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4-Methoxybenzaldehyde 4-(2,3,4,6-tetra-*O*-acetyl-*β*-D-glucopyranosyl)thiosemicarbazone

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Key indicators

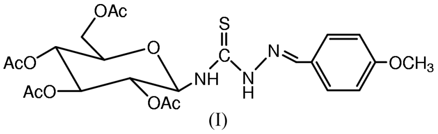
Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.072
wR factor = 0.216
Data-to-parameter ratio = 18.3

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

In the title compound, $C_{23}H_{29}N_3O_{10}S$, the hexapyranosyl ring adopts a chair conformation with all the substituents in equatorial positions. The molecules are linked by C—H···S interactions into ribbons parallel to the *a* axis and two additional intermolecular C—H···O interactions provide further stability in a three-dimensional network.

Comment

Studies of carbohydrate-containing compounds have increased significantly in the last three decades, as they have been found to have many interesting and useful biological properties in addition to providing structural support and energy-storing functions (Mao *et al.*, 1995). *N*,*N*′-Disubstituted thioureas are known to exhibit antiviral and herbicidal activities and to be active against tuberculosis (Li *et al.*, 2001). We have reported the structures of *O*-methyl and *O*-ethyl *N*-(2,3,4,6-tetra-*O*-acetyl-*β*-D-glucopyranosyl)thiocarbamate (Zhang *et al.*, 2001) and *N*-(2,3,4,6-tetra-*O*-acetyl-*β*-D-glycosyl)thiocarbamic benzoylhydrazine (Zhang *et al.*, 2002). The title compound, (I), was prepared and its structure is reported here.



(I)

In (I), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and are comparable with those in the related compounds *O*-ethyl and *O*-methyl *N*-(2,3,4,6-tetra-*O*-acetyl-*β*-D-glucopyranosyl)thiocarbamate (Zhang *et al.*, 2001) and *O*-methyl *N*-(2,3,4-tri-*O*-acetyl-*β*-D-xylopyranosyl)thiocarbamate (Yang *et al.*, 2004). The hexapyranosyl ring adopts a chair conformation (Fig. 1), with atoms C10 and C13 deviating by −0.690 (1) and 0.695 (1) Å, respectively, from the mean plane through atoms C11, C12, C14 and O8.

The thiosemicarbazone moiety is almost planar, the largest deviation from the N1–N3/C8/C9 plane being 0.037 (3) Å for atom S1. The aromatic ring (C1–C6) is twisted 21.6 (2)° from this thiosemicarbazone plane. Each substituent attached to the hexapyranosyl ring is planar, except for the acetoxyl fragment connected to atom C21. Atom O10 is disordered over two positions, with refined site occupancies of 0.47 (4) for O1*A* and 0.53 (4) for O1*B*. These O atoms (O10*A* and O10*B*) are displaced in opposite directions from the O9/C21—C23 plane.

In the crystal structure of (I), the molecules are linked by C11—H11*A*···S1 and C16—H16*B*···S2 interactions into ribbons parallel to the *a* axis (Fig. 2). The packing is further stabilized by two additional C18—H18*C*···O10*B* and C20—

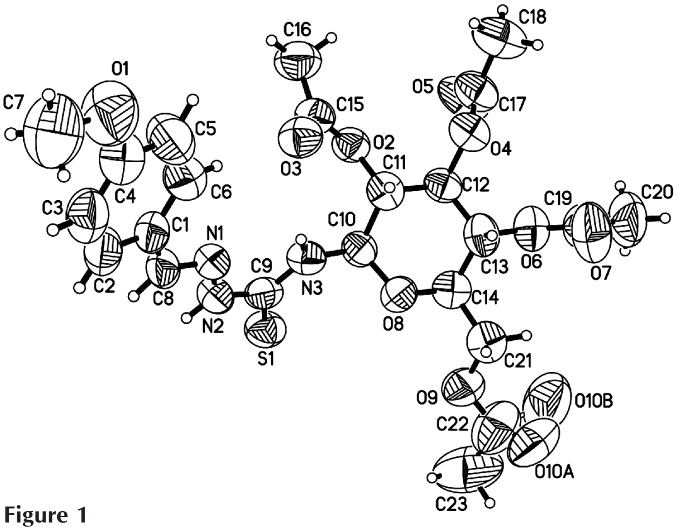


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as small spheres of arbitrary radius.

H20*A*···O3 interactions (Table 2), giving a three-dimensional framework.

Experimental

For the preparation of the title compound, (I), solutions in DMF of *N*-amino-*N*′-(2,3,4,6-tetra-*O*-acetyl-*β*-D-glucopyranosyl)thiourea (4 mmol) and 4-methoxybenzaldehyde (4 mmol) were mixed and stirred under reflux in an oil bath for 2 h. After filtration, the clear colourless filtrate was left at room temperature and yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{23}H_{29}N_3O_{10}S$
$M_r = 539.55$
Orthorhombic, $P2_12_12_1$
$a = 8.7678\ (5)$ Å
$b = 17.6042\ (9)$ Å
$c = 17.9730\ (9)$ Å
$V = 2774.1\ (3)$ Å^3
$Z = 4$
$D_x = 1.292$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6318 reflections
$\theta = 1.5$–27.4°
$\mu = 0.17$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
0.24 × 0.14 × 0.13 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.960$, $T_{\max} = 0.978$
35973 measured reflections
6318 independent reflections
2708 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.078$
$\theta_{\max} = 27.4^\circ$
$h = -11 \rightarrow 11$
$k = -22 \rightarrow 22$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.072$
$wR(F^2) = 0.216$
$S = 0.98$
6318 reflections
345 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1011P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0038 (12)
Absolute structure: Flack (1983), 2757 Friedel pairs
Flack parameter = 0.14 (15)

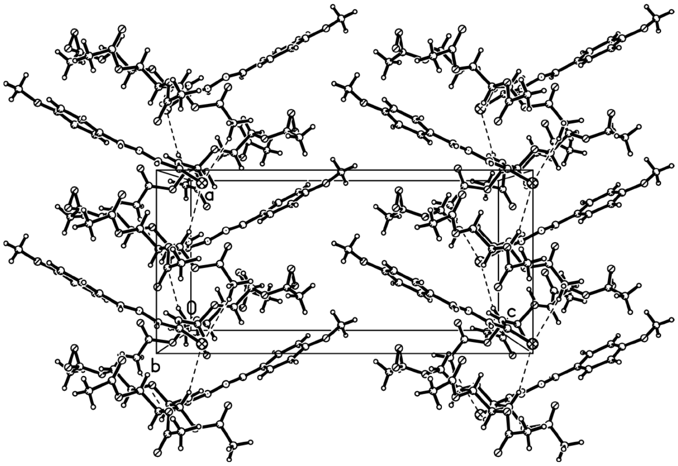


Figure 2
Packing diagram of the title compound, showing the formation of ribbons through hydrogen bonds (dashed lines) along the *a* axis.

Table 1
Selected geometric parameters (Å, °).

S1—C9	1.646 (5)	C11—C12	1.501 (6)
O8—C14	1.427 (6)	C12—C13	1.505 (7)
O8—C10	1.438 (5)	C13—C14	1.510 (7)
C10—C11	1.523 (7)		
C10—N3—C9—N2	179.4 (4)	C17—O4—C12—C13	125.5 (5)
C10—N3—C9—S1	−0.7 (7)	C19—O6—C13—C12	122.8 (5)
C15—O2—C11—C12	−139.2 (4)	C19—O6—C13—C14	−119.3 (6)
C15—O2—C11—C10	101.7 (5)	O8—C14—C21—O9	77.0 (5)
C17—O4—C12—C11	−113.8 (5)	C13—C14—C21—O9	−164.0 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*A*···S1$^{\rm i}$	0.98	2.77	3.713 (5)	162
C16—H16*B*···S1$^{\rm ii}$	0.96	2.85	3.682 (6)	146
C18—H18*C*···O10*B*$^{\rm iii}$	0.96	2.50	3.12 (3)	123
C20—H20*A*···O3$^{\rm iv}$	0.96	2.44	3.240 (9)	140

Symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, -z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, -z$; (iii) $\frac{5}{2}-x, -y, z-\frac{1}{2}$; (iv) $\frac{5}{2}-x, -y, \frac{1}{2}+z$.

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

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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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Li, J. P., Wang, Y. L., Wang, H., Luo, Q. F. & Wang, X. Y. (2001). *Synth. Commun.* **31**, 781–785.

Mao, J.-M., Chen, H.-Y., Zhang, J. & Cai, M.-S. (1995). *Synth. Commun.* **25**, 1563–1565.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXTL.* Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Yang, B., Jiao, K., Li, J.-Z., Li, X.-M. & Zhang, S.-S. (2004). In preparation.

Zhang, S.-S., Lin, J.-H. & Jian, F.-F. (2002). *Chin. J. Chem.* **20**, 183–186.

Zhang, S.-S., Wang, Z.-W., Li, M., Jiao, K., Razak, I. A., Shanmuga Sundara Raj, S. & Fun, H.-K. (2001). *Acta Cryst.* C**57**, 566–568.

o2052 Shu-Sheng Zhang *et al.* • $C_{23}H_{29}N_3O_{10}S$ *Acta Cryst.* (2004). E**60**, o2050–o2052