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Benzaldehyde 1-(2,3,4-tri-*O*-acetyl-*β*-D-xylopyranosyl)thiosemicarbazone

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.115
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{19}H_{23}N_3O_7S$, the hexopyranosyl ring adopts a chair conformation. All the substituents are in equatorial positions. The molecules are linked by C—H$\cdots$S interactions into ribbons, which are connected into a two-dimensional framework *via* intermolecular C—H$\cdots$O short contacts.

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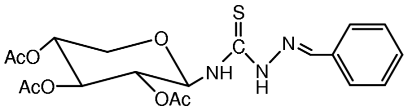
Comment

We have reported the crystal structure of *O*-ethyl *N*-(2,3,4-tri-*O*-acetyl-*β*-D-xylopyranosyl)thiocarbamate (Yang *et al.*, 2004) and 4-methoxybenzaldehyde 1-(2,3,4-tri-*O*-acetyl-*β*-D-xylopyranosyl)thiosemicarbazone (Zhang *et al.*, 2004). As part of our ongoing research on carbohydrate chemistry, the title compound, (I), was synthesized. An X-ray crystallographic analysis was undertaken to elucidate its molecular configuration.



(I)

In (I), the bond lengths and angles of the xylopyranosyl ring are in good agreement with those observed in the related compound *O*-ethyl *N*-(2,3,4-tri-*O*-acetyl-*β*-D-xylopyranosyl)thiocarbamate (Yang *et al.*, 2004). The hexopyranosyl ring adopts a chair conformation (Fig. 1), with atoms C2 and C5 deviating by 0.717 (2) and −0.633 (8) Å, respectively, from the mean plane through the other atoms. All three acetyl groups are individually planar and occupy equatorial positions. Atom S1 is in a synperiplanar position with respect to atom C5, the

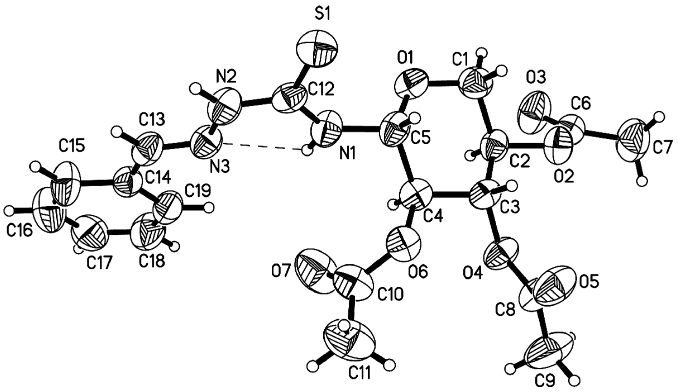


Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The dashed line indicates a hydrogen bond.

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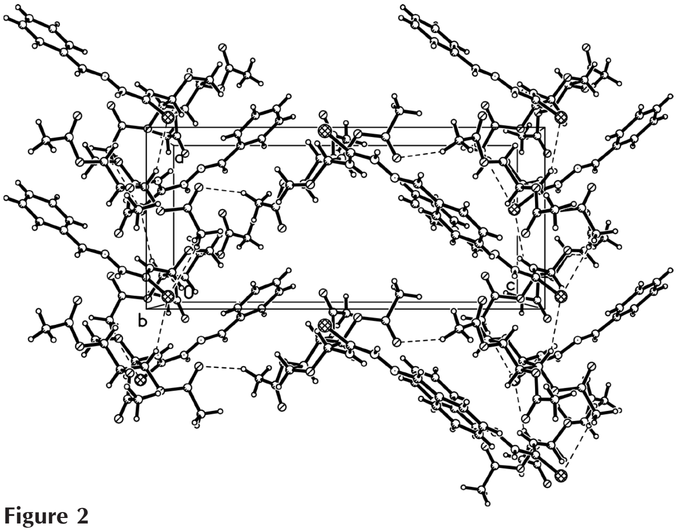

Figure 2
Packing diagram of the title compound, showing the formation of ribbons along the *a* axis. Dashed lines indicate hydrogen bonds.

C5—N1—C12—S1 torsion angle being 0.0 (5)°, while atom N2 is in an antiperiplanar position, with a C5—N1—C12—N2 torsion angle of −179.5 (3)°.

The thiosemicarbazone moiety is almost planar, due to the C═N double bond. Meanwhile, atoms N1 and N3 are involved in an intramolecular N1—H1*A*···N3 interaction, which also contributes to the planarity of the thiosemicarbazone group. The molecules are linked by C—H···S interactions (Table 2) into ribbons, which are connected into a two-dimensional framework (Fig. 2) *via* intermolecular C—H···O short contacts (Table 2).

Experimental

For the preparation of *N*-amino-*N*′-(2,3,4-tri-*O*-acetyl-*β*-D-xylopyranosyl)thiourea, ethanol (100 ml) and hydrazine monohydrate (50% aqueous solution, 1.2 ml) were mixed below 268 K in an ice bath. 2,3,4-Tri-*O*-acetyl-*β*-D-*xylo*pyranosyl isothiocyanate (3.2 g, 10 mmol) in ethanol (50 ml) was added dropwise with stirring. The solution was filtered after stirring for 40 min. Colorless crystals were obtained by recrystallization from ethyl acetate/petroleum ether (1:3). For the preparation of the title compound, (I), tetrahydrofuran (20 ml), *N*-amino-*N*′-(2,3,4-tri-*O*-acetyl-*β*-D-*xylo*pyranosyl)thiourea (4 mmol) and benzaldehyde (4 mmol) were mixed with stirring under reflux in an oil bath for 5 h. After the contents had been concentrated *in vacuo*, deposits were obtained, which were recrystallized from ethyl acetate/petroleum ether (2:3). The title compound was dissolved in the above solvent and, after filtration, the colorless filtrate was left at room temperature. Single crystals suitable for X-ray crystallographic analysis were obtained.

Crystal data

$C_{19}H_{23}N_3O_7S$
$M_r = 437.46$
Orthorhombic, $P2_12_12_1$
$a = 8.8789\ (9)$ Å
$b = 12.6890\ (13)$ Å
$c = 19.506\ (2)$ Å
$V = 2197.6\ (4)$ Å^3
$Z = 4$
$D_x = 1.322$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 3603 reflections
$\theta = 1.5$–25.0°
$\mu = 0.19$ mm^{-1}
$T = 293\ (2)$ K
Block, colorless
0.48 × 0.39 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.914$, $T_{\max} = 0.957$
8924 measured reflections
3873 independent reflections
3603 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 25.0°$
$h = -10 \rightarrow 10$
$k = -15 \rightarrow 13$
$l = -17 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.115$
$S = 1.20$
3873 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.3313P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}
Absolute structure: Flack (1983), 1652 Friedel pairs
Flack parameter = 0.05 (10)

Table 1
Selected geometric parameters (Å, °).

S1—C12	1.675 (3)	C2—C3	1.501 (4)
O1—C5	1.415 (4)	C3—C4	1.506 (4)
O1—C1	1.416 (4)	C4—C5	1.526 (4)
C1—C2	1.519 (4)		
C6—O2—C2—C3	150.6 (3)	C10—O6—C4—C3	−133.5 (3)
C6—O2—C2—C1	−90.7 (3)	C10—O6—C4—C5	104.8 (3)
C8—O4—C3—C2	136.6 (3)	C5—N1—C12—N2	−179.5 (3)
C8—O4—C3—C4	−106.3 (3)	C5—N1—C12—S1	0.0 (5)

Table 2
Short intra- and intermolecular contacts (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N3	0.86	2.15	2.579 (4)	110
C4—H4*A*···S1$^{\rm i}$	0.98	2.76	3.633 (3)	148
C7—H7*C*···O7$^{\rm ii}$	0.96	2.54	3.409 (5)	151
C11—H11*B*···S1$^{\rm iii}$	0.96	2.85	3.553 (4)	131

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

All H atoms were positioned geometrically and treated as riding, with C—H distances of 0.93–0.98 Å and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ [for the methyl H atoms, $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$].

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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