

## 3-(Imidazolidin-2-ylidene)-6-methyl-3,4,5,6-tetrahydro-2H-pyran-2,4-dione

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## Key indicators

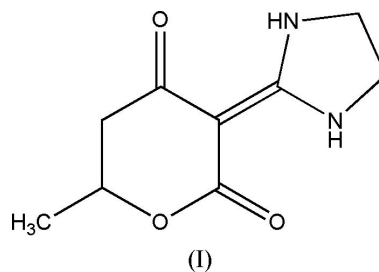
Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
Disorder in main residue  
 $R$  factor = 0.045  
 $wR$  factor = 0.138  
Data-to-parameter ratio = 10.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_9\text{H}_{12}\text{N}_2\text{O}_3$ , was synthesized by the reaction in ethanol of ethane-1,2-diamine and 3-[bis(methylthio)methylene]-6-methyl-3,4,5,6-tetrahydro-2H-pyran-2,4-dione. Two intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds induce a high degree of planarity.

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## Comment

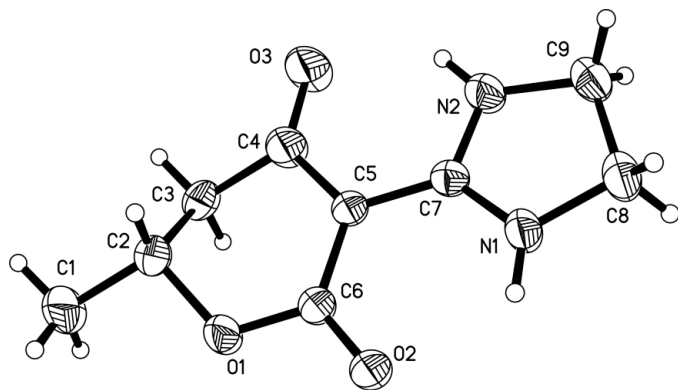
In recent years, there have been a few reports of pyranone derivatives. Some patents report that pyrandione derivatives have inhibiting activity for HIV proteinase (Ellsworth & Lunney, 1995; Thaisrivongs & Yang, 1994). Some bioactivities, such as tobacco virucidal activity, plant growth regulation activity, and fungicidal and herbicidal activities, have also been reported (Li *et al.*, 2004). Significant activities against *Biomphalaria glabrata* egg masses have been reported (de Souza *et al.*, 2004). Nitrogen heterocycles often exhibit some special biological activities, *e.g.* insecticidal, fungicidal and herbicidal (Hou *et al.*, 2003). In view of these facts and in a continuation of our interest in the chemistry of heterocycles, we attempted to synthesize a series of 6-methyl-3,4,5,6-tetrahydro-2H-pyran-2,4-dione derivatives. The title compound, (I), was studied to investigate the relationship between structure and bioactivity. To the best of our knowledge, this is the first reported crystal structure containing the 6-methyl-3,4,5,6-tetrahydro-2H-pyran-2,4-dione ring system.



The molecular structure of (I) is shown in Fig. 1. The X-ray analysis reveals that there are two intramolecular hydrogen bonds, which induce coplanarity of most of the atoms; of the non-H atoms, only C1, C2 and C3 do not lie on a crystallographic mirror plane, and these three atoms are disordered across the plane, together with their attached H atoms. In the crystal structure, glide-related molecules are linked by intermolecular hydrogen bonds and other weak interactions.

## Experimental

The title compound was synthesized by adding ethane-1,2-diamine (0.060 g, 1 mmol) to an absolute ethanol solution (30 ml) of 3-[bis-



**Figure 1**  
A view of compound (I) with the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

(methylthio)methylene]-6-methyl-3,4,5,6-tetrahydro-2*H*-pyran-2,4-dione (0.232 g, 1 mmol). The mixture was stirred for 2 h at room temperature. The product was obtained by silica-gel column chromatography using a 1:3 mixture of ethyl acetate and petroleum ether as eluant. Colourless single crystals suitable for X-ray diffraction analysis were obtained by diffusion of *n*-hexane into a solution of the crude product in dichloromethane.

#### Crystal data

$C_9H_{12}N_2O_3$	$D_x = 1.398 \text{ Mg m}^{-3}$
$M_r = 196.21$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/m$	Cell parameters from 920 reflections
$a = 7.195 (2) \text{ \AA}$	$\theta = 2.2\text{--}25.8^\circ$
$b = 7.106 (2) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 9.131 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 92.866 (5)^\circ$	Block, colourless
$V = 466.3 (2) \text{ \AA}^3$	$0.40 \times 0.10 \times 0.10 \text{ mm}$
$Z = 2$	

#### Data collection

Bruker SMART CCD area-detector diffractometer	1030 independent reflections
$\varphi$ and $\omega$ scans	701 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.947$ , $T_{\text{max}} = 0.989$	$\theta_{\text{max}} = 26.4^\circ$
2713 measured reflections	$h = -8 \rightarrow 8$
	$k = -8 \rightarrow 7$
	$l = -11 \rightarrow 10$

#### Refinement

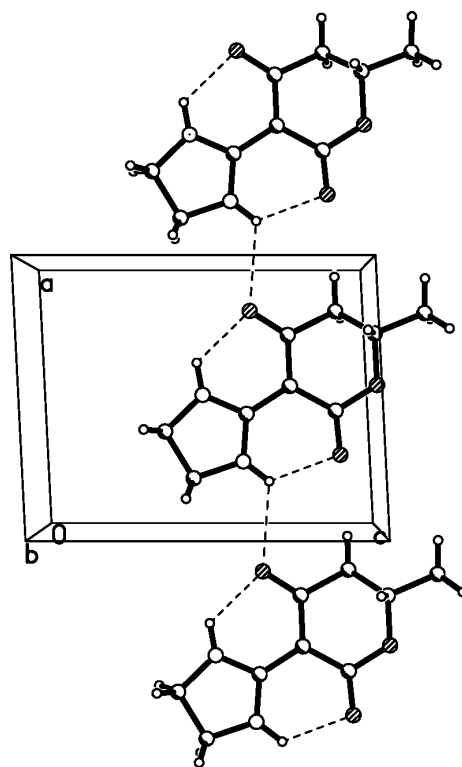
Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.0755P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.138$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
1030 reflections	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
98 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**Table 1**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$N1\text{--}H1A\cdots O2$	0.864 (10)	2.10 (3)	2.681 (3)	124 (2)
$N2\text{--}H2A\cdots O3$	0.867 (10)	1.96 (2)	2.655 (3)	137 (3)
$N1\text{--}H1A\cdots O3^i$	0.864 (10)	2.52 (3)	3.081 (3)	123 (2)

Symmetry code: (i)  $1 + x, y, z$ .



**Figure 2**  
Hydrogen bonds (dashed lines) in the crystal structure of (I), viewed along the *b* axis.

The amine H atoms were located in a difference map and were refined isotropically with the restraint  $N\text{--}H = 0.86 (1) \text{ \AA}$ . All other H atoms were placed in calculated positions ( $C\text{--}H = 0.96 \text{ \AA}$ ) and included in the refinement using a riding model, with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$  [ $1.5U_{\text{eq}}(C)$  for methyl H atoms]. Atoms C1, C2 and C3, together with their attached H atoms, are disordered equally over two positions across the mirror plane passing through the rest of the molecule.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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