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1-Acetyl-2-thiohydantoin

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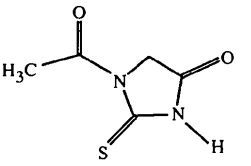
Abstract

In the title compound (1-acetyl-4-oxoimidazolidine-2-thione, $C_5H_6N_2O_2S$), the plane of the acetyl group forms an angle of 6.7° with the essentially planar thiohydantoin ring. N—H···O hydrogen bonds create quasi-planar chains of molecules along the *y* axis.

Comment

The structure of the title compound, (I), has been established as part of a study of the synthesis and characterization of metal complexes of 2-thiohydantoin and its derivatives (Casas *et al.*, 1995).



(I)

The molecular structure of (I) is shown in Fig. 1. The N1—C1—N2—C3—C2 ring and the peripheral S, C4 and O3 atoms define a plane (r.m.s. deviation 0.013 Å), as does the *N*-acetyl N1—C4(=O4)—C5 fragment (r.m.s. deviation 0.006 Å). The angle between the two planes is 6.7°.

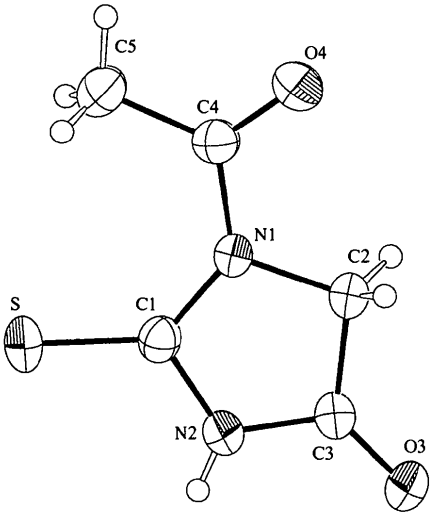


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme and 50% probability displacement ellipsoids.

The bond lengths and angles in the acetyl fragment are similar to those found in 1-acetyl-2-[1-(acetylthio)-ethyl]thiohydantoin (MacKay *et al.*, 1992), although in this C2-substituted thiohydantoin, the angle between the thiohydantoin ring and the acetyl group is 12°. In (I), the thiohydantoin ring bond lengths differing most from those found in 2-thiohydantoin (Devillanova *et al.*, 1987; Walker *et al.*, 1969) are those of C1—N1 and N1—C2, which are longer in the acetyl derivative. The internal ring angle most affected by *N*-acetylation is C2—N1—C1, which widens slightly to approximately the same value as in 1-acetyl-2-[1-(acetylthio)ethyl]thiohydantoin (MacKay *et al.*, 1992). *N*-Acetylation also affects the external angles flanking the C=S group, with N1—C1—S becoming wider and N2—C1—S narrower.

The N2—H2 bond and the O3 atom are involved in a hydrogen bond [N2—H2 0.77 (3), H2···O3^{i} 2.08 (4), N2···O3^{i} 2.849 (3) Å and N2—H2···O3^{i} 170 (3)°; sym-

metry code: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$] which links the molecules in quasi-planar chains along the y axis. Thus, *N*-acetylation simplifies the hydrogen-bond network found in 2-thiohydantoin which creates two-dimensional sheets rather than ribbons (Walker *et al.*, 1969).

Experimental

The title compound was prepared by refluxing 3.32 g of thiohydantoin in 20 ml of acetic anhydride for 30 min. The solid formed was filtered off and washed with ethyl ether. Crystals were obtained by slow evaporation of an acetone solution.

Crystal data

$C_5H_6N_2O_2S$
$M_r = 158.18$
Monoclinic
$P2_1/n$
$a = 8.2968\ (11)$ Å
$b = 7.7364\ (11)$ Å
$c = 10.6066\ (15)$ Å
$\beta = 93.434\ (11)°$
$V = 679.6\ (2)$ Å^3
$Z = 4$
$D_x = 1.546$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.07–18.41°$
$\mu = 0.411$ mm^{-1}
$T = 293\ (2)$ K
Prism
0.20 × 0.15 × 0.15 mm
Colourless

Data collection

Enraf–Nonius MACH3 diffractometer
ω scans
Absorption correction: none
1458 measured reflections
1384 independent reflections
1026 reflections with $I > 2\sigma(I)$

$R_{int} = 0.031$
$\theta_{max} = 26.29°$
$h = -10 \rightarrow 10$
$k = -9 \rightarrow 0$
$l = 0 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.115$
$S = 1.038$
1384 reflections
116 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.2702P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.214$ e Å^{-3}
$\Delta\rho_{min} = -0.230$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.035 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S—C1	1.638 (2)	N1—C2	1.469 (3)
O3—C3	1.218 (3)	N2—C3	1.362 (3)
O4—C4	1.208 (3)	N2—C1	1.381 (3)
N1—C1	1.370 (3)	C2—C3	1.492 (3)
N1—C4	1.409 (3)	C4—C5	1.486 (4)
C1—N1—C4	131.4 (2)	N1—C2—C3	102.5 (2)
C1—N1—C2	111.3 (2)	O3—C3—N2	125.4 (2)
C4—N1—C2	117.4 (2)	O3—C3—C2	128.2 (2)
C3—N2—C1	113.6 (2)	N2—C3—C2	106.4 (2)
N1—C1—N2	106.1 (2)	O4—C4—N1	117.3 (2)
N1—C1—S	132.0 (2)	O4—C4—C5	122.3 (2)
N2—C1—S	121.9 (2)	N1—C4—C5	120.3 (2)

Data collection: *CAD-4 EXPRESS Software* (Enraf–Nonius, 1995). Cell refinement: *CAD-4 EXPRESS Software*. Data reduction: *HELENA* (Spek, 1996). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1996). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1500). Services for accessing these data are described at the back of the journal.

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(3*S*)-4,4-Dimethyl-2-oxotetrahydrofuran-3-yl (2*S*)-2-(1,4-Benzodioxin-6-yl)propionate

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Abstract

The 2-oxofuran moiety in the title compound, $C_{17}H_{18}O_6$, has a skew-envelope form. The heterocycle of the 1,4-benzodioxin-6-yl moiety has the typical half-chair form and steric hindrance between the substituents produces