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5-Deoxy-5-*C*-(5-ethoxycarbonyl-1,2,3-triazol-1-yl)-1,2-*O*-isopropylidene-α-D-xylofuranose

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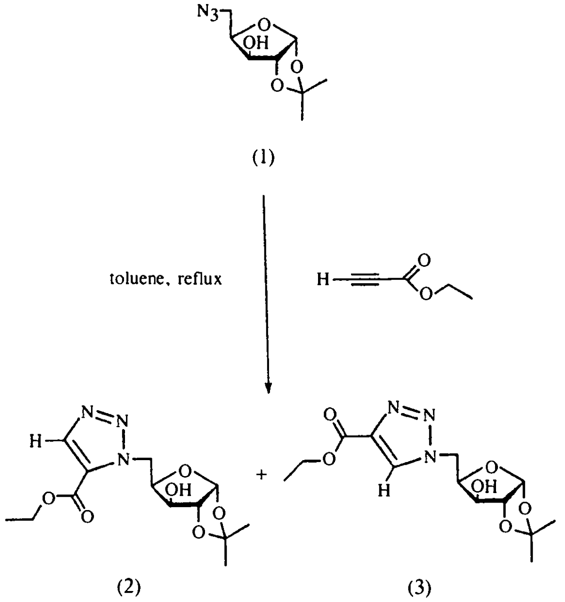
Abstract

Two unrelated molecules of the title compound, ethyl 1-(5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranos-5-*C*-yl)-1,2,3-triazole-5-carboxylate, $C_{13}H_{19}N_3O_6$, that are not linked by hydrogen bonding, comprise the asymmetric unit. There are no unusual bond lengths or angles. The two molecules differ in the degree of rotation around the methylene C atom that joins the triazole ring to the sugar part of the molecule. Molecules of the same conformation form infinite chains joined by hydrogen bonding between a H atom on the hydroxyl group of one molecule and an N atom in the triazole ring of another molecule generated by the 2_1 screw axis. Relevant intermolecular N···O distances are 3.013 (3) and 2.806 (3) Å.

Comment

One aspect of our research (Norris, Horton & Levine, 1996) is the addition reaction of sugar-derived azides to alkynes. Such addition reactions generally lead to two products, *e.g.* (2) and (3) (see scheme below). We believe that the major product of this reaction is (3), but a considerable amount of another product (26% yield), which revealed spectroscopic properties almost identical to those of (3), was obtained (Norris, Horton & Levine, 1996). We suspected that the identity of the minor product was indeed (2) and thus undertook an X-ray diffraction study of (2) with the aim of providing proof of the structure of this compound.



Compound (2) crystallizes with two independent molecules comprising the asymmetric unit. *ORTEP*II (Johnson, 1976) representations of the two molecules (forms 1 and 2) and their atomic labelling schemes are shown in Figs. 1 and 2. The bond lengths and valence angles given in Table 2 are all within the ranges expected for this type of compound. The absolute configuration was assigned to agree with the known chirality of the sugar moiety (*i.e.* α-D-xylofuranose). In fact, this rules out the possibility of the molecule being arranged around a center of inversion since this would result in an inversion at all of the chiral centers in the molecule.

Molecules of the same conformation form infinite chains joined by hydrogen bonding between a H atom on the hydroxyl group of one molecule and an N atom in the triazole ring of another molecule generated by the 2_1 screw axis. The O—H distances in the two hydroxyl groups are similar; O14—H119 0.83 (3) and O24—H219 0.84 (5) Å. The two hydrogen-bond distances are slightly different, however; H119···N13 2.19 (6) and H229···N23 1.99 (5) Å. This may be related to differences in the shape of the two conformers.

There are also some significant differences in the bond distances between the two solid-state forms of (2). For example, the bond lengths O11—C14 of 1.435 (6) and N11—C15 of 1.470 (4) Å in form 1 of compound (2) are significantly longer than the equivalent ones in form 2, namely, O21—C24 of 1.418 (4) and N21—C25 of 1.447 (4) Å. Furthermore, there are also significant differences in some bond

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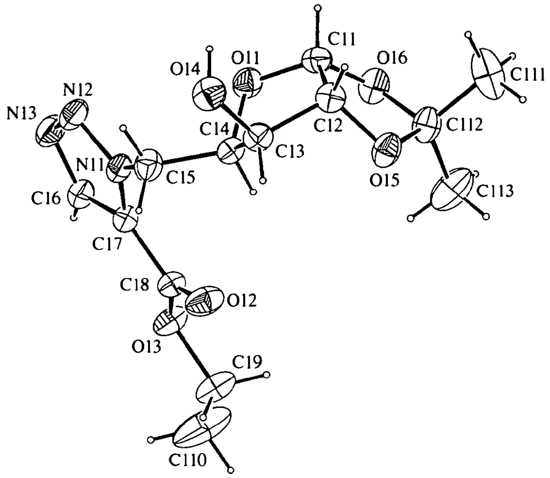

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of form 1 of compound (2) showing 50% probability displacement ellipsoids. H atoms are shown as arbitrarily sized uniform circles.

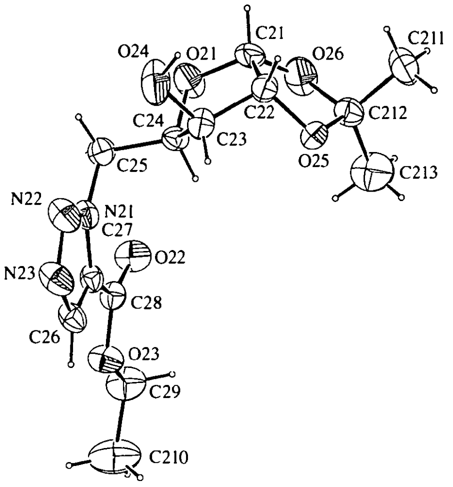

Fig. 2. *ORTEP*II (Johnson, 1976) drawing of form 2 of compound (2) showing 50% probability displacement ellipsoids. H atoms are shown as arbitrarily sized uniform circles.

angles, specifically O14—C13—C14 of 112.9 (2) *versus* O24—C23—C24 of 108.8 (3)°, C13—C14—C15 of 113.5 (2) *versus* C23—C24—C25 of 115.1 (3)° and N11—C15—C14 of 114.3 (2) *versus* N21—C25—C24 of 110.1 (3)°. These differences are also probably related to the different arrangement of the sugar and triazole ring moieties in the two molecules around the equivalent methylene C15 and C25 atoms (in forms 1 and 2, respectively).

Experimental

Crystals of (2) were obtained by slow crystallization from an ethyl acetate solution of compound (1).

Crystal data

$C_{13}H_{19}N_3O_6$
$M_r = 313.315$
Monoclinic
$P2_1$
$a = 9.493\,(1)$ Å
$b = 11.426\,(1)$ Å
$c = 14.932\,(2)$ Å
$\beta = 102.77\,(1)°$
$V = 1579.5\,(5)$ Å^3
$Z = 2$
$D_x = 0.658$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 15 reflections
$\theta = 12\text{–}21°$
$\mu = 0.45$ mm^{-1}
$T = 296$ K
Irregular
0.40 × 0.37 × 0.35 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega\text{–}2\theta$ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.693$, $T_{\max} = 0.743$
3591 measured reflections

3591 independent reflections
3379 observed reflections [$F > 3\sigma(F)$]
$\theta_{\max} = 74°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 14$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 250 min
intensity decay: 1.8%

Refinement

Refinement on F
$R = 0.052$
$wR = 0.060$
$S = 2.19$
3379 reflections
404 parameters
H atoms: see below
$w = 1/[\sigma^2(F) + 0.0004F^2]$
$(\Delta/\sigma)_{\max} = 0.25$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)
Absolute configuration: assigned to agree with the known chirality of the sugar moiety

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O11	0.3064 (2)	0.3906 (2)	0.9208 (1)	0.0513 (4)
O12	−0.0222 (2)	0.6358 (2)	0.8573 (1)	0.0659 (5)
O13	0.0131 (2)	0.7570 (2)	0.7464 (1)	0.0594 (5)
O14	0.2684 (2)	0.3652 (2)	1.1103 (1)	0.0593 (5)
O15	0.0384 (2)	0.2201 (2)	0.9165 (1)	0.0572 (5)
O16	0.1956 (2)	0.2421 (2)	0.8249 (1)	0.0583 (5)
N11	0.2885 (2)	0.644	0.9297 (1)	0.0460 (5)
N12	0.4288 (2)	0.6719 (2)	0.9447 (2)	0.0574 (5)
N13	0.4442 (2)	0.7493 (2)	0.8827 (2)	0.0607 (6)
C11	0.2753 (2)	0.2702 (2)	0.9132 (2)	0.0475 (5)
C12	0.1729 (3)	0.2430 (2)	0.9773 (2)	0.0480 (5)
C13	0.1646 (2)	0.3583 (2)	1.0265 (1)	0.0465 (5)
C14	0.1927 (2)	0.4453 (2)	0.9550 (1)	0.0433 (5)
C15	0.2418 (3)	0.5645 (2)	0.9947 (2)	0.0518 (6)
C16	0.3146 (2)	0.7706 (2)	0.8277 (2)	0.0496 (5)
C17	0.2127 (2)	0.7041 (2)	0.8563 (2)	0.0427 (5)
C18	0.0549 (2)	0.6935 (2)	0.8211 (2)	0.0464 (5)
C19	−0.1422 (3)	0.7510 (4)	0.7038 (2)	0.0747 (9)
C110	−0.1749 (4)	0.8360 (6)	0.6298 (3)	0.133 (1)
C111	0.0921 (5)	0.0518 (3)	0.8315 (2)	0.095 (1)
C112	0.0671 (3)	0.1821 (3)	0.8310 (2)	0.0570 (6)
C113	−0.0524 (4)	0.2229 (5)	0.7552 (2)	0.100 (1)

O21	0.3955 (2)	0.8576 (2)	0.5025 (1)	0.0892 (5)
O22	0.5640 (2)	0.6102 (2)	0.6759 (1)	0.0714 (6)
O23	0.5171 (2)	0.4774 (2)	0.7759 (1)	0.0664 (5)
O24	0.0894 (3)	0.8146 (3)	0.4658 (2)	0.1241 (8)
O25	0.2791 (2)	1.0039 (2)	0.6484 (1)	0.0528 (4)
O26	0.4637 (2)	1.0305 (2)	0.5795 (2)	0.0889 (6)
N21	0.2633 (2)	0.5843 (2)	0.5801 (2)	0.0519 (5)
N22	0.1231 (3)	0.5551 (2)	0.5586 (2)	0.0722 (8)
N23	0.0985 (3)	0.4877 (3)	0.6253 (2)	0.0762 (8)
C21	0.3578 (4)	0.9745 (3)	0.5145 (2)	0.0822 (9)
C22	0.2237 (3)	0.9722 (3)	0.5556 (2)	0.0600 (6)
C23	0.1799 (3)	0.8439 (3)	0.5504 (2)	0.0564 (6)
C24	0.3245 (3)	0.7867 (2)	0.5568 (2)	0.0467 (5)
C25	0.3188 (3)	0.6627 (3)	0.5200 (2)	0.0580 (6)
C26	0.2215 (3)	0.4731 (2)	0.6882 (2)	0.0584 (6)
C27	0.3290 (2)	0.5343 (2)	0.6613 (2)	0.0467 (5)
C28	0.4832 (2)	0.5466 (2)	0.7040 (2)	0.0490 (6)
C29	0.6671 (3)	0.4818 (4)	0.8265 (2)	0.083 (1)
C210	0.6817 (4)	0.3992 (6)	0.9038 (3)	0.119 (1)
C211	0.3629 (5)	1.2008 (4)	0.6356 (3)	0.104 (1)
C212	0.4030 (3)	1.0738 (3)	0.6519 (2)	0.0621 (8)
C213	0.5059 (4)	1.0528 (5)	0.7431 (3)	0.112 (1)

Table 2. *Selected geometric parameters (Å, °)*

O11—C11	1.406 (4)	O21—C21	1.404 (5)
O11—C14	1.435 (3)	O21—C24	1.417 (3)
O14—C13	1.414 (3)	O24—C23	1.402 (4)
N11—N12	1.338 (3)	N21—N22	1.340 (4)
N11—C15	1.471 (4)	N21—C25	1.447 (4)
N11—C17	1.355 (3)	N21—C27	1.360 (3)
N12—N13	1.312 (4)	N22—N23	1.320 (4)
N13—C16	1.343 (4)	N23—C26	1.337 (4)
C11—C12	1.540 (4)	C21—C22	1.532 (6)
C12—C13	1.519 (4)	C22—C23	1.521 (5)
C13—C14	1.524 (4)	C23—C24	1.505 (4)
C14—C15	1.517 (4)	C24—C25	1.516 (4)
C16—C17	1.370 (4)	C26—C27	1.369 (4)
C11—O11—C14	107.1 (2)	C21—O21—C24	107.8 (2)
N12—N11—C15	118.0 (2)	N22—N21—C25	119.0 (3)
N12—N11—C17	110.4 (2)	N22—N21—C27	110.1 (3)
C15—N11—C17	131.5 (2)	C25—N21—C27	130.9 (2)
N11—N12—N13	107.6 (2)	N21—N22—N23	107.2 (2)
N12—N13—C16	108.9 (2)	N22—N23—C26	109.3 (3)
O11—C11—C12	107.3 (2)	O21—C21—C22	107.0 (3)
C11—C12—C13	103.4 (2)	C21—C22—C23	103.8 (3)
O14—C13—C12	112.1 (2)	O24—C23—C22	112.4 (3)
O14—C13—C14	112.8 (2)	O24—C23—C24	108.7 (3)
C12—C13—C14	101.1 (2)	C22—C23—C24	100.4 (3)
O11—C14—C13	103.1 (2)	O21—C24—C23	104.9 (2)
O11—C14—C15	109.9 (2)	O21—C24—C25	108.2 (2)
C13—C14—C15	113.4 (2)	C23—C24—C25	115.1 (3)
N11—C15—C14	114.3 (2)	N21—C25—C24	110.1 (2)
N13—C16—C17	108.9 (3)	N23—C26—C27	108.8 (3)
N11—C17—C16	104.3 (2)	N21—C27—C26	104.5 (2)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O14—H119···N13^{i}	0.83 (6)	2.19 (6)	3.013 (3)	175 (5)
O24—H219···N23^{ii}	0.84 (5)	1.99 (5)	2.806 (3)	165 (4)

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

All H atoms, except those attached to O atoms, were fixed at ideal positions (C—H distances set at 0.95 Å), with common isotropic displacement parameters fixed at $1.3B_{eq}$ of the atoms to which they are attached. From the systematic absences of $0k0$ and from subsequent least-squares refinement, the space group was determined to be $P2_1$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *MolEN*. Program(s) used to refine structure: *MolEN*. Molecular graphics: *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1203). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Polymorph of Bis(2-nitrophenyl) Trisulfide

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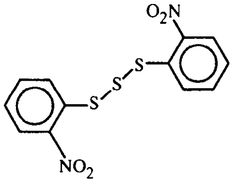
Abstract

Unlike the triclinic form, a molecular twofold axis in the orthorhombic form of bis(2-nitrophenyl) trisulfide, $C_{12}H_8N_2O_4S_3$, corresponds with the crystallographic symmetry.

Comment

A crystallographic study of the triclinic form of bis(2-nitrophenyl) trisulfide, (I), including details of its non-bonded S···O interactions has been reported previously (Howie & Wardell, 1996).



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