Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

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

Zao-Zao Qiu and Peng-Fei Xu*

State Key Laboratory of Applied Organic Chemistry, Lanzhou University, Lanzhou 73000, People's Republic of China

Correspondence e-mail: xupf@lzu.edu.cn

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.046
Data-to-parameter ratio = 15.8

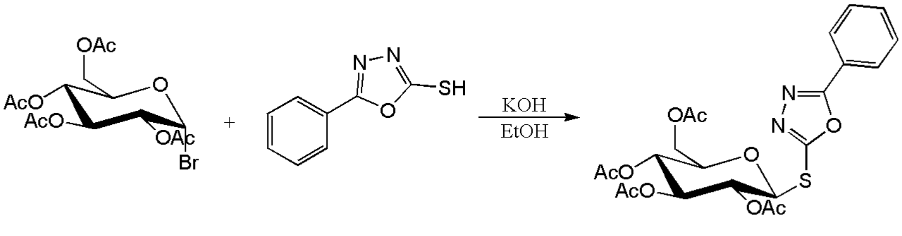
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 29 April 2004
Accepted 17 June 2004
Online 17 July 2004

The crystal structure of the title compound, $C_{22}H_{24}N_2O_{10}S$, has been determined by X-ray diffraction. The molecule contains a sugar ring and a planar substituted heterocyclic ring linked, unusually, by an S atom. The sugar ring is in the normal chair conformation.

Comment

1,3,4-Oxadiazole derivatives have widespread uses as dyes (Naito *et al.*, 1999), photosensitive and electrical materials (Xu *et al.*, 2000), and have a broad spectrum of biological activities, such as anti-HIV (Eissa, 1998), antibacterial (Holla *et al.*, 2000) and antifungal (Tinperciuc *et al.*, 1999). The application of 1,3,4-oxadiazoles is limited because of their poor solubility in both organic solvents and water. In the course of identifying new chemical structures which may serve as leads for designing novel antiviral agents, we were particularly interested in the linking of thio-1,3,4-dioxazoles to hydrophilic moieties such as D-glucose.



(I)

The structure determination of the title compound, (I), is part of an investigation into the nucleophilic substitution of bromosugars by thioheterocyclic compounds. In the present case, the substitution affords the desired compound, (I), with good selectivity and we report here its crystal structure. The acetylglucose ring adopts a $^4C_1$ chair conformation, with all substituents in equatorial positions. The structure determination shows clearly that compound (I) is in the β configuration. The C1—S—C15 angle is 102.9 (1)°. The heterocyclic ring is planar within experimental error and the endocyclic bond lengths N1—C15 and N2—C16 of 1.287 (3) and 1.297 (3) Å, respectively, clearly indicate they are double bonds. The C—N—N angles in the ring show large deviations from the value of 120° usually found in the trigonal planar arrangement, which is common in five-membered rings (Downie *et al.*, 1972).

Experimental

1,3,4-Oxadiazole-5-thiol (0.516 g, 3 mmol), α-acetobromoglucose (1.230 g, 3 mmol) and KOH (0.168 g, 3 mmol) were reacted at room temperature for 12 h. The product was obtained by column chromatographic purification and the crystals were recrystallized by slow evaporation of a petroleum ether–ethyl acetate solution (PE–EA,

DOI: 10.1107/S1600536804014734

4:1).^{1}H NMR (300 Hz, CDCl$_3$): 2.02–2.08 (*m*, 12H), 3.89 (*m*, 1H), 4.13 (*d*, J = 12.6 Hz, 1H), 4.29 (*dd*, J = 4.5, 12.6 Hz, 1H), 5.13–5.36 (*m*, 3H), 5.52 (*d*, J = 10.2 Hz, 1H), 7.52 (*m*, 3H), 8.01 (*d*, J = 7.8 Hz, 2H).

Crystal data

$C_{22}H_{24}N_2O_{10}S$
M_r = 508.50
Monoclinic, $P2_1$
a = 11.50 (2) Å
b = 7.35 (1) Å
c = 14.30 (2) Å
β = 92.42 (2)°
V = 1208 (3) Å^3
Z = 2

D_x = 1.398 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3903 reflections
θ = 3.1–27.5°
μ = 0.19 mm^{-1}
T = 173.2 K
Block, colorless
0.35 × 0.20 × 0.15 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{\min}$ = 0.897, $T_{\max}$ = 0.972
9357 measured reflections

4988 independent reflections
4441 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int}$ = 0.021
$\theta_{\max}$ = 27.4°
$h = -14 \rightarrow 14$
$k = -7 \rightarrow 9$
$l = -18 \rightarrow 12$

Refinement

Refinement on F
R = 0.036
wR = 0.046
S = 1.06
4971 reflections
315 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o) + 0.00063|F_o|^2]$
$(\Delta/\sigma)_{\max}$ = 0.003
$\Delta\rho_{\max}$ = 0.22 e Å^{-3}
$\Delta\rho_{\min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983); 2009 Friedel pairs
Flack parameter = 0.028 (6)

Table 1
Selected geometric parameters (Å, °).

S—C1	1.805 (4)	O6—C11	1.359 (3)
S—C15	1.745 (4)	O7—C11	1.203 (3)
O1—C1	1.424 (3)	O8—C2	1.440 (3)
O1—C5	1.434 (4)	O8—C13	1.378 (3)
O2—C6	1.442 (4)	O9—C13	1.194 (3)
O2—C7	1.354 (4)	O10—C15	1.364 (3)
O3—C7	1.211 (3)	O10—C16	1.375 (3)
O4—C4	1.446 (4)	N1—N2	1.413 (4)
O4—C9	1.359 (4)	N1—C15	1.287 (4)
O5—C9	1.199 (3)	N2—C16	1.297 (4)
O6—C3	1.443 (4)		
C1—S—C15	102.93 (10)	O2—C6—C5	109.25 (17)
C1—O1—C5	111.37 (13)	O2—C7—O3	122.9 (2)
C6—O2—C7	117.12 (15)	O2—C7—C8	111.19 (19)
C4—O4—C9	118.44 (16)	O3—C7—C8	125.9 (2)
C3—O6—C11	118.37 (15)	O4—C9—O5	123.0 (2)
C2—O8—C13	117.65 (14)	O4—C9—C10	110.19 (19)
C15—O10—C16	102.00 (15)	O5—C9—C10	126.8 (2)
N2—N1—C15	105.76 (19)	O6—C11—O7	123.28 (18)
N1—N2—C16	106.4 (2)	O6—C11—C12	109.96 (19)
S—C1—O1	109.24 (12)	O7—C11—C12	126.75 (19)
S—C1—C2	115.42 (16)	O8—C13—O9	122.93 (15)
O1—C1—C2	110.06 (17)	O8—C13—C14	110.51 (17)
O8—C2—C1	109.94 (16)	O9—C13—C14	126.6 (2)
O8—C2—C3	107.20 (13)	S—C15—O10	120.37 (16)
O6—C3—C2	105.60 (17)	S—C15—N1	125.96 (17)
O6—C3—C4	109.88 (17)	O10—C15—N1	113.56 (19)
O4—C4—C3	109.44 (14)	O10—C16—N2	112.28 (19)
O4—C4—C5	107.33 (17)	O10—C16—C17	118.29 (19)
O1—C5—C4	107.74 (17)	N2—C16—C17	129.4 (2)
O1—C5—C6	108.73 (15)		

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.97 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

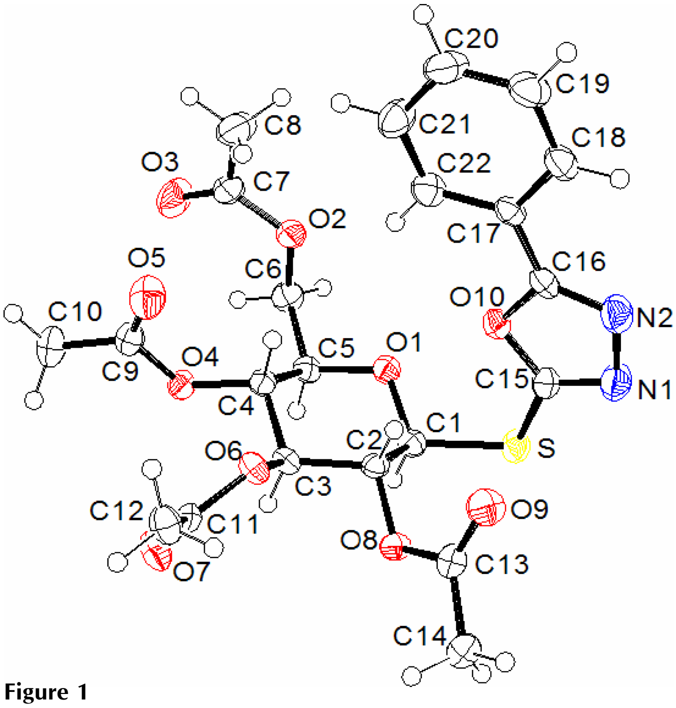


Figure 1
View (50% probability displacement ellipsoids) of the title compound, with the atom-numbering scheme.

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: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *teXsan*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *PLATON* (Spek, 2003).

Financial support by NSFC (20372027 and 20021001), and the collection and processing of the X-ray data by Professor K. Tatsumi and co-workers is gratefully acknowledged.

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