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

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.054
wR factor = 0.116
Data-to-parameter ratio = 13.5

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

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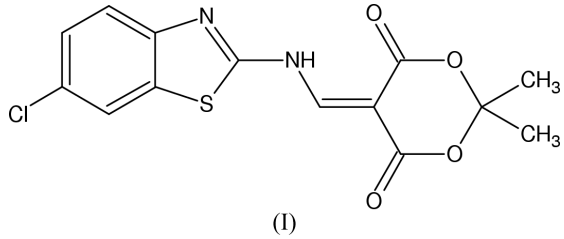
The molecules of the title compound, $C_{14}H_{11}ClN_2O_4S$, are linked together into ribbons formed by weak C—H···O hydrogen bonds.

Comment

The title compound, (I), is a benzothiazole derivative prepared in our search for biologically active molecules by a similar procedure to that reported previously for GURGIP (Cannon *et al.*, 2001; Cambridge Structural Database, Allen & Kennard, 1993).



(I)

GURGIP and compound (I) have similar molecular structures but have totally different supramolecular structures.

GURGIP 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.

Compound (I) has one intramolecular hydrogen bond, N21—H21···O14, which forms an $R(6)$ ring (Bernstein *et al.*, 1995). The molecules are linked together into ribbons formed by the weak hydrogen bonds, C5—H5···O16 and C7—H7···O14. These two bonds combine to form a $C^2_2(10)$ chain. These hydrogen bonds link the molecules head-to-tail as a result of the centres of symmetry at (1, 0, 0.5) for the former bond and (0.5, 0, 1) for the latter bond, forming in each case $R^2_2(22)$ rings and hence forming ribbons (Fig. 2). The details of the hydrogen bonding are given in Table 1.

Examination of the structure with *PLATON* (Spek, 2001) showed that there were no solvent-accessible voids in the crystal lattice.

Experimental

A solution of Meldrum's acid (3.45 mmol) and trimethyl orthoformate (17.3 mmol) was heated to reflux for 2.5 h, then 2-amino-6-chlorobenzothiazole (3.45 mmol) was added and the reaction mixture was heated for a further 30 min. The title compound precipitated, was separated by filtration, and recrystallized from ethanol, affording crystals suitable for X-ray diffraction (m.p.: 524 K, yield: 65%).

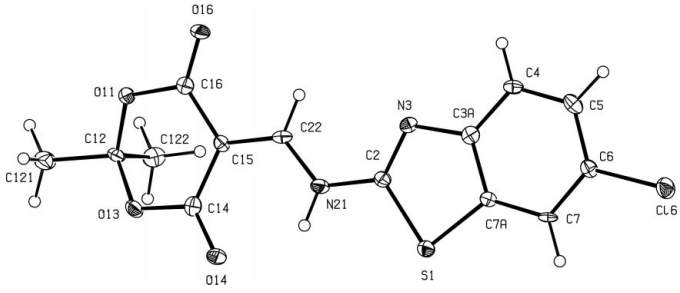


Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

Crystal data

$C_{14}H_{11}ClN_2O_4S$	D_x = 1.583 Mg m^{-3}
M_r = 338.76	Mo Kα radiation
Monoclinic, $P2_1/c$	Cell parameters from 2714 reflections
a = 5.3389 (4) Å	θ = 3.1–27.4°
b = 28.583 (2) Å	μ = 0.44 mm^{-1}
c = 9.8618 (8) Å	T = 120 (1) K
β = 109.178 (4)°	Plate, yellow
V = 1421.41 (19) Å^3	0.20 × 0.15 × 0.01 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	2714 independent reflections
φ scans, and ω scans with κ offsets	1430 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*DENZO-SMN*; Otwinowski & Minor, 1997)	R_{int} = 0.066
	θ_{max} = 27.4°
T_{min} = 0.918, T_{max} = 0.996	$h = -6 \rightarrow 6$
6289 measured reflections	$k = -37 \rightarrow 29$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.054	$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.116	$(\Delta/\sigma)_{max} < 0.001$
S = 0.94	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
2714 reflections	$\Delta\rho_{min}$ = −0.36 e Å^{-3}
201 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N21—H21···O14	0.88	2.01	2.671 (4)	131
C5—H5···O16^{i}	0.95	2.38	3.299 (5)	163
C7—H7···O14^{ii}	0.95	2.43	3.205 (5)	138

Symmetry codes: (i) $2-x, -y, 1-z$; (ii) $1-x, -y, 2-z$.

H atoms were treated as riding atoms with C—H = 0.95–0.98 Å and N—H = 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, 2001); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

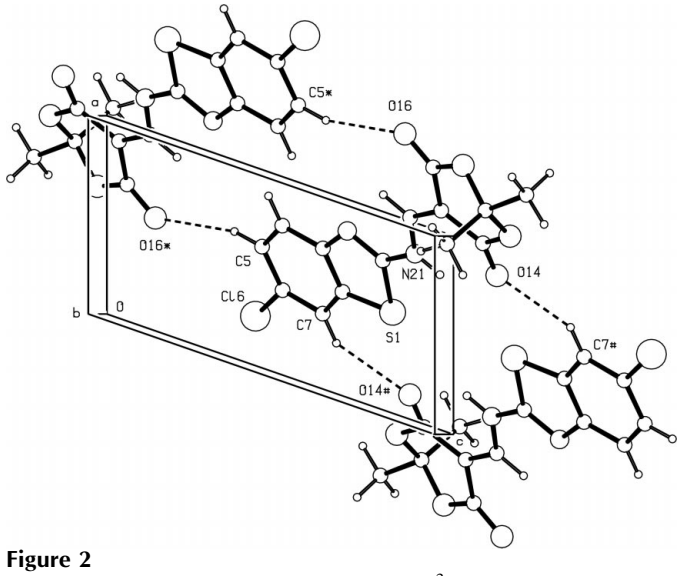


Figure 2
A view of the crystal structure showing the $C^2_2(10)$ chains and the two $R^2_2(22)$ rings. The molecule labelled with an asterisk (*) is at $(2-x, -y, 1-z)$ and that labelled with a hash (#) is at $(1-x, -y, 2-z)$.

X-ray data were collected at the EPSRC X-ray Crystallographic Service, University of Southampton, using a Nonius KappaCCD diffractometer. The authors thank the staff for all their help and advice. JNL thanks NCR Self Service Dundee for grants which have provided computing facilities for this work.

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