Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2-Dimethyl-5-[(4-methylthiazol-2-ylamino)-methylene]-1,3-dioxane-4,6-dione

Luiz Everson da Silva,[a,b] Antonio Carlos Joussef,[a] Carla Regina Andrighetti-Fröhner,[a] Cláudia Maria Oliviera Simões[c] and Adailton José Bortoluzzi[a]*

[a]Departamento de Química–UFSC, 88040-900 Florianópolis, SC, Brazil, [b]Clemens-Schöpf-Institut für Organische Chemie und Biochemie, Technische Universität Darmstadt, Petersenstrasse 22, D-64287 Darmstadt, Germany, and [c]Departamento de Ciências Farmacêuticas–UFSC, 88040-900 Florianópolis, SC, Brazil

Correspondence e-mail: luiz_everson@yahoo.de

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.125
Data-to-parameter ratio = 13.2

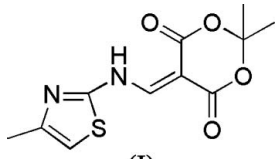
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 February 2006
Accepted 18 February 2006

In the title compound, $C_{11}H_{12}N_2O_4S$, the thiazole ring is nearly planar. The 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The NH group forms an intramolecular contact to a carbonyl O atom, forming a six-membered ring, and an intermolecular N—H···O hydrogen bond, with an H···O distance of 2.16 Å, is also observed.

Comment

Thiazoles constitute an important class of heterocyclic compounds for which pharmalogical properties have been reported (Krasovsky *et al.*, 2002). 5-Arylaminemethylene Meldrum's acid analogs play an important role in heterocyclic chemistry as pivotal intermediates in the formation of cyclic products (Chen, 1991). As part of a continuing study of the conformation in the solid state of 5-aminomethylene Meldrum's acid derivatives (Joussef *et al.*, 2005*a*,*b*; da Silva *et al.*, 2005*a*,*b*, 2006), 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 half-chair conformation. The dihedral angle C11—N1—C2—C3 is 174.5 (2)° and the distances N1—C11 and C2—C3 indicate delocalization of the conjugated system. The amino H atom has an intramolecular contact to O1, with an H···O distance of 2.24 Å, forming a six-membered 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 carbonyl groups (Blake *et al.*, 2003). Details of the hydrogen bonding are given in Table 1.

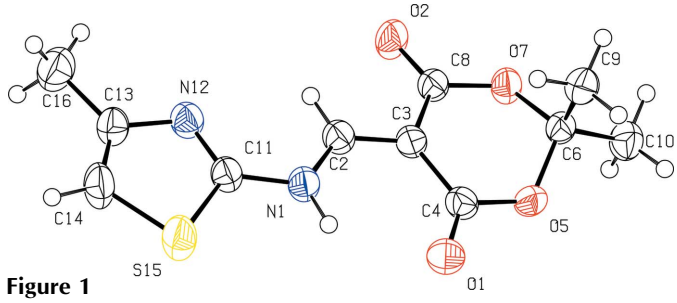


Figure 1
The molecular structure of (I), with the atom labeling and displacement ellipsoids drawn at the 50% probability level.

Experimental

The title compound was prepared according to a literature procedure (Cassis *et al.*, 1985) and was recrystallized from methanol (m.p.: 468–470 K).

Crystal data

$C_{11}H_{12}N_2O_4S$
$M_r = 268.29$
Monoclinic, $P2_1/c$
$a = 5.6384\ (6)$ Å
$b = 18.562\ (4)$ Å
$c = 11.920\ (2)$ Å
$\beta = 103.14\ (1)^\circ$
$V = 1214.9\ (3)$ Å^3
$Z = 4$
$D_x = 1.467$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.0$–18.1°
$\mu = 0.28$ mm^{-1}
$T = 293\ (2)$ K
Irregular block, yellow
$0.36 \times 0.26 \times 0.23$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2249 measured reflections
2144 independent reflections
1590 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.1^\circ$
$h = -6 \rightarrow 6$
$k = -22 \rightarrow 0$
$l = -14 \rightarrow 0$
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.125$
$S = 1.04$
2144 reflections
163 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.5695P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.91	2.24	2.836 (3)	123
N1—H1···O1^{i}	0.91	2.16	2.996 (3)	154

Symmetry code: (i) $-x+2, -y+1, -z+2$.

All H atoms were positioned with idealized geometry and were refined with isotropic displacement parameters (set to 1.2 times U_{eq} of the parent atom, 1.5 for methyl groups) using a riding model with N—H = 0.91 Å, C—H = 0.93 Å (aromatic), 0.96 Å (methyl).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; 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*.

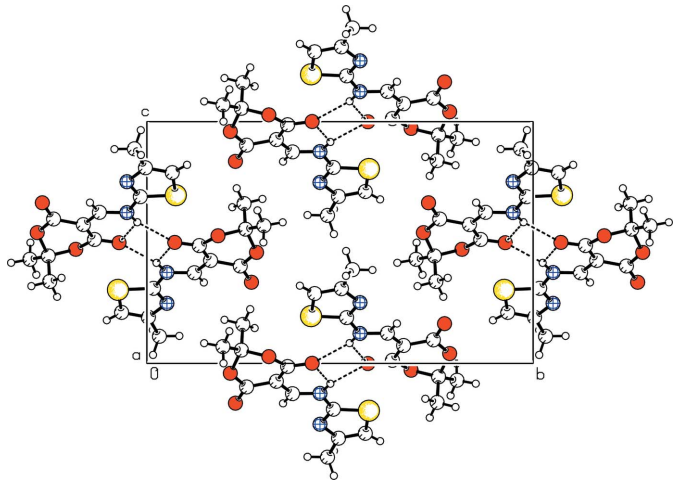


Figure 2
Molecular packing of (I), with hydrogen bonds shown as dashed lines.

The authors thank Sabine Foro, TU–Darmstadt, Germany, for her help and advice.

References

Blake, A. J., McNab, H. & Withell, K. (2003). *Acta Cryst.* E**59**, o841–o842.
Cassis, R., Tapia, R. & Valderrama, J. A. (1985). *Synth. Commun.* **15**, 125–129.
Chen, B. C. (1991). *Heterocycles*, **32**, 529–597.
Enraf–Nonius (1993). *CAD-4 EXPRESS*. Version 1.2. Enraf-Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Joussef, A. C., da Silva, L. E., Bortoluzzi, A. J. & Foro, S. (2005*a*). *Acta Cryst.* E**61**, o2642–o2643.
Joussef, A. C., da Silva, L. E., Bortoluzzi, A. J. & Foro, S. (2005*b*). *Acta Cryst.* E**61**, o2873–o2874.
Krasovsky, A. L. Moiseev, A. M., Nenajdenko, V. G. & Balenkova, E. S. (2002). *Chem. Heterocycl. Compds*, **38**, 231–237.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Silva, L. E. da, Joussef, A. C., Foro, S., Schmidt, B. (2006). *Acta Cryst.* E**62**, o742–743.
Silva, L. E. da, Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*a*). *Acta Cryst.* E**61**, o4121–4122.
Silva, L. E. da, Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*b*). *Acta Cryst.* E**61**, o4252–4253.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.