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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.041
wR factor = 0.103
Data-to-parameter ratio = 13.6

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

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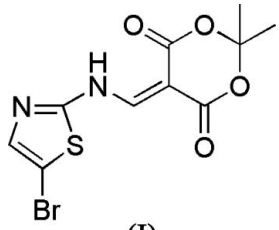
In the title compound, $C_{10}H_9BrN_2O_4S$, the 1,3-dioxane-4,6-dione ring exhibits a sofa conformation. The thiazole ring is nearly planar. The amino H atom has an intramolecular N—H···O contact, forming a six-membered ring, and an intermolecular hydrogen bond to a carbonyl O is also observed.

Comment

Thiazoles comprise an important class of heterocyclic compounds present in many potent biologically active molecules. This heterocyclic ring also shows various application in the synthesis of new organic compounds (de Souza *et al.*, 2005). We have focused on arylaminomethylene derivatives of Meldrum's acid in our search for biologically active molecules (Joussef *et al.*, 2005*a*,*b*; da Silva *et al.*, 2005*a*,*b*, 2006). As an extension of this approach, we report an X-ray crystallographic study of the title compound, (I).



(I)

In (I), the 1,3-dioxane-4,6-dione ring exhibits a sofa conformation. The torsion angle C5—N6—C7—C8 is 176.5 (4)° and the distances N6—C5 and C7—C8 indicate delocalization of the conjugated system. The amino H atom forms an intramolecular contact to O9, with an H···O distance of 2.24 Å, forming a six-membered ring. Delocalization of the N-atom lone pair into the Meldrum's acid ring may be favoured in the direction of one of the two carbonyl groups (Blake *et al.*, 2003); details of the hydrogen bonding are given in Table 1.

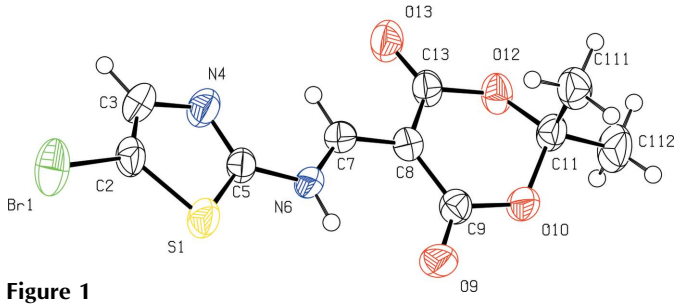


Figure 1
The molecular structure of (I), showing the atom labeling and displacement ellipsoids drawn at the 50% probability level.

Experimental

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

Crystal data

$C_{10}H_9BrN_2O_4S$
M_r = 333.16
Monoclinic, $P2_1/c$
a = 5.408 (5) Å
b = 18.013 (5) Å
c = 13.114 (5) Å
β = 100.717 (5)°
V = 1255.2 (13) Å^3
Z = 4
D_x = 1.763 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 5.5–23.7°
μ = 3.45 mm^{-1}
T = 293 (2) K
Prism, colorless
0.50 × 0.16 × 0.13 mm

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.504, T_{max} = 0.641
2330 measured reflections
2230 independent reflections
1269 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.1°
$h = -6 \rightarrow 6$
$k = -21 \rightarrow 0$
$l = -15 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.103
S = 1.01
2230 reflections
164 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.077P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O9	0.87	2.24	2.798 (5)	122
N6—H6···O9^{i}	0.87	2.18	2.996 (5)	156

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

All H atoms were positioned with idealized geometry and were refined with isotropic displacement parameters (set at 1.2 times U_{eq} of the parent atom or at 1.5 times U_{eq} for methyl groups) using a riding model, with N—H = 0.87 Å, and C—H = 0.93 (aromatic) or 0.96 Å (methyl). The isotropic displacement parameter of the amino H atom was refined.

Data collection: *CAD-4/PC Software* (Nonius, 1993); cell refinement: *CAD-4/PC Software*; 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*.

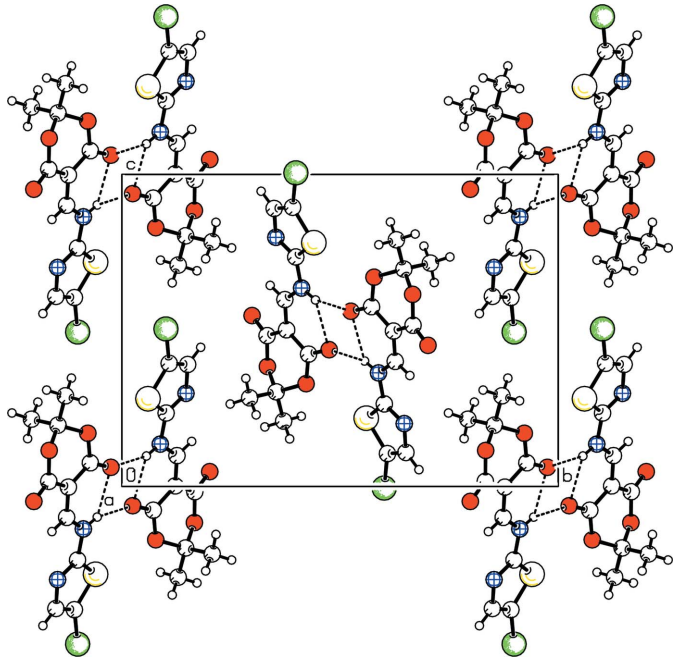


Figure 2
The molecular packing of (I), with hydrogen bonds shown as dashed lines.

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