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5-(5-Ethyl-1,3,4-thiadiazol-2-ylaminomethylene)-2,2-dimethyl-1,3-dioxane- 4,6-dione

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In the title compound, $C_{11}H_{13}N_3O_4S$, the thiadiazole ring is nearly planar, while the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The NH group makes one intramolecular contact with a carbonyl group, forming a six-membered ring. The same functional group is involved in an intermolecular N—H···N hydrogen bond.

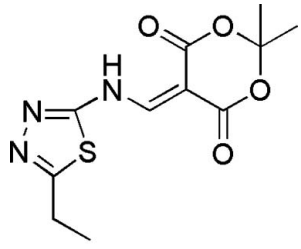
Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The determination of the structure of the title compound, (I), is part of a continuing study on conformational analysis in the solid state of Meldrum's acid derivatives (Joussef *et al.*, 2005*a*,*b*), within a project to investigate potential antiviral and antiparasitic activities of these compounds.



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The torsion angle C5—N6—C7—C8 is 179.9 (2)° and the distances N6···C7 and C7···C8 (Table 1) indicate delocalization of the conjugated system. The H atom of the NH group has one intramolecular contact to O9 (Table 2), forming an S(6) ring. The delocalization of the N atom lone pair into the Meldrum's acid ring may be favoured in the direction of one of the two available carbonyl groups C9═O9 and C13═O13 (Blake *et al.*, 2003). Finally, the same NH group is also involved in a weak intermolecular N—H···N contact.

Experimental

The title compound was prepared according to a literature procedure (Cassis *et al.*, 1985) and was recrystallized from methanol.

Crystal data

$C_{11}H_{13}N_3O_4S$
M_r = 283.30
Orthorhombic, *Pbca*
a = 10.733 (2) Å
b = 9.842 (1) Å
c = 24.650 (2) Å
V = 2603.9 (6) Å^3
Z = 8
D_x = 1.445 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 5.4–13.7°
μ = 0.26 mm^{-1}
T = 299 (2) K
Prism, colourless
0.40 × 0.40 × 0.13 mm

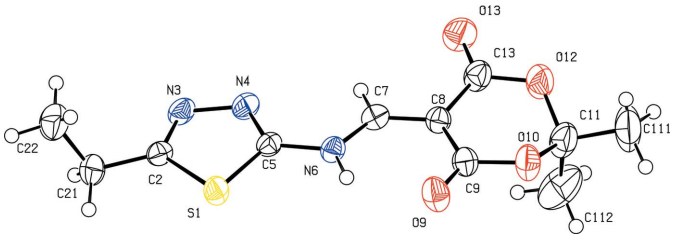


Figure 1
The molecular structure of (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level for non-H atoms.

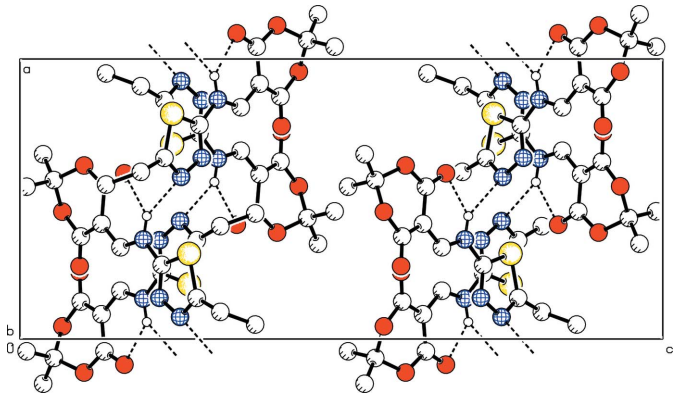


Figure 2
The molecular packing of (I), with hydrogen bonds shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.878, T_{max} = 0.974
2543 measured reflections
2543 independent reflections
1700 reflections with $I > 2\sigma(I)$
θ_{max} = 26.0°
$h = -13 \rightarrow 0$
$k = -12 \rightarrow 0$
$l = 0 \rightarrow 30$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.116
S = 1.04
2543 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 0.4796P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected bond lengths (Å).

S1—C5	1.722 (2)	N6—C7	1.334 (3)
S1—C2	1.727 (2)	C7—C8	1.368 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O9	0.93	2.04	2.716 (3)	128
N6—H6···N3^{i}	0.93	2.28	3.098 (3)	146

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

All H atoms were positioned with idealized geometry and refined with fixed isotropic displacement parameters (set at 1.5U_{eq} of the parent atom for methyl groups and at 1.2U_{eq} of the parent atom for all others), using a riding model, with N—H = 0.93 Å and C—H = 0.93 (aromatic), 0.97 (methylene) or 0.96 Å (methyl).

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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