Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[(6-Fluoro-1,3-benzothiazol-2-ylamino)-methylene]-2,2-dimethyl-1,3-dioxane-4,6-dione

Luiz Everson da Silva,[a,b] Antonio Carlos Joussef,[a] Sabine Foro[b] and Boris Schmidt[b]*

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

Correspondence e-mail: foro@tu-darmstadt.de

Key indicators

Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.094
Data-to-parameter ratio = 10.5

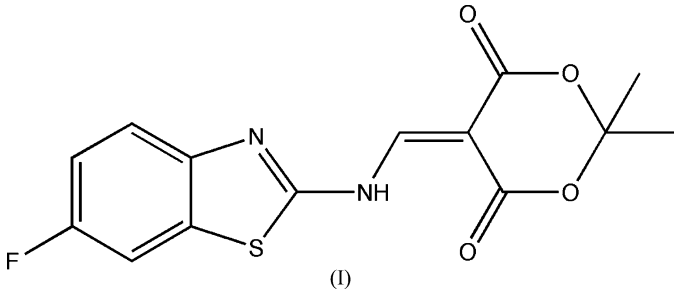
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 January 2006
Accepted 18 January 2006

In the title compound, $C_{20}H_{16}N_2O_4S$, the 1,3-dioxane-4,6-dione ring exhibits a half-chair conformation. The amino H atom forms an intramolecular contact to a carbonyl O atom, forming a six-membered ring, and an intermolecular contact to a carbonyl O atom. Additional intermolecular hydrogen bonds of the the types C—H···O and C—H···F are also observed.

Comment

We have focused on arylaminemethylene 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*). As an extension of this approach, we report here 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 C1—N2—C8—C9 torsion angle is −177.1 (2)° and the C1—N2 [1.400 (2) Å] and C8—C9 [1.371 (3) Å] distances indicate delocalization. The delocalization of the N-atom lone pair into the ring of Meldrum's acid may be favoured in the direction of one of the two carbonyl groups (Blake *et al.*, 2003).

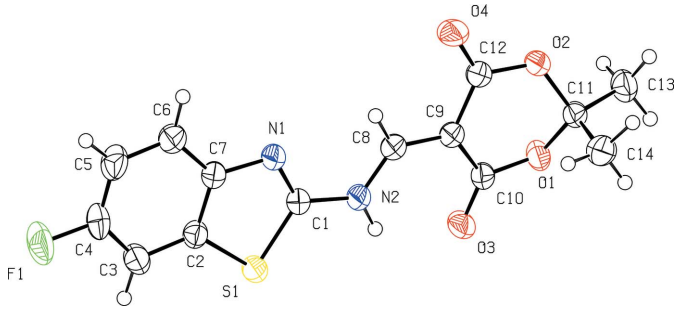


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level.

One intramolecular hydrogen bond of the type N—H···O was observed, together with three intermolecular hydrogen bonds of types N—H···O, C—H···O and C—H···F. These connect molecules to form a three-dimensional network, as represented in Fig. 2. Details of the hydrogen-bonding parameters are given in Table 1.

Experimental

The title compound was prepared according to the literature procedure of Cannon *et al.* (2001) and was recrystallized from ethyl acetate (m.p. 495–596 K; yield 78%).

Crystal data

$C_{14}H_{11}FN_2O_4S$
M_r = 322.31
Monoclinic, $P2_1/c$
a = 6.433 (1) Å
b = 19.532 (2) Å
c = 11.218 (2) Å
β = 102.52 (1)°
V = 1376.0 (4) Å^3
Z = 4
D_x = 1.556 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 4.6–18.6°
μ = 2.41 mm^{-1}
T = 299 (2) K
Rod, light brown
0.20 × 0.13 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2707 measured reflections
2454 independent reflections
1949 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 67.0°
$h = -7 \rightarrow 1$
$k = -23 \rightarrow 0$
$l = -13 \rightarrow 13$
3 standard reflections
frequency: 120 min
intensity decay: 2.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.094
S = 1.01
2454 reflections
233 parameters
Only H-atom coordinates refined
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.3778P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0043 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*N*···O3	0.89 (2)	2.19 (2)	2.801 (2)	125.5 (19)
N2—H2*N*···O3^{i}	0.89 (2)	2.23 (2)	3.045 (2)	151 (2)
C3—H3···O4^{ii}	0.94 (3)	2.58 (3)	3.394 (3)	145 (2)
C5—H5···F1^{iii}	0.90 (3)	2.46 (3)	3.356 (3)	174 (2)

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+4, -y, -z+1$.

All H atoms were located in a difference map, refined [0.90 (3)–1.03 (3) Å] and assigned isotropic displacement parameters of $1.2U_{eq}$(parent atom).

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); 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*.

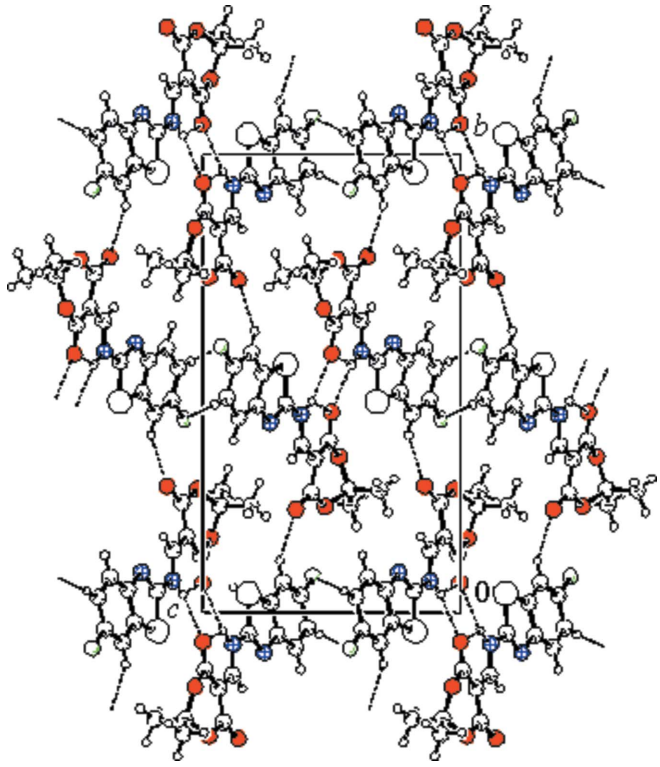


Figure 2
The molecular packing (arbitary spheres) of (I), viewed down the *a* axis, with hydrogen bonds shown as dashed lines.

The authors thank Professor Dr Hartmut Fuess, FG Strukturforschung, FB Material- und Geowissenschaften, Technische Universität Darmstadt, for diffractometer time.

References

Blake, A. J., McNab, H. & Withell, K. (2003). *Acta Cryst.* E**59**, o841–o842.
Cannon, D., Quesada, A., Quiroga, J., Insuasty, B., Abonia, R., Hernández, P., Cobo, J., Nogueras, M., Sánchez A. & Low, J. N. (2001). *Acta Cryst.* E**57**, o180–181.
Enraf–Nonius (1993). *CAD-4-PC*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.
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.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Silva, L. E. da, Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*a*). *Acta Cryst.* E**61**, o4121–o4122.
Silva, L. E. da, Joussef, A. C., Nunes, R. J., Andrighetti-Fröhner, C. R. & Bortoluzzi, A. J. (2005*b*). *Acta Cryst.* E**61**, o4252–o4253.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (1987). *REDU4*. Version 6.2c. Stoe & Cie GmbH, Darmstadt, Germany.