

# 4-Amino-3-(1,2,3,4,5-pentahydroxypentyl)-1,2,4-1*H*-triazole-5(4*H*)-thione

Li-Xue Zhang,<sup>a</sup> An-Jiang Zhang,<sup>a</sup>  
Xin-Xiang Lei,<sup>a</sup> Kai-Huang Zou<sup>a</sup>  
and Seik Weng Ng<sup>a,b\*</sup>

<sup>a</sup>School of Chemistry and Materials Science, Wenzhou Normal College, Wenzhou 325027, People's Republic of China, and <sup>b</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

## Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean  $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$

$R$  factor = 0.047

$wR$  factor = 0.095

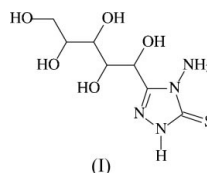
Data-to-parameter ratio = 6.6

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

In the title compound,  $\text{C}_7\text{H}_{14}\text{N}_4\text{O}_5\text{S}$ , the 5-mercapto substituent of the 1,2,4-triazole exists in the thione form [ $\text{C}=\text{S} = 1.666(5)\text{ \AA}$ ], with the protonated N atom of the triazolyl ring, the amine group and the hydroxy groups of the pentyl chain engaged in extensive hydrogen-bonding interactions, giving rise to a three-dimensional network structure.

## Comment

The 3-substituted 4-amino-1,2,4-triazole-5-thione class of heterocycles, as represented by the methyl homolog, are heterocyclic compounds that can chelate to metal atoms through amine and double-bonded S atoms (Sen *et al.*, 1996). These compounds possess useful anti-inflammatory and antimicrobial properties. The number of crystallographically verified compounds is, however, small, and these include the methyl (Escobar-Valderrama *et al.*, 1989), trifluoromethyl (Menzies & Squattrito, 2001), ethyl (Kajdan *et al.*, 2000), propyl (McCarrick *et al.*, 2000) and 2-pyridyl (Dallavalle *et al.*, 2002) derivatives.



The present *D*-gluco (1,2,3,4,5-pentahydroxypentyl) derivative (Fig. 1) is a chiral compound, the chirality arising from the starting reagent. The bond dimensions of the triazolyl ring and the  $\text{C}=\text{S}$  bond are similar to those found in the reported derivatives. The  $\text{C}=\text{S}$  bond [ $1.668(5)\text{ \AA}$ ] is comparable to those [ $1.678(2)$  and  $1.686(2)\text{ \AA}$ ] in two Schiff base derivatives of 4-amino-3-propyl-1,2,4-triazol-5-thione (McCarrick *et al.*, 1999), as well as that [ $1.653(3)\text{ \AA}$ ] in the Schiff base derivative of 4-amino-3-methyl-1,2,4-triazol-5-thione (Liu *et al.*, 1999). The bond distance is, however, shorter than the  $\text{C}-\text{S}$  bond [ $1.681(2)\text{ \AA}$ ] in mesionic triphenyltriazoliumthiolate (Cheung *et al.*, 1993). Arising from the hydroxy groups in the substituent, the hydrogen bonds (Table 2) consolidate the structure into a tightly held three-dimensional network.

## Experimental

Carbon disulfide (13 ml, 0.22 mol) was dropped into 85% hydrazine hydrate (24 ml, 0.40 mol) and water (75 ml). The mixture was refluxed for 1 h and then cooled in an ice bath. The solid product that separated was collected and washed with ethanol and then with diethyl ether. The thiocarbonylhydrazide was purified by recrystalliza-

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tion from water to which several drops of hydrochloric acid were added (m.p. 442–445 K). This compound (5 mmol) and D-gluconolactone in pyridine were refluxed for 4 h. The product was recrystallized three times from 20% aqueous ethanol, affording the pure title compound [m.p. 473–475 K; literature m.p. 473–474 K (Awad & El Ashry, 1999)] in about 80% yield. Analysis calculated for C<sub>7</sub>H<sub>14</sub>N<sub>4</sub>O<sub>5</sub>S: C 31.57, H 5.30, N 21.04%; found: C 31.36, H 5.25, N 20.87%. <sup>1</sup>H NMR (p.p.m.): 13.52 (s, 1H, NH), 5.49 (s, 2H, NH<sub>2</sub>), 5.04–4.20 (m, 5H, OH), 3.55–3.10 (m, 8H, CH); <sup>13</sup>C NMR (p.p.m.): 165.84, 152.15, 71.43, 70.90, 70.56, 66.36, 63.51. This method is a general method for the preparation of nucleotides of 1,2,4-triazoles (El Ashry *et al.*, 2000).

Crystal data

C<sub>7</sub>H<sub>14</sub>N<sub>4</sub>O<sub>5</sub>S  
*M<sub>r</sub>* = 266.28  
 Triclinic, *P*1  
*a* = 4.875 (1) Å  
*b* = 5.306 (2) Å  
*c* = 10.669 (3) Å  
 α = 101.486 (4)°  
 β = 92.884 (4)°  
 γ = 90.339 (4)°  
*V* = 270.1 (1) Å<sup>3</sup>  
*Z* = 1  
*D<sub>x</sub>* = 1.637 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 582 reflections  
 θ = 3.9–24.5°  
 μ = 0.32 mm<sup>-1</sup>  
*T* = 298 (2) K  
 Plate, colorless  
 0.42 × 0.35 × 0.06 mm

Data collection

Bruker APEX area-detector diffractometer  
 φ and ω scans  
 Absorption correction: none  
 1583 measured reflections  
 1285 independent reflections  
 1002 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.024  
*θ*<sub>max</sub> = 26.2°  
*h* = -6 → 5  
*k* = -4 → 6  
*l* = -13 → 13

Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.047  
*wR*(*F*<sup>2</sup>) = 0.095  
*S* = 1.00  
 1062 reflections  
 160 parameters  
 H-atom parameters constrained  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0455*P*)<sup>2</sup>]  
 where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
 (Δ/σ)<sub>max</sub> = 0.001  
 Δρ<sub>max</sub> = 0.23 e Å<sup>-3</sup>  
 Δρ<sub>min</sub> = -0.24 e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

O1–C3	1.421 (5)	N3–C1	1.360 (6)
O2–C4	1.434 (6)	N3–C2	1.362 (6)
O3–C5	1.429 (6)	N3–N4	1.399 (6)
O4–C6	1.430 (6)	C2–C3	1.512 (7)
O5–C7	1.433 (6)	C3–C4	1.535 (7)
N1–C2	1.295 (6)	C4–C5	1.519 (7)
N1–N2	1.387 (5)	C5–C6	1.526 (6)
N2–C1	1.336 (7)	C6–C7	1.505 (8)
C2–N1–N2	103.2 (4)	O1–C3–C4	106.6 (4)
C1–N2–N1	113.5 (4)	C2–C3–C4	114.8 (4)
C1–N3–C2	109.1 (4)	O2–C4–C5	110.6 (4)
C1–N3–N4	125.9 (4)	O2–C4–C3	107.0 (4)
C2–N3–N4	125.1 (4)	C5–C4–C3	114.0 (4)
N2–C1–N3	102.9 (4)	O3–C5–C4	110.3 (4)
N2–C1–S1	129.1 (4)	O3–C5–C6	106.8 (4)
N3–C1–S1	128.0 (4)	C4–C5–C6	111.6 (4)
N1–C2–N3	111.3 (4)	O4–C6–C7	109.1 (4)
N1–C2–C3	125.9 (4)	O4–C6–C5	106.6 (4)
N3–C2–C3	122.7 (4)	C7–C6–C5	113.5 (4)
O1–C3–C2	109.7 (4)	O5–C7–C6	113.8 (4)

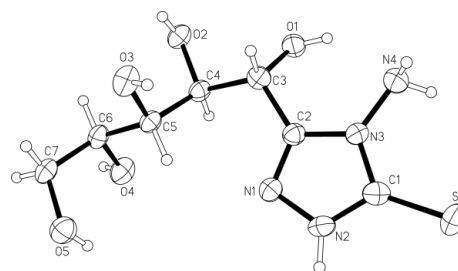


Figure 1

A view of (I), with displacement ellipsoids at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1O...N4	0.82	2.12	2.778 (6)	138
O2–H2O...O4 <sup>i</sup>	0.82	2.13	2.824 (5)	143
O3–H3O...O2 <sup>ii</sup>	0.82	1.96	2.777 (5)	179
O4–H4O...O5 <sup>iii</sup>	0.82	1.94	2.755 (5)	174
O5–H5O...O3 <sup>iv</sup>	0.82	2.07	2.827 (5)	154
N2–H2A...O1 <sup>v</sup>	0.86	1.98	2.784 (5)	156
N4–H4C...O5 <sup>vi</sup>	0.89	2.33	3.147 (6)	153
N4–H4A...S1 <sup>i</sup>	0.89	2.72	3.574 (5)	161

Symmetry codes: (i) *x*, *y* – 1, *z*; (ii) 1 + *x*, *y*, *z*; (iii) *x* – 1, *y*, *z*; (iv) *x*, 1 + *y*, *z*; (v) 1 + *x*, 1 + *y*, *z*; (vi) *x*, *y*, 1 + *z*.

H atoms attached to C, N and O atoms were located in a difference Fourier map, but they were included in the refinement in calculated positions in the riding-model approximation [C–H = 0.98 Å (methine), C–H = 0.97 Å (methylene), N–H = 0.86 Å (azolyli), N–H = 0.89 Å (amino) and O–H = 0.82 Å (hydroxy)], with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(parent atom). Despite the presence of a sulfur atom in the molecule, refinement of the absolute configuration was unsuccessful, the Flack (1983) parameter having an intermediate value with large uncertainty. The correct configuration is known from the starting material and was assumed, and Friedel pairs were merged.

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: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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