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4-(2,3,4,6-Tetra-*O*-acetyl-β-D-galacto-pyranosyl)thiosemicarbazide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.127
Data-to-parameter ratio = 15.4

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

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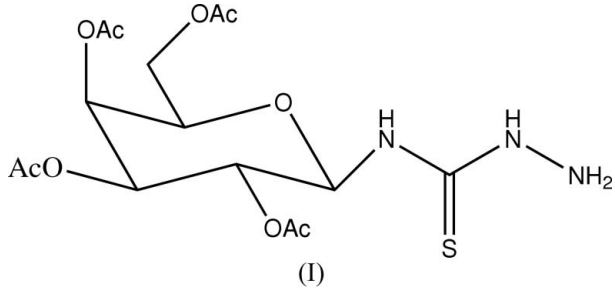
In the title compound, $C_{15}H_{23}N_3O_9S$, the hexopyranosyl ring adopts a chair conformation. The acetyl group at C4 occupies an axial position, while all other substituents are equatorial. The molecules are linked by C—H···O hydrogen bonds into ribbons parallel to the *a* axis, and N—H···O interactions provide further stability in a three-dimensional network.

Comment

Substituted thioureas and thiosemicarbazides have attracted much attention in recent years because of their anti-HIV potential (Venkatachalam *et al.*, 2001) and their importance in the preparation of corresponding semicarbazides (Li *et al.*, 2001) and heterocyclic compounds (Wang *et al.*, 2001). We have reported the synthesis and structure of *N*-amino-*N*-(2,3,4,6-tetra-*O*-acetyl-β-D-xylopyranosyl)thiocarbamide (II) (Yang *et al.*, 2004) and this work has been extended to the synthesis of the title compound (I) whose structure is reported here (Fig. 1, Table 1). All bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987), and compare well with those values in compound (II). The acetyl group at C4 occupies an axial position, while all other substituents are equatorial. The pyranosyl ring adopts a chair conformation with the S1 atom in a synperiplanar position with respect to C1. The C1—N1—C15—S1 torsion angle is −5.5 (4)°. Atom O5 is disordered over two positions, with refined site occupancies of 0.32 (6) for O5*A* and 0.68 (6) for O5*B*.



(I)

In the crystal structure of (I), molecules are linked into ribbons along the *a* axis by C12—H12*C*···O5*B* hydrogen bonds (Fig. 2). The packing is further stabilized by N2—H2*A*···O1 interactions (Table 2), forming a three-dimensional framework.

Experimental

The title compound was prepared by the method of Yang *et al.* (2004). Colourless crystals suitable for X-ray crystallographic analysis were obtained by recrystallization from ethyl acetate/petroleum ether (1:2 *v*/*v*).

Crystal data

$C_{15}H_{23}N_3O_9S$
M_r = 421.42
Orthorhombic, $P2_12_12_1$
a = 7.5445 (11) Å
b = 8.6792 (13) Å
c = 31.229 (5) Å
V = 2044.9 (5) Å^3
Z = 4
D_x = 1.369 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3602 reflections
θ = 2.4–22.3°
μ = 0.21 mm^{-1}
T = 293 (2) K
Needle, colourless
0.30 × 0.14 × 0.11 mm

Data collection

Simens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.940, T_{max} = 0.977
11659 measured reflections
4041 independent reflections
3407 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.0°
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 10$
$l = -37 \rightarrow 38$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.127
S = 1.02
4041 reflections
263 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.2979P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1690 Friedel pairs
Flack parameter: 0.03 (12)

Table 1
Selected geometric parameters (Å, °).

S1—C15	1.679 (3)	N1—C1	1.414 (3)
O1—C1	1.426 (3)	N2—C15	1.332 (4)
O1—C5	1.427 (3)	N2—N3	1.395 (4)
N1—C15	1.350 (3)		
N3—N2—C15—N1	5.7 (4)	C1—N1—C15—N2	175.9 (2)
N3—N2—C15—S1	−172.9 (2)	C1—N1—C15—S1	−5.5 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.86	2.58	3.427 (3)	168
C12—H12C···O5B^{ii}	0.96	2.31	3.12 (2)	141

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

All H atoms were positioned geometrically and treated as riding, with C—H = 0.96–0.98 Å [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for the methyl H atoms] and N—H = 0.86 Å [U_{iso}(H) = 1.2U_{eq}(N)].

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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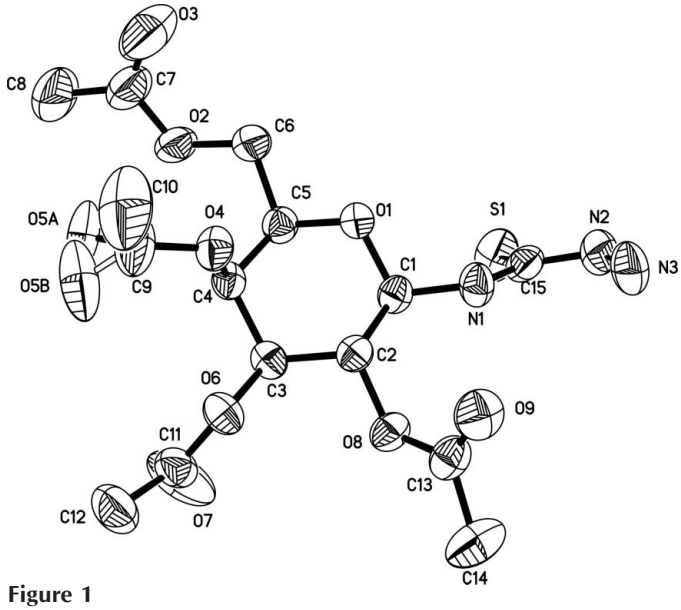

Figure 1
The structure of compound (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are omitted for clarity.

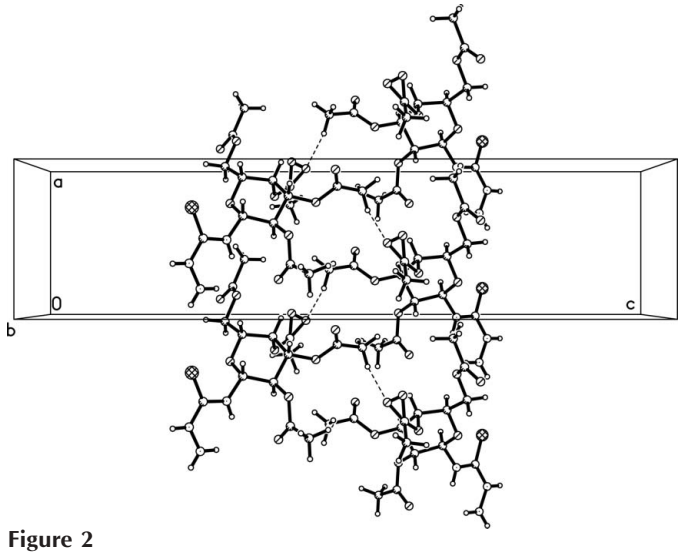

Figure 2
Packing diagram for (I), showing the formation of ribbons along the *a* axis. H bonds are drawn as dashed lines.

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