.0 (4)
O61'—C61'—C62'	125.6 (4)	C94—N9—C911	120.3 (4)
N3—C1''—C2''	118.0 (3)	C911—N9—C912	115.9 (4)

Table 3. Some intramolecular contact distances (Å)

C3'...O31'	2.687 (7)	C72...O2	2.911 (6)
C3'...O1''	3.047 (5)	C76...O	3.086 (6)
C5'...O3'	2.895 (5)	C82...S	3.254 (5)
C5'...O51'	2.684 (7)	C82...O2	3.040 (5)
C6'...O	2.940 (6)	C96...S	3.267 (4)

The intensity data were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *DIRDIF* (Beurskens *et al.*, 1984) and refined by full-matrix least squares with anisotropic displacement parameters for non-H atoms. Calculations were performed with *PARST* (Nardelli, 1983). The y coordinate of the S atom was fixed. The H atoms were placed in calculated positions; their isotropic displacement parameters were not refined. $(\Delta/\sigma)_{\max}$ was 0.18 for C94 and C912.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71441 (39 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1040]

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