Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

5-Phenyl-1,3,4-thiadiazol-2-yl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-glucopyranoside

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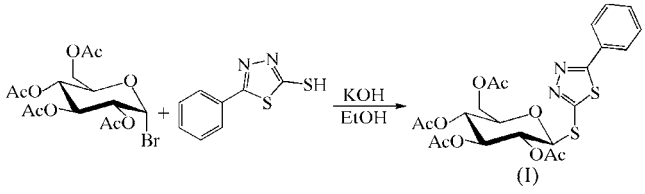
Received 11 October 2004
Accepted 3 May 2005
Online 9 July 2005

The structure of the title compound, $C_{22}H_{24}N_2O_9S_2$, is described. This compound consists of a sugar ring and a heterocyclic base linked unusually by an S atom. The sugar is in a $^4C_1$ chair conformation and forms dihedral angles of 49.54 (4) and 33.42 (5)° with the thiadiazole and phenyl rings, respectively. The S atom occupies an equatorial position of the sugar ring and lies 1.807 (2) Å out of the corresponding mean plane.

Comment

1,3,4-Thiadiazole derivatives display a broad spectrum of biological activities, such as fungicidal (Zou *et al.*, 2002), antibacterial (Deibel *et al.*, 2004), antidepressant (Clerici *et al.*, 2001) and anti-epileptic (Masereel *et al.*, 2002). However, the application of 1,3,4-thiadiazoles is limited because of their poor solubility both in organic solvents and in water. In the course of identifying new chemical structures that may serve as leads in the design of novel antiviral agents, we were particularly interested in the linking of thio-1,3,4-thiadiazoles to hydrophilic moieties such as D-glucose. The present structure determination is part of an investigation into the nucleophilic substitution of a bromosugar to a thiaheterocyclic compound. We report here the X-ray crystal structure determination of the title compound, (I) (Fig. 1).



(I)

The 2,3,4,6-tetra-*O*-acetylglucopyranosyl ring of (I) assumes a $^4C_1$ chair conformation, with atoms C2 and C5 displaced from the C1/C3/C4/O1 mean plane. The mean C—C and C—O(acetyl) bond lengths of 1.519 (2) and 1.422 (2) Å, respectively, in the sugar moiety compare well with similar averages observed in pyranose sugars (Berman *et al.*, 1967).

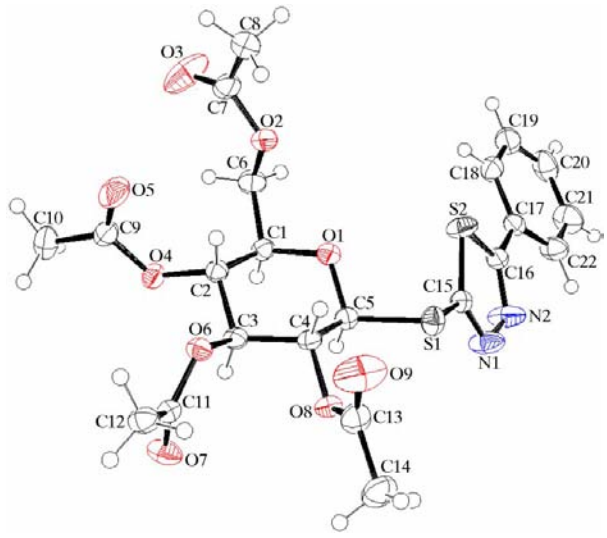


Figure 1
A view of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

The two endocyclic C—O bonds, C1—O1 and C5—O1, are nearly equal [1.431 (2) and 1.418 (2) Å, respectively]. The conformation about the exocyclic C1—C6 bond is *gauche–trans*, with torsion angles O2—C6—C1—O1 = −59.6 (2)° and O2—C6—C1—C2 = 61.0 (2)°.

The heterocyclic 1,3,4-thiadiazole ring is planar to within experimental error and the lengths of the endocyclic bonds [C15—N1 = 1.299 (2) Å and C16—N2 = 1.303 (2) Å], clearly indicate that they are double bonds. The N1—N2 bond length of 1.375 (2) Å is slightly shorter than the single-bond value of 1.393 (4) Å in the hydrochloride of 5-(4-methoxyphenyl)-4-phenyl-1,3,4-thiadiazolium-2-phenylaminide (Cheung *et al.*, 1992). The large deviation of the bond angle [S2—C15—N1 = 114.16 (13)°] in the ring from the value of 120° usually found in trigonal–planar arrangements is common in five-membered rings (Downie *et al.*, 1972). The phenyl ring linked to atom C16 forms a dihedral angle of 16.95 (6)° with the heterocyclic plane.

The S atom of the thioglucosidic linkage bridges the sugar and heterocyclic rings. The dihedral angles between the mean plane of the sugar ring and the thiadiazole and phenyl planes are 49.54 (4) and 33.42 (5)°, respectively. The orientation of the thiadiazole moiety relative to the glucose ring may be described by the torsion angles N1—C15—S1—C5 = −107.0 (2)°, S2—C15—S1—C5 = 78.1 (1)°, C15—S1—C5—O1 = −67.2 (1)° and C15—S1—C5—C4 = 176.3 (1)°.

Experimental

5-Mecapto-2-phenyl-1,3,4-thiadiazole (0.582 g, 3 mmol), α-acetobromoglucose (1.230 g, 3 mol) and KOH (0.168 g, 3 mmol) were reacted at room temperature for 12 h. The product was obtained *via* column-chromatograpic purification and the crystals were recrystallized by slow evaporation from petroleum ether–ethyl acetate (4:1)

 DOI: 10.1107/S0108270105003951

(m.p. 424–426 K). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.018 (*s*, 3H), 2.040 (*s*, 3H), 2.060 (*s*, 3H), 2.088 (*s*, 3H), 3.845–3.886 (*m*, 1H), 4.166 (*dd*, *J* = 2.0 and 12.8 Hz, 1H), 4.302 (*dd*, *J* = 4.8 and 12.8 Hz, 1H), 5.137–5.205 (*m*, 2H), 5.309 (*t*, *J* = 9.2 Hz, 1H), 5.444 (*d*, *J* = 10.4 Hz, 1H), 7.459–7.511 (*m*, 3H), 7.899–7.922 (*m*, 2H).

Crystal data

$C_{22}H_{24}N_2O_9S_2$
M_r = 524.56
Orthorhombic, $P2_12_12_1$
a = 8.010 (2) Å
b = 10.526 (2) Å
c = 28.685 (6) Å
V = 2418.7 (9) Å^3
Z = 4
D_x = 1.440 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7511 reflections
θ = 3.2–27.5°
μ = 0.28 mm^{-1}
T = 173.2 K
Block, colourless
0.35 × 0.25 × 0.20 mm

Data collection

Rigaku/MSC Mercury CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*TEXSAN*; Molecular Structure Corporation, 1999)
T_{min} = 0.825, T_{max} = 0.947
19569 measured reflections
5524 independent reflections
5151 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.037
θ_{max} = 27.5°
$h = -10 \rightarrow 9$
$k = -11 \rightarrow 13$
$l = -37 \rightarrow 37$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.046
S = 1.06
5499 reflections
317 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o) + 0.00063|F_o|^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Absolute structure: Flack (1983), 2357 Friedel pairs
Flack parameter: 0.00 (5)

All H atoms were placed in calculated positions with C—H distances of 0.97 Å, and were included in the final cycles of refinement with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1991); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *PLATON* (Spek, 2003).

Financial support by the Natural Science Foundation of China (grant Nos. 20372027 and 20021001) and the Natural Science Foundation of Gansu Province (grant No. YS031-A21-002) is gratefully acknowledged. The collection and processing of the X-ray data in Professor K. Tatsumi's group by Dr T. Matsumoto (Nagoya University) is also gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TY1004). Services for accessing these data are described at the back of the journal.

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