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6-(4-Chlorophenyl)-3-(D-*gluco*-pentitol-1-yl)-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazine

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.030
wR factor = 0.070
Data-to-parameter ratio = 9.5

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

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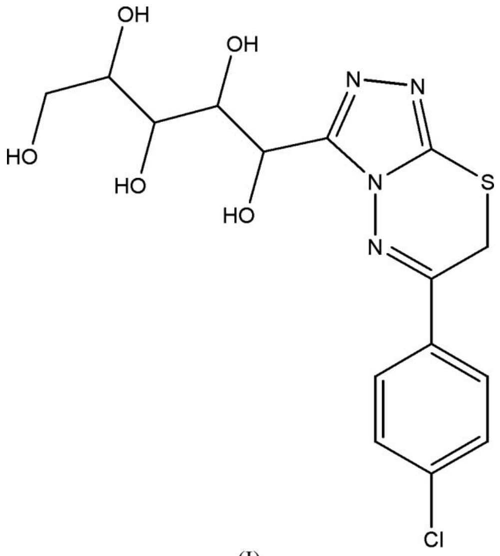
In the title compound, $C_{15}H_{17}ClN_4O_5S$, the five-membered triazole ring and the benzene ring are each planar, while the six-membered thiadiazine ring is slightly distorted, with an r.m.s. deviation of 0.236 (1) Å. Intermolecular O—H···O and O—H···N hydrogen-bond interactions are found in the crystal structure, and these link molecules into a two-dimensional network.

Comment

3,6-Disubstituted-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazines are among the various heterocycles that have received considerable attention over the past two decades as potential biologically active reagents, such as antimicrobial (Feng *et al.*, 1992), antibacterial (Dhiman *et al.*, 2001), antifungal (Hui, Dong *et al.*, 2000), herbicidal (Nizamuddin *et al.*, 1999), anti-inflammatory (Prasad *et al.*, 1989), diuretic (Mohan & Anjaneyulu, 1987), anthelmintic and analgesic compounds (Turan *et al.*, 1999). They can also be used as plant-growth inhibitors (Feng *et al.*, 1991) and inhibitors of malignant cellular proliferation (Deshmukh *et al.*, 1984). Most of the compounds of this type that have been synthesized previously have had alkyl or aryl substituents in the 3- and 6-positions of the triazolothiadiazine system (Hui, Zhang & Zhang, 2000; Inviata *et al.*, 1997). This is a disadvantage for biological applications because of their poor water solubility.



(I)

D-Glucono residues are known to play a role in living systems and are also readily soluble in water, and we report here the synthesis and crystal structure of the title compound, $C_{15}H_{17}ClN_4O_5S$, (I), a chiral D-*gluco* (1*R*,2*S*,3*R*,4*R*,5-penta-

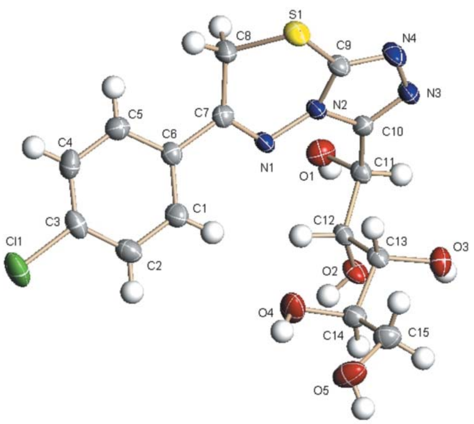


Figure 1
The molecular structure of (I) with the atom numbering, showing displacement ellipsoids at the 30% probability level.

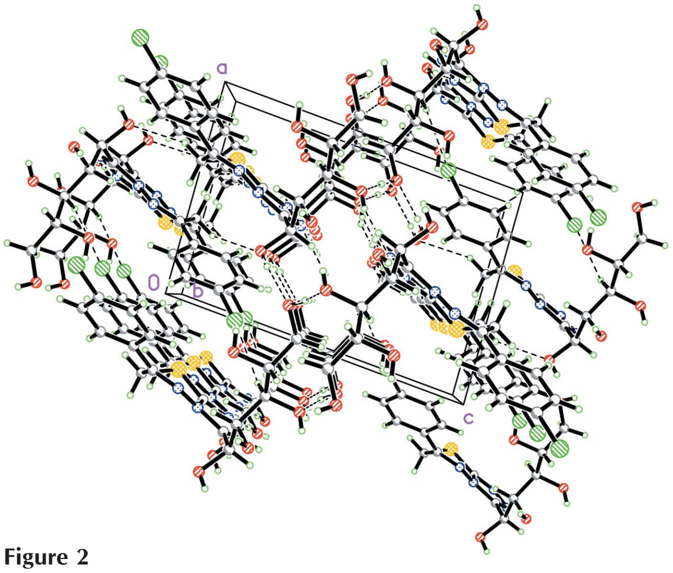


Figure 2
The packing of (I), with hydrogen bonds shown as dashed lines.

hydroxylpentyl) derivative (Fig. 1), with the chirality arising from the starting D-*gluco*-triazole reagent.

In (I), the five-membered triazole ring (atoms N2–N4, C9 and C10) and the benzene ring (C1–C6) are each planar, while the six-membered thiadiazine ring, composed of atoms N1, N2, C7, C8, C9 and S1, is distorted from planarity, with an r.m.s. deviation of 0.236 (1) Å, and may be regarded as having a screw–boat conformation (Fig 1). Both the S—C (with mean distance 1.773 Å) and C—N bond lengths are in line with those observed in related compounds (Sert *et al.*, 2003; Xiang *et al.*, 2004). The bond lengths in the triazole ring show normal values (Allen *et al.*, 1987; Jin *et al.*, 2004; Table 1). In the crystal structure, O—H···N and O—H···O intermolecular hydrogen-bonding interactions link the molecules into a two-dimensional network (Fig. 2 and Table 2).

Experimental

The key intermediate 4-amino-5-mercapto-3-(D-*gluco*-pentitol-1-yl)-1,2,4-triazole was prepared by the literature method of Laila *et al.* (1999). To a solution of 3-(D-*gluco*-pentitol-1-yl)-4-amino-5-mercapto-1,2,4-triazole (0.01 mol) in 95% ethanol was added 2-bromo-4-chloroacetophenone (0.01 mol). The mixture was refluxed for 4 h. The solid obtained on cooling was filtered, dried and recrystallized from 95% ethanol to give the pure product, (I). The purified product was dissolved in 95% ethanol and single crystals were obtained after 6 d. IR (KBr, cm^{-1}): 3194 (O—H), 2940 (CH$_2$), 1641 (C═N), 1544, 1510, 1484 (aromatic ring skeleton), 1472 (C—S), 703 (C—S—C). ^{1}H NMR (dimethylsulfoxide-d_6): 8.07–8.04 (*d*, 2H, Ar—H), 7.66–7.63 (*d*, 2H, Ar—H), 5.04–4.36 (*m*, 5H, O—H), 3.55–3.11 (*m*, 8H, C—H). ^{13}C NMR (dimethylsulfoxide-d_6): 153.78, 153.66, 140.64, 136.86, 132.57, 129.57, 129.16, 71.45, 70.63, 70.54, 65.99, 63.56, 22.92 p.p.m. Elemental analysis for $C_{15}H_{17}ClN_4O_5S$: C 44.99, H 4.36, N 14.31%; calculated: C 44.89, H 4.27, N 14.03%. M.p. 448–450 K.

Crystal data

$C_{15}H_{17}ClN_4O_5S$
$M_r = 400.84$
Monoclinic, $P2_1$
$a = 8.5235$ (8) Å
$b = 8.0933$ (7) Å
$c = 12.3116$ (11) Å
$\beta = 94.681$ (2)°
$V = 846.46$ (13) Å^3
$Z = 2$

$D_x = 1.573$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2325 reflections
$\theta = 2.5$–25.0°
$\mu = 0.39$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.24 × 0.20 × 0.15 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.913$, $T_{\max} = 0.944$
4506 measured reflections

2269 independent reflections
2172 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 25.2°$
$h = -10 \rightarrow 9$
$k = -6 \rightarrow 9$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.070$
$S = 1.05$
2269 reflections
240 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.1355P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Absolute structure: Flack (1983), 1633 Friedel pairs
Flack parameter: 0.02 (6)

Table 1
Selected geometric parameters (Å, °).

S1—C9	1.733 (3)	N2—C10	1.383 (3)
S1—C8	1.814 (3)	N3—C10	1.291 (4)
N1—C7	1.293 (3)	N3—N4	1.394 (3)
N1—N2	1.389 (3)	N4—C9	1.303 (3)
N2—C9	1.367 (3)		
C9—S1—C8	94.39 (13)	C9—N4—N3	106.8 (2)
C7—N1—N2	115.5 (2)	N4—C9—N2	110.3 (2)
C9—N2—N1	127.8 (2)	N4—C9—S1	128.5 (2)
C10—N2—N1	124.9 (2)	N2—C9—S1	121.17 (19)
C10—N3—N4	108.57 (19)	N3—C10—N2	109.3 (2)
C7—N1—N2—C9	28.1 (4)	N1—N2—C9—N4	165.1 (2)
C7—N1—N2—C10	−171.7 (2)	N1—N2—C9—S1	−13.9 (4)
C10—N3—N4—C9	−0.8 (3)	N4—N3—C10—N2	2.0 (3)
N3—N4—C9—N2	−0.7 (3)	N1—N2—C10—N3	−166.2 (2)
N3—N4—C9—S1	178.1 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O5^{i}	0.82	1.99	2.809 (3)	172
O2—H2···N4^{ii}	0.82	1.93	2.742 (3)	174
O3—H3···N3^{iii}	0.82	2.19	2.964 (3)	156
O3—H3···O2	0.82	2.37	2.803 (3)	114
O4—H4···O5	0.82	2.14	2.631 (3)	118
O5—H5···O3^{iv}	0.82	2.11	2.866 (3)	154

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms at C—H distances of 0.93–0.98 Å and O—H distances of 0.82 Å, with $U_{\rm iso}$(H) =1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$(O).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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