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1-(2,4,6-Trioxo-1,3-diazinan-5-ylidene)-thiosemicarbazide

Viviane C. D. Bittencourt,[a] Vanessa Carratu Gervini,[a]* Leandro Bresolin,[a] Aline Locatelli[b] and Adriano Bof de Oliveira[c]

[a]Escola de Química e Alimentos, Universidade Federal do Rio Grande, Av. Itália km 08, Campus Carreiros, 96203-900, Rio Grande, RS, Brazil, [b]Departamento de Química, Universidade Federal de Santa Maria, Av. Roraima, Campus, 97105-900, Santa Maria, RS, Brazil, and [c]Departamento de Química, Universidade Federal de Sergipe, Av. Marechal Rondon s/n, Campus, 49100-000, São Cristóvão, SE, Brazil
Correspondence e-mail: adriano@daad-alumni.de

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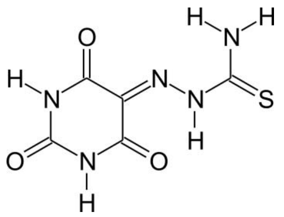
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.056; wR factor = 0.148; data-to-parameter ratio = 13.1.

The title molecule, $C_5H_5N_5O_3S$, is approximately planar, with a maximum deviation from the mean plane through the non-H atoms of 0.182 (3) Å for the amine N atom. In the crystal, molecules are connected *via* N—H···O and N—H···S interactions, building a three-dimensional hydrogen-bonded network. Additionally, a weak intramolecular N—H···O hydrogen bond is observed.

Related literature

For the synthesis of alloxan-5-thiosemicarbazone, see: Beyer *et al.* (1956). For the antibacterial activity of alloxan-5-thiosemicarbazone against *Staphylococcus aureus* and *Escherichia coli*, see: Douros *et al.* (1973).



Experimental

Crystal data

$C_5H_5N_5O_3S$
M_r = 215.20
Monoclinic, $P2_1/n$
a = 10.6415 (8) Å
b = 7.3370 (6) Å
c = 11.160 (1) Å
β = 107.380 (5)°
V = 831.55 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 293 K
0.14 × 0.10 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.949, T_{max} = 0.967
15454 measured reflections
1929 independent reflections
955 reflections with $I > 2\sigma(I)$
R_{int} = 0.090

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.148
S = 1.00
1929 reflections
147 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H4···O2^{i}	0.91 (4)	2.26 (4)	3.036 (4)	143 (3)
N2—H2···O3^{ii}	0.86 (4)	1.98 (4)	2.837 (4)	173 (4)
N5—H5···O1^{iii}	0.89 (5)	2.08 (5)	2.916 (4)	158 (4)
N4—H3···O1	0.88 (4)	2.01 (4)	2.631 (4)	126 (4)
N1—H1···O3^{iv}	0.70 (4)	2.46 (4)	2.923 (4)	125 (4)
N1—H1···S1^{v}	0.70 (4)	3.03 (4)	3.468 (4)	123 (4)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+1, -y, -z+2$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $x-\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$; (v) $-x, -y+1, -z+2$.

Data collection: *COSMO* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2271).

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supplementary materials

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1-(2,4,6-Trioxo-1,3-diazinan-5-ylidene)thiosemicarbazide

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Comment

Thiosemicarbazone derivatives have a wide range of biological properties. For example, an alloxan-thiosemicarbazone derivative shows antibacterial activity against several pathologic agents like *Staphylococcus aureus* and *Escherichia coli* (Douros *et al.*, 1973). As part of our study of thiosemicarbazone derivatives, we report herein the crystal structure of alloxan-5-thiosemicarbazone. In the title compound (Fig. 1), the molecule is planar and the maximal deviation from the least squares plane through all non-hydrogen atoms is observed for N5 (-0,1822 (30) Å). The mean deviations from the least squares planes for the alloxan fragment C1/C2/C3/C4/N1/N2/O1/O2/O3 and for the thiosemicarbazone fragment C5/N3/N4/N5/S1 amount to 0.0319 (23) Å for O3 and -0.0278 (26) Å for N4, respectively, and the dihedral angle between the two planes is 8,16 (17)°. The bond angles suggest sp^2 hybridization for the C and N atoms and explain the planarity of the molecule. The crystal packing is stabilized by intermolecular N—H···O and N—H···S as well as intramolecular N—H···O hydrogen bonding building a three-dimensional H-bonded network (Fig. 2 and Table 1).

Experimental

Starting materials were commercially available and were used without further purification. The synthesis was adapted from a procedure reported previously (Beyer *et al.*, 1956). The hydrochloric acid catalyzed reaction of alloxan monohydrate (6,25 mmol) and thiosemicarbazide (6,25 mmol) in ethanol (60 ml) was refluxed for 7 h. After cooling and filtering, crystals suitable for X-ray diffraction were obtained from a recrystallization in methanol.

Refinement

All hydrogen atoms were localized in a difference density Fourier map. Their positions and isotropic displacement parameters were refined.

Computing details

Data collection: COSMO (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

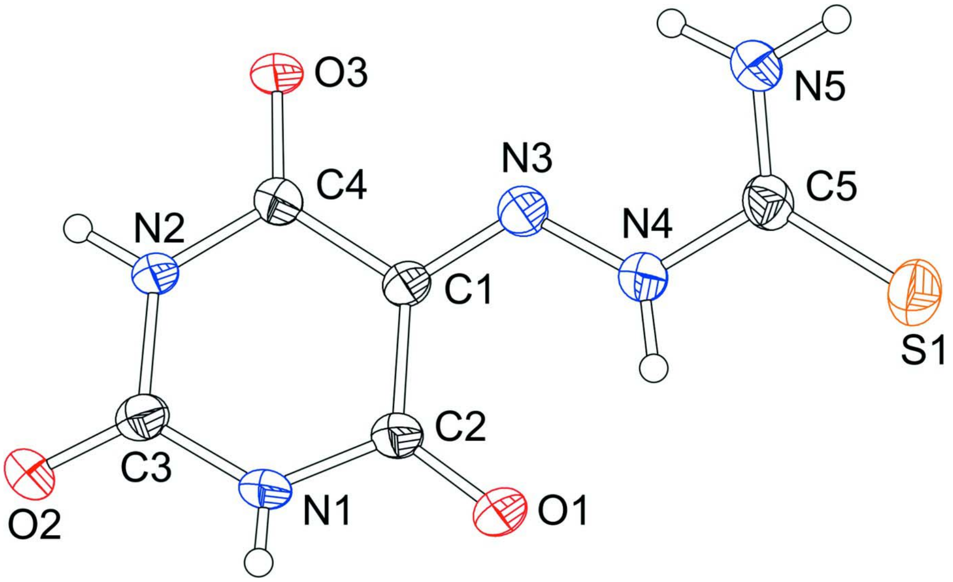

Figure 1

The molecular structure of the title compound with labeling and displacement ellipsoids drawn at the 40% probability level.

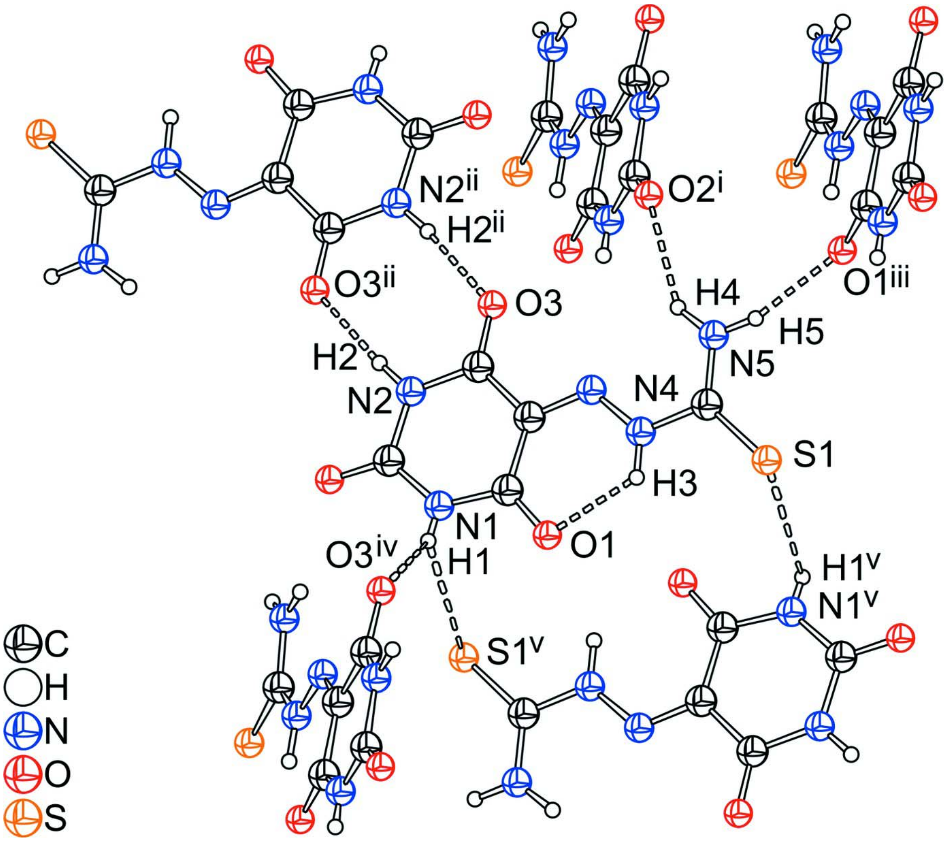

Figure 2

The crystal structure of the title compound showing the molecules connected through N—H···S hydrogen bonds. Hydrogen bonding is indicated as dashed lines. Symmetry codes: (i) *x* + 1/2, -*y* + 1/2, *z* + 1/2; (ii) -*x* + 1/2, -*y*, -*z* + 2; (iii) *x* + 1/2, -*y* + 3/2, *z* + 1/2; (iv) *x* - 1/2, -*y* + 1/2, *z* - 1/2; (v) -*x*, -*y* + 1, -*z* + 2.

1-(2,4,6-Trioxo-1,3-diazinan-5-ylidene)thiosemicarbazide

Crystal data

$C_5H_5N_5O_3S$
$M_r = 215.20$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.6415$ (8) Å
$b = 7.3370$ (6) Å
$c = 11.160$ (1) Å
$\beta = 107.380$ (5)°
$V = 831.55$ (12) Å^3
$Z = 4$

$F(000) = 440$
$D_x = 1.719$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1283 reflections
$\theta = 2.3$–19.9°
$\mu = 0.38$ mm^{-1}
$T = 293$ K
Block, red
0.14 × 0.10 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.949$, $T_{max} = 0.967$
15454 measured reflections
1929 independent reflections
955 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.090$
$\theta_{\max} = 27.6°$, $\theta_{\min} = 2.3°$
$h = -13 \rightarrow 13$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.148$
$S = 1.00$
1929 reflections
147 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.6101P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{\text{iso}}*/U_{\text{eq}}$
S1	0.17866 (11)	0.94933 (14)	1.13122 (11)	0.0487 (4)
O3	0.4802 (2)	0.2077 (3)	1.0689 (2)	0.0426 (8)
O2	0.1726 (2)	−0.0867 (4)	0.7631 (3)	0.0486 (8)
O1	0.0566 (2)	0.4570 (3)	0.8863 (3)	0.0456 (8)
N5	0.4116 (3)	0.7883 (5)	1.1940 (3)	0.0412 (9)
N3	0.3183 (3)	0.4951 (4)	1.0600 (3)	0.0335 (8)
N4	0.2392 (3)	0.6328 (4)	1.0593 (3)	0.0385 (9)
N2	0.3282 (3)	0.0686 (4)	0.9108 (3)	0.0321 (8)
N1	0.1178 (3)	0.1858 (4)	0.8272 (3)	0.0356 (9)
C5	0.2851 (4)	0.7858 (5)	1.1334 (4)	0.0340 (9)
C4	0.3687 (3)	0.2088 (5)	0.9943 (4)	0.0322 (9)
C3	0.2034 (3)	0.0473 (5)	0.8282 (4)	0.0329 (9)
C1	0.2734 (3)	0.3545 (4)	0.9874 (3)	0.0294 (9)
C2	0.1418 (3)	0.3399 (5)	0.8981 (3)	0.0335 (9)
H4	0.465 (4)	0.694 (5)	1.189 (3)	0.043 (12)*
H2	0.382 (4)	−0.019 (5)	0.912 (3)	0.040 (12)*
H5	0.440 (5)	0.887 (7)	1.240 (5)	0.083 (17)*
H3	0.156 (5)	0.639 (6)	1.015 (4)	0.072 (15)*
H1	0.054 (4)	0.175 (5)	0.785 (4)	0.044 (14)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0504 (7)	0.0356 (6)	0.0614 (8)	0.0045 (5)	0.0185 (5)	−0.0041 (6)
O3	0.0296 (14)	0.0404 (15)	0.0424 (17)	0.0099 (12)	−0.0126 (13)	−0.0101 (13)
O2	0.0382 (15)	0.0385 (17)	0.057 (2)	0.0004 (12)	−0.0034 (14)	−0.0207 (14)
O1	0.0344 (15)	0.0366 (16)	0.0530 (19)	0.0122 (13)	−0.0066 (13)	−0.0042 (14)
N5	0.034 (2)	0.031 (2)	0.052 (2)	−0.0026 (16)	0.0027 (17)	−0.0090 (18)
N3	0.0344 (18)	0.0289 (17)	0.0325 (19)	0.0029 (14)	0.0027 (14)	−0.0003 (14)
N4	0.0332 (19)	0.0305 (18)	0.044 (2)	0.0037 (15)	0.0001 (17)	−0.0064 (16)
N2	0.0257 (17)	0.0281 (17)	0.034 (2)	0.0058 (14)	−0.0034 (14)	−0.0037 (14)
N1	0.0248 (18)	0.0354 (19)	0.037 (2)	0.0010 (15)	−0.0055 (16)	−0.0062 (16)
C5	0.039 (2)	0.0248 (19)	0.037 (2)	−0.0024 (17)	0.0101 (18)	0.0021 (17)
C4	0.0299 (19)	0.030 (2)	0.033 (2)	0.0011 (16)	0.0035 (17)	−0.0035 (17)
C3	0.027 (2)	0.033 (2)	0.036 (2)	0.0024 (17)	0.0047 (17)	0.0017 (19)
C1	0.0280 (19)	0.0268 (19)	0.029 (2)	0.0048 (15)	0.0015 (16)	0.0018 (16)
C2	0.029 (2)	0.032 (2)	0.033 (2)	0.0023 (17)	0.0000 (17)	−0.0010 (17)

Geometric parameters (Å, º)

S1—C5	1.645 (4)	N4—H3	0.88 (4)
O3—C4	1.229 (4)	N2—C4	1.368 (4)
O2—C3	1.208 (4)	N2—C3	1.380 (4)
O1—C2	1.227 (4)	N2—H2	0.86 (4)
N5—C5	1.314 (4)	N1—C2	1.360 (5)
N5—H4	0.91 (4)	N1—C3	1.362 (5)
N5—H5	0.89 (5)	N1—H1	0.70 (4)
N3—C1	1.311 (4)	C4—C1	1.460 (5)
N3—N4	1.313 (4)	C1—C2	1.460 (5)
N4—C5	1.394 (4)		

C5—N5—H4	121 (2)	N5—C5—S1	126.3 (3)
C5—N5—H5	115 (3)	N4—C5—S1	117.5 (3)
H4—N5—H5	123 (4)	O3—C4—N2	120.1 (3)
C1—N3—N4	119.1 (3)	O3—C4—C1	123.7 (3)
N3—N4—C5	120.4 (3)	N2—C4—C1	116.2 (3)
N3—N4—H3	126 (3)	O2—C3—N1	122.8 (3)
C5—N4—H3	114 (3)	O2—C3—N2	121.8 (3)
C4—N2—C3	125.8 (3)	N1—C3—N2	115.4 (3)
C4—N2—H2	119 (2)	N3—C1—C2	125.4 (3)
C3—N2—H2	115 (2)	N3—C1—C4	115.1 (3)
C2—N1—C3	127.3 (3)	C2—C1—C4	119.5 (3)
C2—N1—H1	117 (3)	O1—C2—N1	121.0 (3)
C3—N1—H1	116 (3)	O1—C2—C1	123.3 (3)
N5—C5—N4	116.1 (3)	N1—C2—C1	115.7 (3)

C1—N3—N4—C5	−178.6 (4)	O3—C4—C1—N3	5.6 (6)
N3—N4—C5—N5	5.3 (5)	N2—C4—C1—N3	−174.0 (3)
N3—N4—C5—S1	−177.8 (3)	O3—C4—C1—C2	−178.0 (4)
C3—N2—C4—O3	175.4 (4)	N2—C4—C1—C2	2.5 (5)

C3—N2—C4—C1	−5.1 (6)	C3—N1—C2—O1	178.4 (4)
C2—N1—C3—O2	178.7 (4)	C3—N1—C2—C1	−1.7 (6)
C2—N1—C3—N2	−0.4 (6)	N3—C1—C2—O1	−3.5 (6)
C4—N2—C3—O2	−175.0 (4)	C4—C1—C2—O1	−179.6 (4)
C4—N2—C3—N1	4.1 (6)	N3—C1—C2—N1	176.6 (4)
N4—N3—C1—C2	3.5 (6)	C4—C1—C2—N1	0.6 (5)
N4—N3—C1—C4	179.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H4···O2$^{\mathrm{i}}$	0.91 (4)	2.26 (4)	3.036 (4)	143 (3)
N2—H2···O3$^{\mathrm{ii}}$	0.86 (4)	1.98 (4)	2.837 (4)	173 (4)
N5—H5···O1$^{\mathrm{iii}}$	0.89 (5)	2.08 (5)	2.916 (4)	158 (4)
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