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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.063
 wR factor = 0.132
Data-to-parameter ratio = 15.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Benzaldehyde 1-(2,3,4,6-tetra-*O*-acetyl- β -D-galactopyranosyl)thiosemicarbazone

In the title compound, $\text{C}_{22}\text{H}_{27}\text{N}_3\text{O}_9\text{S}$, the six-membered pyranosyl ring adopts a chair conformation. The acetyl group opposite the thiosemicarbazone substituent occupies an axial position, while all other substituents are in equatorial positions. The molecules are linked by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds into chains parallel to the b axis, and two additional $\text{C}-\text{H}\cdots\text{O}$ interactions provide further stability in a three-dimensional network.

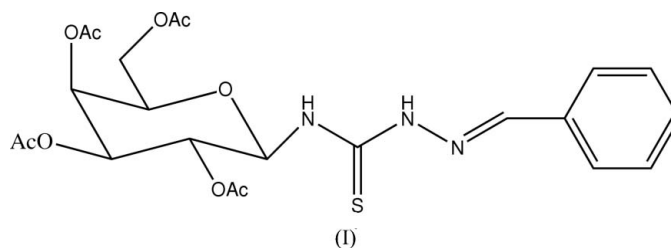
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Comment

In the title compound, (I), the bond lengths and angles of the xylopyranosyl ring are comparable to those in the related compounds 4-methoxybenzaldehyde 4-(2,3,4,6-tetra-*O*-acetyl- β -D-glucopyranosyl)thiosemicarbazone (Zhang *et al.*, 2004) and benzaldehyde 1-(2,3,4-tri-*O*-acetyl- β -D-xylopyranosyl)thiosemicarbazone (Yang *et al.*, 2004). The acetyl group at atom C4 occupies an axial position, while all other substituents are in equatorial positions. The pyranosyl ring adopts a chair conformation, and atom S1 is in a synperiplanar position with respect to atom C1, the C1–N1–C15–S1 torsion angle being $8.7(6)^\circ$.



In the crystal structure of (I), the molecules are linked by $\text{C11}-\text{H11B}\cdots\text{O5}^{\text{ii}}$ hydrogen bonds (Table 2) into chains parallel to the b axis (Fig. 2). The packing is further stabilized by two additional $\text{C7}-\text{H7B}\cdots\text{O10B}^{\text{i}}$ and $\text{C16}-\text{H16A}\cdots\text{O9}^{\text{i}}$ interactions (Table 2), giving a three-dimensional framework.

Experimental

The title compound was prepared according to the method described by Zhang *et al.* (2004).

Crystal data

$\text{C}_{22}\text{H}_{27}\text{N}_3\text{O}_9\text{S}$
 $M_r = 509.53$
Monoclinic, $P2_1$
 $a = 11.547(5)$ Å
 $b = 9.113(4)$ Å
 $c = 12.801(5)$ Å
 $\beta = 101.651(8)^\circ$
 $V = 1319.3(9)$ Å³
 $Z = 2$

$D_x = 1.283$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 1389
reflections
 $\theta = 2.6-19.0^\circ$
 $\mu = 0.18$ mm⁻¹
 $T = 293(2)$ K
Needle, colorless
 $0.55 \times 0.08 \times 0.08$ mm

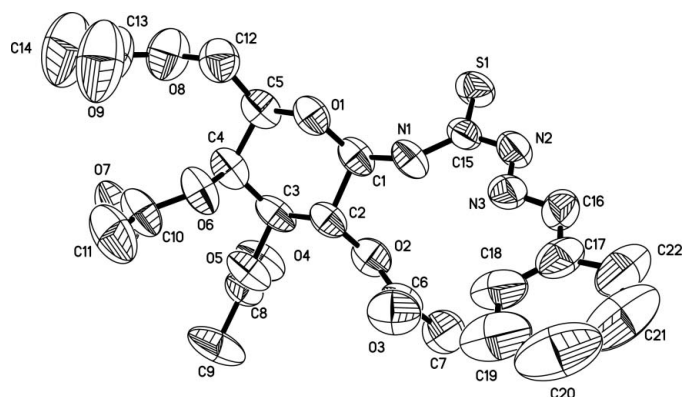


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

Data collection

Siemens SMART CCD area detector diffractometer	5040 independent reflections
ω scans	3120 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.030$
$T_{\text{min}} = 0.910$, $T_{\text{max}} = 0.986$	$\theta_{\text{max}} = 26.2^\circ$
7435 measured reflections	$h = -7 \rightarrow 14$
	$k = -11 \rightarrow 11$
	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.063$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.132$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.19 \text{ e } \text{\AA}^{-3}$
5040 reflections	$\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{\AA}^{-3}$
316 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	2229 Friedel pairs
	Flack parameter: 0.06 (11)

Table 1
Selected geometric parameters (\AA , $^\circ$).

S1—C15	1.665 (4)	N1—C1	1.429 (5)
O1—C5	1.417 (5)	N2—C15	1.352 (5)
O1—C1	1.420 (5)	N2—N3	1.373 (5)
N1—C15	1.332 (5)	N3—C16	1.274 (5)
C1—N1—C15—N2	-170.6 (4)	C1—N1—C15—S1	8.7 (6)

Table 2
Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7B \cdots O1 ⁱ	0.96	2.53	3.477 (6)	169
C11—H11B \cdots O5 ⁱⁱ	0.96	2.45	3.300 (7)	147
C16—H16A \cdots O9 ⁱ	0.93	2.45	3.244 (8)	143

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

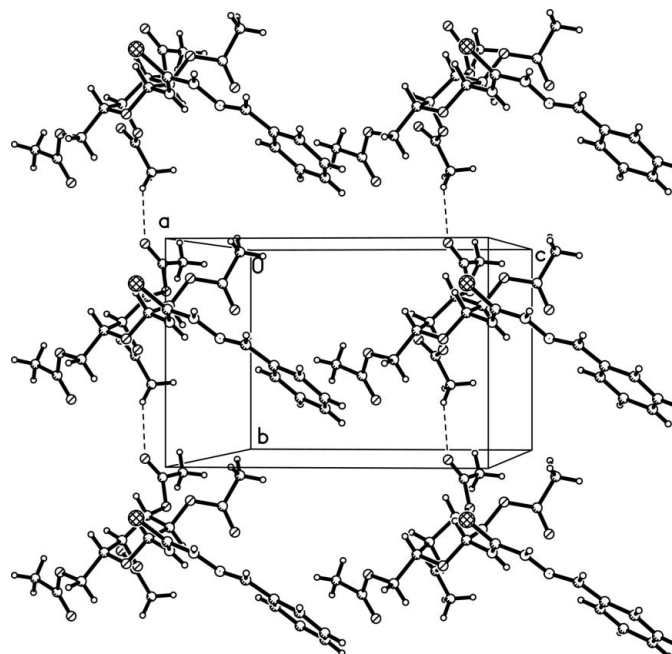


Figure 2
Packing diagram of (I), showing the formation of chains along the b axis. Dashed lines indicate C—H \cdots O hydrogen bonds.

All H atoms were positioned geometrically and treated as riding, with C—H = 0.93–0.98 \AA , N—H = 0.86 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$, or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl H atoms.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

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