organic papers

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.052
wR factor = 0.133
Data-to-parameter ratio = 12.7

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

N-Amino-N-(2,3,4-tri-O-acetyl-β-D-xylo-pyranosyl)thiocarbamide

In the title compound, $C_{12}H_{19}N_3O_7S$, the xylopyranosyl ring adopts a chair conformation. All the substituents are in equatorial positions. There are four intramolecular interactions forming four closed rings. C—H$\cdots$O and N—H$\cdots$S interactions link the molecules into a three-dimensional framework.

Comment

Thiosemicarbazones are important because of their wide range of applications in industry, in medicine and in the analytical determination of various metal ions (Alicia *et al.*, 1998). *N,N′*-Disubstituted thioureas are known to exhibit antiviral, antitubercular and herbicidal activities (Li *et al.*, 2001). Recently, we have synthesized the title compound, (I). An X-ray crystallographic analysis of (I) was undertaken to establish its stereochemistry.


(I)
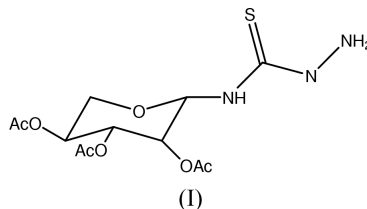
The bond lengths and angles in (I) have normal values and are in good agreement with those in a related compound, *O*-methyl-*N*-(2,3,4-tri-*O*-acetyl-β-D-xylopyranosyl)thiocarb-amate (Yang *et al.*, 2004). The xylopyranosyl ring adopts a chair conformation (Fig. 1). The sum of the bond angles around atom N3 indicates a pyramidal configuration. Each of the ring substituents is planar and occupies an equatorial position. All the substituents are involved in intramolecular interactions (Table 2), forming one seven-membered ring and three five-membered rings. The molecular packing is stabilized by C—H$\cdots$O and N—H$\cdots$S (Table 2 and Fig. 2) interactions, which lead to the formation of a three-dimensional framework.

Experimental

Ethanol (100 ml) and hydrazine hydrate (50% aqueous solution, 1.2 ml) were mixed below 278 K in an ice bath. (2,3,4-Tri-*O*-methyl)-β-D-xylopyranosyl isothiocyanate (3.27 g) in ethanol (30 ml) was added dropwise with stirring. The solution was filtered after 10 min of stirring. Pale-yellow single crystals suitable for X-ray crystallographic analysis were obtained by recrystallization from ethyl acetate/petroleum ether (1:3, *v*/*v*).
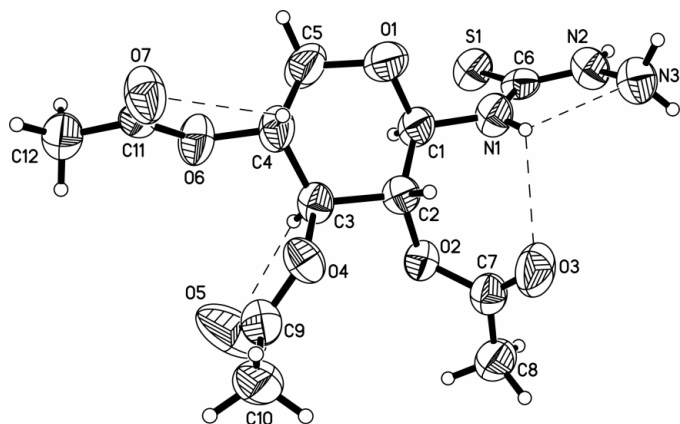
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Crystal data

$C_{12}H_{19}N_3O_7S$	$D_x = 1.339$ Mg m^{-3}
$M_r = 349.36$	Mo $K\alpha$ radiation
Monoclinic, $C2$	Cell parameters from 2772
$a = 22.560$ (8) Å	reflections
$b = 5.4253$ (19) Å	$\theta = 1.6$–$26.4°$
$c = 15.057$ (5) Å	$\mu = 0.22$ mm^{-1}
$\beta = 109.886$ (6)°	$T = 293$ (2) K
$V = 1733.0$ (10) Å^3	Needle, yellow
$Z = 4$	$0.26 \times 0.14 \times 0.12$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	2772 independent reflections
	2023 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.030$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 26.4°$
	$h = -27 \rightarrow 28$
$T_{min} = 0.944$, $T_{max} = 0.974$	$k = -6 \rightarrow 5$
5020 measured reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.052$	$+ 1.2246P]$
$wR(F^2) = 0.133$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
2772 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
219 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Absolute structure: Flack (1983), 803 Friedel pairs
	Flack parameter = -0.07 (16)

Table 1
Selected geometric parameters (Å, °).

S1—C6	1.685 (5)	O2—C2	1.442 (5)
N1—C6	1.342 (5)	O3—C7	1.175 (6)
N1—C1	1.434 (6)	O4—C9	1.314 (6)
N2—C6	1.324 (6)	O4—C3	1.442 (5)
N2—N3	1.407 (6)	O5—C9	1.177 (7)
O1—C1	1.423 (6)	O6—C11	1.335 (5)
O1—C5	1.429 (7)	O6—C4	1.455 (6)
O2—C7	1.357 (6)	O7—C11	1.184 (6)
N3—N2—C6—N1	−2.4 (6)	C2—O2—C7—C8	176.1 (4)
N3—N2—C6—S1	−179.9 (3)	C3—O4—C9—O5	−2.4 (8)
C1—N1—C6—N2	171.1 (4)	C3—O4—C9—C10	177.6 (4)
C1—N1—C6—S1	−11.5 (6)	C4—O6—C11—O7	−1.7 (7)
C2—O2—C7—O3	−4.6 (7)	C4—O6—C11—C12	179.3 (4)
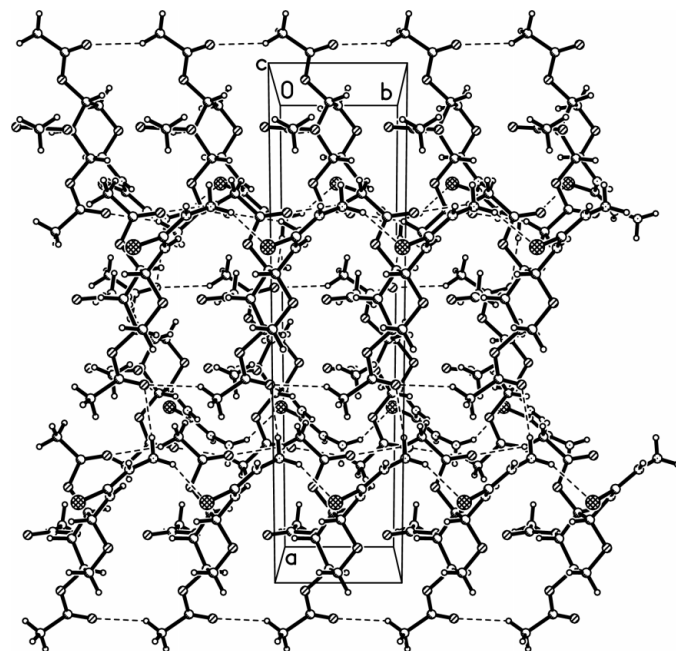
Figure 2
Packing diagram of (I), showing the formation of a three-dimensional framework.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O3	0.86	2.57	3.117 (6)	123
N1—H1$\cdots$N3	0.86	2.25	2.630 (6)	107
N2—H2$\cdots$S1^i	0.86	2.55	3.322 (5)	150
N3—H3A$\cdots$O7^{ii}	0.90 (3)	2.31 (3)	3.198 (6)	174 (4)
N3—H3B$\cdots$S1^{iii}	0.89 (3)	2.78 (6)	3.520 (5)	142 (5)
C1—H1A$\cdots$S1	0.98	2.66	3.090 (5)	107
C3—H3$\cdots$O5	0.98	2.31	2.712 (7)	104
C4—H4$\cdots$O7	0.98	2.35	2.727 (6)	102
C8—H8C$\cdots$O3^{iv}	0.96	2.41	3.300 (7)	154
C12—H12B$\cdots$O7^{iv}	0.96	2.46	3.281 (7)	144

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

Atoms H3A and H3B were located in a difference map and included in the refinement with an N—H distance restraint of 0.89 (1) Å. The remaining H atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.96–0.98 Å) and treated as riding, with $U_{iso}(H) = 1.2U_{eq}(C,N)$ [for methyl H atoms, $U_{iso}(H) = 1.5U_{eq}(C)$]. A rotating-group model was used for the methyl groups.

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, 1990).

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