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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(C-C)$ = 0.003 Å

R factor = 0.054

wR factor = 0.151

Data-to-parameter ratio = 14.9

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-Methyl-N-(4-oxo-2-thioxoimidazolidin-3-yl)-
1,2,3-thiadiazole-5-carboxamide

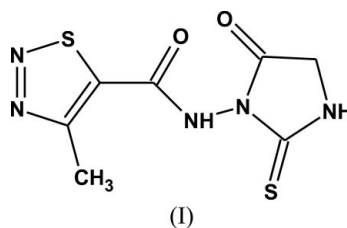
The molecule of the title compound, $C_7H_7N_5O_2S_2$, is non-planar. The dihedral angles formed by the thiohydantoin and methylthiadiazole systems with the amide group are 85.5 (1) and 38.4 (3)°, respectively. The crystal structure is stabilized by intermolecular $N-H \cdots N/O$ hydrogen bonds and short $S \cdots O$ contacts, forming a three-dimensional network.

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Comment

In a continuation of our studies on the intramolecular dehydrative cyclization of acyl thiosemicarbazide derivatives, we have reported the synthesis of some new 1,2,4-triazole and 1,3,4-thiadiazole derivatives (Siwek *et al.*, 2006). It was expected that the synthetic procedures described by Dobosz *et al.* (1996) should be similarly applicable to the synthesis of 5-(4-methyl-1,2,3-thiadiazol-5-yl)-1,3,4-thiadiazole-2-aminoacetic acid. However, the cyclization of 4-ethoxycarbonylmethyl-1-(4-methyl-1,2,3-thiadiazol-5-ylcarbonyl)thiosemicarbazide furnished, in high yield, an unexpected compound, *viz.* the title compound, (I). The results of an elemental analysis and spectroscopic data (IR and ¹H NMR) were not sufficient to elucidate the structure, so an X-ray diffraction study was undertaken.



This showed that the molecule of (I) consists of a 2-thiohydantoin ring substituted in the 3-position by a (4-methyl-1,2,3-thiadiazol-5-yl)aminocarbonyl system. Fig. 1 shows a displacement ellipsoid representation of (I). The bond lengths are within normal ranges (Allen *et al.*, 1987). The thiadiazole and thiohydantoin rings are each essentially planar, the maximum deviation from the least-squares planes being 0.013 (2) Å for atom N4 in the thiohydantoin ring. The dihedral angles formed by the thiohydantoin and methylthiadiazole systems with the amide group are 85.5 (1) and 38.4 (3)°, respectively. In the crystal structure, a pair of molecules forms centrosymmetric dimers linked by $N5-H5N \cdots O1^{ii}$ hydrogen bonds (symmetry codes as in Table 2). Dimers linked by $N3-H3N \cdots N1^i$ hydrogen bonds produce two-dimensional layers parallel to the (120) plane (Fig. 2) The layers are linked by $N5-H5N \cdots N2^{iii}$ hydrogen bonds and short $S1 \cdots O2(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$ contacts of 3.147 (3) Å.

Experimental

4-Ethoxycarbonylmethyl-1-(4-methyl-1,2,3-thiadiazol-5-ylcarbonyl)-thiosemicarbazide (0.01 mol) was dissolved in anhydrous acetic acid (10 ml) and the reaction mixture was refluxed for 9 h. It was then kept at room temperature to complete the precipitation of the product, which was filtered, dried and crystallized from ethanol.

Crystal data

$C_7H_7N_5O_2S_2$
 $M_r = 257.30$
 Monoclinic, $P2_1/c$
 $a = 7.528$ (2) Å
 $b = 7.239$ (1) Å
 $c = 19.843$ (4) Å
 $\beta = 91.74$ (3)°
 $V = 1080.8$ (4) Å³
 $Z = 4$

$D_x = 1.581$ Mg m⁻³
 Cu $K\alpha$ radiation
 Cell parameters from 66 reflections
 $\theta = 6\text{--}21^\circ$
 $\mu = 4.46$ mm⁻¹
 $T = 293$ (2) K
 Prism, orange
 $0.6 \times 0.4 \times 0.1$ mm

Data collection

Kuma KM-4 four-circle diffractometer
 ω - 2θ scans
 Absorption correction: spherical (Dwiggins, 1975)
 $T_{\min} = 0.035$, $T_{\max} = 0.135$
 2227 measured reflections
 2184 independent reflections
 1640 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$
 $\theta_{\text{max}} = 75.1^\circ$
 $h = -9 \rightarrow 9$
 $k = -9 \rightarrow 0$
 $l = -24 \rightarrow 0$
 3 standard reflections every 100 reflections
 intensity decay: 0.5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.151$
 $S = 1.06$
 2184 reflections
 147 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1205P)^2 + 0.0129P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.62$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.46$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0078 (13)

Table 1

Selected bond lengths (Å).

S1–N2	1.652 (2)	N4–C7	1.389 (3)
S1–C3	1.676 (2)	N5–C9	1.332 (3)
S2–C9	1.644 (2)	N5–C8	1.446 (3)
N1–N2	1.290 (3)	O2–C7	1.200 (3)
N1–C4	1.371 (3)	C3–C4	1.374 (3)
N3–N4	1.378 (2)	C7–C8	1.500 (3)
N4–C9	1.382 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N3-H3N\cdots N1^i$	0.98	1.96	2.912 (3)	163
$N5-H5N\cdots O1^{ii}$	0.99	2.21	2.933 (3)	129
$N5-H5N\cdots N2^{iii}$	0.99	2.28	3.118 (3)	142

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

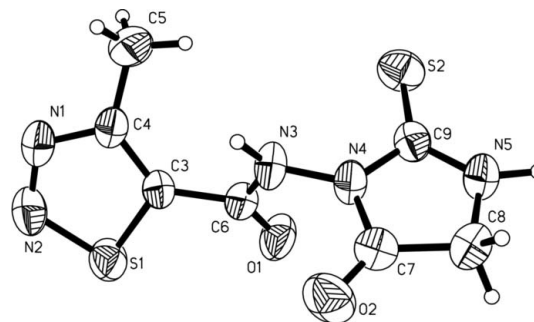


Figure 1

Perspective view of (I). Displacement ellipsoids are drawn at the 50% probability level.

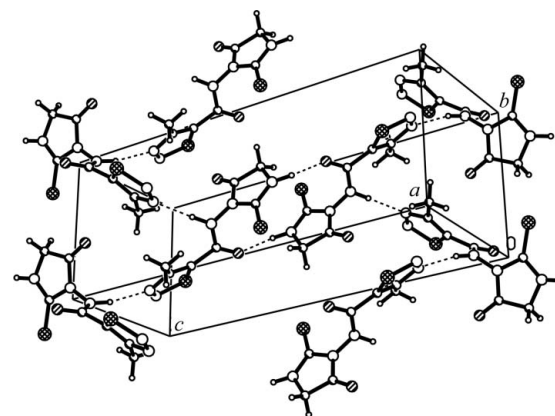


Figure 2

Part of the crystal structure of (I), showing the formation of a layer. Dashed lines indicate hydrogen bonds.

H atoms bonded to C were positioned geometrically, while those bonded to N atoms were found in difference maps. All H atoms were included in the refinement in the riding-model approximation, with $U_{\text{iso}}(\text{H})$ values constrained to be $1.2U_{\text{eq}}(\text{C or N})$, and with $C-H = 0.96\text{--}0.97$ Å and $N-H = 0.98\text{--}0.99$ Å.

Data collection: *KM-4 Software* (Kuma, 1991); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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