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5-(1,3-Benzothiazol-2-yliminomethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.123
Data-to-parameter ratio = 17.7

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

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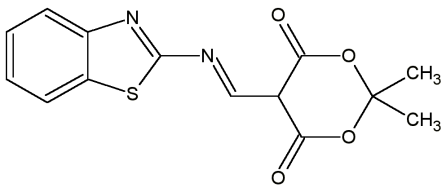
The title compound, $C_{14}H_{12}N_2O_4S$, has a supramolecular structure consisting of base-paired dimers with an $R_2^2(12)$ motif formed by a centrosymmetrically related pair of N—H···O hydrogen bonds.

Comment

We have focused on benzothiazole derivatives, which have shown diverse applications, as analgesics (Mehra *et al.*, 1980), anti-inflammatory agents, antineoplasics (Cheng *et al.*, 1993) and antimicrobial agents (Mehra *et al.*, 1980; El-Shaaer *et al.*, 1998), in our search for biologically active molecules. Benzothiazole derivatives have been prepared by a known reaction (Quiroga *et al.*, 1998). The title compound, (I), was prepared by condensation of Meldrum's acid, 2,2-dimethyl-1,3-dioxane-4,6-dione), 2-aminobenzothiazole and trimethyl orthoformate.



(I)

The supramolecular structure consists of base-paired dimers with an $R_2^2(12)$ motif (Bernstein *et al.*, 1995), formed by the N51—H51···O61^{i} hydrogen bond which is repeated across the centre at $(1,\frac{1}{2},1)$.

Geometric parameters are given in Table 1 and details of hydrogen bonds are given in Table 2. Fig. 1 shows a view of the molecule.

Examination of the structure with *PLATON* (Spek, 2000) showed that there were no solvent-accessible voids in the crystal lattice.

Experimental

A mixture of Meldrum's acid (6.94 mmol) and trimethyl orthoformate (34.7 mmol) was heated to reflux for 2.5 h, then 2-aminobenzothiazole (6.94 mmol) was added and the mixture was heated for a further 5 min. The title compound precipitated, was separated by filtration and was recrystallized from a dimethylformamide–ethanol mixture, affording crystals suitable for X-ray diffraction (m.p. 480–481 K, yield: 60%).

Crystal data

$C_{14}H_{12}N_2O_4S$
M_r = 304.32
Monoclinic, $P2_1/c$
a = 6.3459 (13) Å
b = 19.235 (4) Å
c = 11.144 (2) Å
β = 103.07 (3)°
V = 1325.0 (5) Å^3
Z = 4
D_x = 1.526 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6132 reflections
θ = 1.0–30.5°
μ = 0.26 mm^{-1}
T = 150 (1) K
Needle, orange
0.38 × 0.05 × 0.05 mm

Data collection

KappaCCD diffractometer
φ and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.908, T_{max} = 0.987
12 128 measured reflections
3392 independent reflections
2645 reflections with $I > 2\sigma(I)$
R_{int} = 0.081
θ_{max} = 30.5°
$h = -9 \rightarrow 8$
$k = -22 \rightarrow 27$
$l = -13 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
S = 1.08
3392 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0684P)^2 + 0.1432P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C51—N51	1.330 (2)	S51—C52	1.7391 (15)
N51—C52	1.402 (2)	C52—N53	1.290 (2)
S51—C59	1.7355 (16)	N53—C54	1.389 (2)
C51—N51—C52	120.14 (14)	C52—N53—C54	109.19 (13)
C59—S51—C52	87.69 (8)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N51—H51···O61^{i}	0.88	2.23	3.028 (2)	151
N51—H51···O61	0.88	2.22	2.807 (2)	124

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

H atoms were treated as riding with C—H distances in the range 0.95–0.98 Å and an N—H distance of 0.88 Å.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

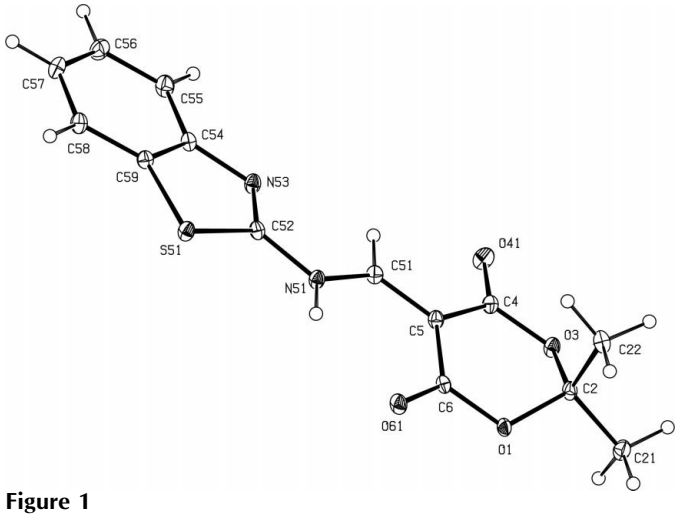


Figure 1
A view of the title molecule with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

X-ray data were collected at the EPSRC, X-ray Crystallographic Service, University of Southampton, using an Enraf—Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. We are grateful to the Ministerio de Educación *y* Cultura for the award of a grant to one of the authors (AQ).

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5-(1,3-Benzothiazol-2-yliminomethyl)-2,2-dimethyl-1,3-dioxane-4,6-dione. Erratum

Debbie Cannon,[a] Antonio Quesada,[a]† Jairo Quiroga,[b] Braulio Insuasty,[b] Rodrigo Abonia,[b] Pedro Hernández,[b] Justo Cobo,[c] Manuel Nogueras,[c] Adolfo Sánchez[c] and John Nicolson Low[d]*

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In the paper by Cannon *et al.* [*Acta Cryst.* (2001), E**57**, o180–o181], there is an error in the scheme. The correct scheme is given below.