

2-(3-Methyl-5-thioxo-4,5-dihydro-1H-1,2,4-triazol-4-yl)acetic acid

Rafal Kruszynski,^{a*} Agata Trzesowska,^a Magdalena Przybycin,^a Kamil Gil^a and Maria Dobosz^b

^aInstitute of General and Ecological Chemistry, Technical University of Łódź, ul. Żeromskiego 116, 90-924 Łódź, Poland, and ^bDepartment of Organic Chemistry, Medical University, Staszica 6, 20-081 Lublin, Poland
Correspondence e-mail: rafal.kruszynski@p.lodz.pl

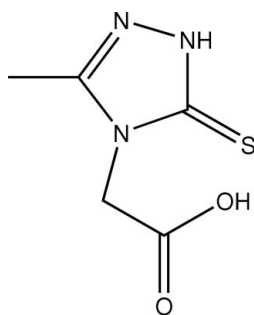
Received 15 October 2007; accepted 17 October 2007

Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.034; wR factor = 0.087; data-to-parameter ratio = 13.3.

All interatomic distances in the title compound, $\text{C}_5\text{H}_7\text{N}_3\text{O}_2\text{S}$, are normal. The 1,2,4-triazoline ring is planar and it is inclined at $78.61(7)^\circ$ to the planar acetic acid group. The molecules of the title compound are connected *via* $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds into zigzag chains along the $[101]$ direction and by $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds into a three-dimensional net. In addition, a weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond can also be found in the structure.

Related literature

For potential applications, see: Bohn & Karow (1981); Potts (1961); Santus (1980). For general synthetic procedures, see: Bany & Dobosz (1972); Veverka & Marchalin (1987). For related literature, see: Bernstein *et al.* (1995); Desiraju & Steiner (1999).



Experimental

Crystal data

 $\text{C}_5\text{H}_7\text{N}_3\text{O}_2\text{S}$
 $M_r = 173.20$

 Monoclinic, $P2_1/n$
 $a = 5.1706(3)$ Å

 $b = 9.4818(6)$ Å

 $c = 15.6763(9)$ Å

 $\beta = 99.250(5)^\circ$
 $V = 758.56(8)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.38$ mm⁻¹
 $T = 291(2)$ K

 $0.22 \times 0.07 \times 0.07$ mm

Data collection

Kuma KM-4 diffractometer

Absorption correction: numerical

 (*X-RED*; Stoe & Cie, 1999)

 $T_{\text{min}} = 0.918$, $T_{\text{max}} = 0.983$

1354 measured reflections

1354 independent reflections

 1108 reflections with $I > 2\sigma(I)$

3 standard reflections

every 100 reflections

intensity decay: 2.1%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.087$
 $S = 1.09$

1354 reflections

102 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1O}\cdots\text{N2}^{\text{i}}$	0.82	1.96	2.746 (2)	162
$\text{N1}-\text{H1N}\cdots\text{S1}^{\text{ii}}$	0.86	2.39	3.2499 (18)	176
$\text{C3}-\text{H3A}\cdots\text{O2}^{\text{iii}}$	0.97	2.60	3.567 (2)	176

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

Data collection: *KM-4 Software* (Kuma, 1993); cell refinement: *KM-4 Software*; data reduction: *DATAPROC* (Galdecki *et al.*, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL/PC* (Sheldrick, 1990b) and *ORTEP-3 for Windows* (Version 1.062; Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

This work was supported by funds allocated by the Ministry of Science and Higher Education to the Institute of General and Ecological Chemistry, Technical University of Łódź, Poland.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2545).

References

- Bany, T. & Dobosz, M. (1972). *Rocz. Chem. Ann. Soc. Chim. Pol.* **46**, 1123–1129.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bohn, R. & Karow, C. (1981). *Pharmazie*, **36**, 243–247.
- Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. IUCr Monograph on Crystallography 9. Oxford University Press.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Galdecki, Z., Kowalski, A. & Uszynski, I. (1998). *DATAPROC*. Version 10.0.4. Kuma Diffraction, Wrocław, Poland.
- Kuma (1993). *KM-4 Software*. Kuma Diffraction, Wrocław, Poland.
- Potts, K. T. (1961). *Chem. Rev.* **61**, 87–127.
- Santus, M. (1980). *Acta Pol. Pharm.* **37**, 293–300.
- Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1990b). *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Stoe & Cie (1999). *X-RED*. Version 1.18. Stoe & Cie GmbH, Darmstadt, Germany.
- Veverka, M. & Marchalin, M. (1987). *Collect. Czech. Chem. Commun.* **25**, 113–119.

supplementary materials

Acta Cryst. (2007). E63, o4378 [doi:10.1107/S1600536807051185]

2-(3-Methyl-5-thioxo-4,5-dihydro-1H-1,2,4-triazol-4-yl)acetic acid

R. Kruszynski, A. Trzesowska, M. Przybycin, K. Gil and M. Dobosz

Comment

The title compound, (I), is a member of 1,2,4-triazoline-3-thione derivatives family, known to possess antibacterial, antitumour and antiviral activity (Veverka and Marchalin, 1987; Bohn and Karow, 1981; Potts, 1961; Santus, 1980).

All interatomic distances in (I) are normal. The 1,2,4-triazoline ring of (I) can be considered as planar in the range of experimental error. The most deviating N1 atom deviates 0.0046 (13) Å from weighted least squares plane of the ring. The C3, C5, S1 atoms deviate respectively 0.082 (3), -0.004 (4), -0.049 (3) Å from this plane. The acetic acid moiety is planar (Table 1) and the most deviating atom is C4 [0.0005 (17) Å] from the weighted O1/O2/C3/C4 least-squares plane. The N3 atom deviates 0.106 (4) Å from this plane. The above mentioned weighted least-squares planes are inclined at 78.61 (7)°.

The molecules of (I) are connected *via* O1—H1O...N2 hydrogen bonds (Table 1, $C_1^1(7)$ motif (Bernstein *et al.*, 1995)) to zigzag chains extended along the [101] axis. The N1—H1N...S1 hydrogen bonds (Table 1, $R_2^2(8)$ motif) expands the chains to a folded three dimensional sheet in the (-101) plane. In (I) can be found also one C—H...O short contact (Table 1), which, according to Desiraju and Steiner (1999), can be classified as weak hydrogen bond ($C_1^1(4)$ motif).

Experimental

The title compound was synthesized according to method of Veverka and Marchalin (1987). Crystals were obtained by crystallization from mixture of water, methanol, ethanol and 2-butanone (3:1:5:1).

Refinement

The hydrogen atoms were placed in calculated positions after four cycles of anisotropic refinement and were refined as riding on the parent atom with $U_{iso}(H) = 1.2U_{eq}(C\text{-non-methyl or N})$ and $U_{iso}(H) = 1.5U_{eq}(C\text{-methyl or O})$. The methyl and hydroxyl group was allowed to rotate about its local threefold axis (AFIX 137 and 147 respectively).

Figures

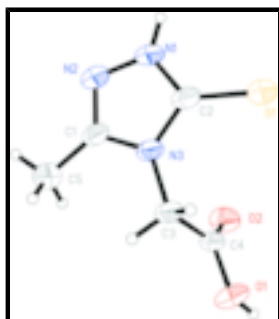


Fig. 1. Molecular structure of the title compound (I). Displacement ellipsoids are drawn at the 50% probability level.

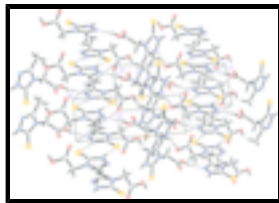


Fig. 2. A part of the molecular packing of the title compound. Hydrogen bonds are indicated by dashed lines.

2-(3-Methyl-5-thioxo-4,5-dihydro-1H-1,2,4-triazol-4-yl)acetic acid

Crystal data

$C_5H_7N_3O_2S$

$M_r = 173.20$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1/n$

$a = 5.1706\ (3)\ \text{\AA}$

$b = 9.4818\ (6)\ \text{\AA}$

$c = 15.6763\ (9)\ \text{\AA}$

$\beta = 99.250\ (5)^\circ$

$V = 758.56\ (8)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 360$

$D_x = 1.517\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 99 reflections

$\theta = 2\text{--}20^\circ$

$\mu = 0.38\ \text{mm}^{-1}$

$T = 291\ (2)\ \text{K}$

Needle, colourless

$0.22 \times 0.07 \times 0.07\ \text{mm}$

Data collection

Kuma KM-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291\ (2)\ \text{K}$

$\omega\text{--}2\theta$ scans

Absorption correction: numerical
(X-RED; Stoe & Cie, 1999)

$T_{\min} = 0.918$, $T_{\max} = 0.983$

1354 measured reflections

1354 independent reflections

1108 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.0000$

$\theta_{\max} = 25.1^\circ$

$\theta_{\min} = 3.4^\circ$

$h = -6\text{--}6$

$k = 0\text{--}11$

$l = 0\text{--}18$

3 standard reflections

every 100 reflections

intensity decay: 2.1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.087$

$S = 1.09$

Secondary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

1354 reflections $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
 102 parameters $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.99527 (11)	1.04539 (6)	0.63708 (3)	0.0393 (2)
O1	0.7717 (3)	0.88549 (17)	0.88096 (9)	0.0437 (4)
H1O	0.8832	0.8517	0.9185	0.066*
C4	0.8051 (4)	0.8362 (2)	0.80424 (12)	0.0301 (5)
O2	0.9822 (3)	0.76167 (17)	0.79093 (9)	0.0420 (4)
N1	0.7338 (3)	0.8532 (2)	0.52587 (10)	0.0401 (5)
H1N	0.8106	0.8759	0.4831	0.048*
N3	0.6155 (3)	0.84909 (19)	0.64966 (10)	0.0313 (4)
C3	0.5851 (4)	0.8886 (2)	0.73708 (12)	0.0336 (5)
H3A	0.4208	0.8508	0.7495	0.040*
H3B	0.5763	0.9905	0.7407	0.040*
C2	0.7812 (4)	0.9166 (2)	0.60293 (12)	0.0335 (5)
N2	0.5497 (4)	0.7480 (2)	0.52213 (11)	0.0413 (5)
C1	0.4797 (4)	0.7476 (2)	0.59811 (12)	0.0347 (5)
C5	0.2828 (4)	0.6513 (3)	0.62525 (15)	0.0438 (6)
H5A	0.2063	0.5947	0.5769	0.066*
H5B	0.1484	0.7056	0.6456	0.066*
H5C	0.3661	0.5911	0.6708	0.066*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0452 (3)	0.0464 (4)	0.0274 (3)	-0.0021 (3)	0.0090 (2)	-0.0016 (2)
O1	0.0578 (10)	0.0515 (10)	0.0206 (8)	0.0089 (8)	0.0029 (7)	-0.0014 (7)
C4	0.0347 (11)	0.0345 (11)	0.0222 (10)	-0.0032 (9)	0.0082 (8)	0.0018 (9)
O2	0.0366 (8)	0.0546 (10)	0.0356 (8)	0.0104 (8)	0.0087 (7)	0.0068 (7)
N1	0.0495 (11)	0.0521 (12)	0.0202 (9)	-0.0065 (10)	0.0095 (8)	0.0007 (8)
N3	0.0322 (9)	0.0425 (10)	0.0198 (8)	0.0043 (8)	0.0065 (7)	0.0005 (8)

supplementary materials

C3	0.0347 (11)	0.0459 (13)	0.0212 (10)	0.0065 (10)	0.0073 (8)	-0.0003 (9)
C2	0.0339 (11)	0.0451 (13)	0.0216 (10)	0.0079 (10)	0.0049 (8)	0.0022 (9)
N2	0.0481 (11)	0.0523 (12)	0.0231 (9)	-0.0059 (10)	0.0042 (8)	-0.0024 (8)
C1	0.0366 (11)	0.0445 (13)	0.0220 (10)	0.0046 (10)	0.0015 (8)	0.0012 (9)
C5	0.0442 (13)	0.0501 (14)	0.0375 (13)	-0.0011 (11)	0.0077 (10)	0.0021 (11)

Geometric parameters (Å, °)

S1—C2	1.677 (2)	N3—C1	1.374 (3)
O1—C4	1.327 (2)	N3—C3	1.453 (2)
O1—H1O	0.8200	C3—H3A	0.9700
C4—O2	1.201 (2)	C3—H3B	0.9700
C4—C3	1.505 (3)	N2—C1	1.299 (3)
N1—C2	1.336 (3)	C1—C5	1.480 (3)
N1—N2	1.374 (3)	C5—H5A	0.9600
N1—H1N	0.8600	C5—H5B	0.9600
N3—C2	1.372 (3)	C5—H5C	0.9600
C4—O1—H1O	109.5	H3A—C3—H3B	107.8
O2—C4—O1	125.34 (19)	N1—C2—N3	103.51 (18)
O2—C4—C3	125.76 (18)	N1—C2—S1	129.18 (17)
O1—C4—C3	108.89 (17)	N3—C2—S1	127.30 (15)
C2—N1—N2	112.98 (17)	C1—N2—N1	104.81 (17)
C2—N1—H1N	123.5	N2—C1—N3	110.26 (19)
N2—N1—H1N	123.5	N2—C1—C5	124.8 (2)
C2—N3—C1	108.44 (16)	N3—C1—C5	124.95 (18)
C2—N3—C3	123.71 (18)	C1—C5—H5A	109.5
C1—N3—C3	127.75 (17)	C1—C5—H5B	109.5
N3—C3—C4	112.98 (16)	H5A—C5—H5B	109.5
N3—C3—H3A	109.0	C1—C5—H5C	109.5
C4—C3—H3A	109.0	H5A—C5—H5C	109.5
N3—C3—H3B	109.0	H5B—C5—H5C	109.5
C4—C3—H3B	109.0		
N3—C3—C4—O1	175.46 (17)	N3—C3—C4—O2	-4.6 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1O \cdots N2 ⁱ	0.82	1.96	2.746 (2)	162
N1—H1N \cdots S1 ⁱⁱ	0.86	2.39	3.2499 (18)	176
C3—H3A \cdots O2 ⁱⁱⁱ	0.97	2.60	3.567 (2)	176

Symmetry codes: (i) $x+1/2, -y+3/2, z+1/2$; (ii) $-x+2, -y+2, -z+1$; (iii) $x-1, y, z$.

Fig. 1

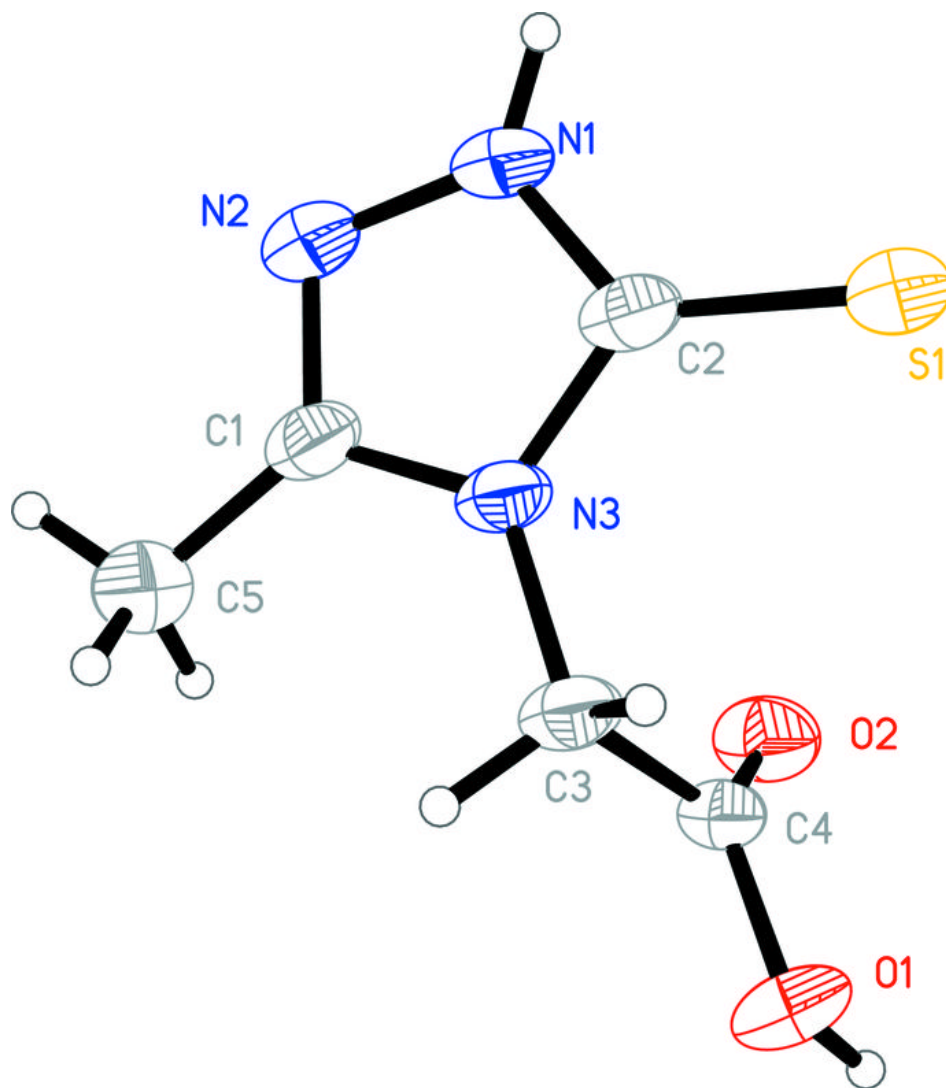


Fig. 2

