Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-[(4-Bromo-2-thienyl)methylenehydrazinocarbonyl]-1*H*-1,2,4-triazole

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.048
wR factor = 0.128
Data-to-parameter ratio = 13.3

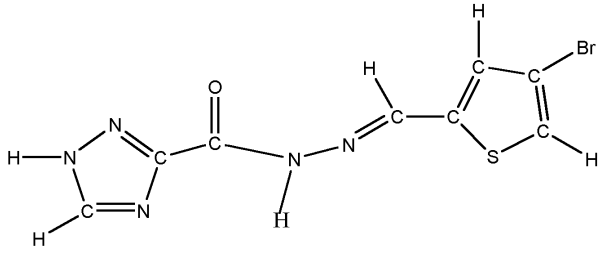
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_8H_6BrN_5OS$, was synthesized by the reaction of (1*H*-1,2,4-triazol-3-ylcarbonyl)hydrazine with 4-bromo-2-thiophenecarboxaldehyde in ethanol. The molecule is essentially planar and the crystal structure involves intermolecular N—H···O and N—H···N hydrogen bonds.

Received 8 October 2004
Accepted 20 October 2004
Online 20 November 2004

Comment

Azole derivatives, such as derivatives of pyrazole, imidazole, triazole (including benzotriazole), tetrazole, indole, *etc*., exhibit extensive biological activities. They have become a central focus in the study of agricultural chemicals, medicine, adjustment reagents for plant growth and so on (Ernest, 1982). A Schiff base is a good type of biologically active substructure and a study of a type of triazole Schiff base has been reported (Sauter *et al.*, 1991). The hydrazone–carboxyl grouping has also been shown to be bioactive (Zhi *et al.*, 2003). However, no structure of a triazole compound containing the hydrazone–carboxyl group has been reported. In a search for more effective antibacterial medicines, we have synthesized the title compound, (I).



(I)

The title molecule (Fig. 1) is essentially planar, with an r.m.s. deviation of 0.0076 Å. The bond lengths and angles are unexceptional (Allen *et al.*, 1987). Intermolecular N—H···O

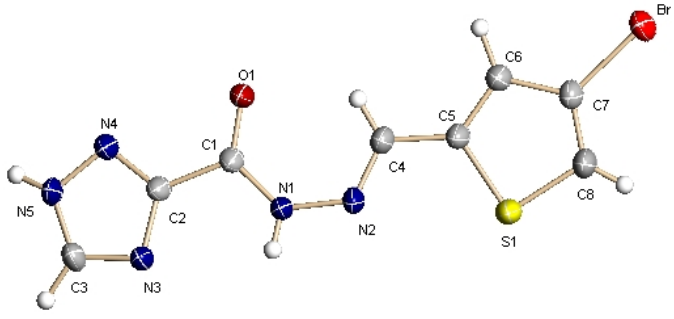


Figure 1
The structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

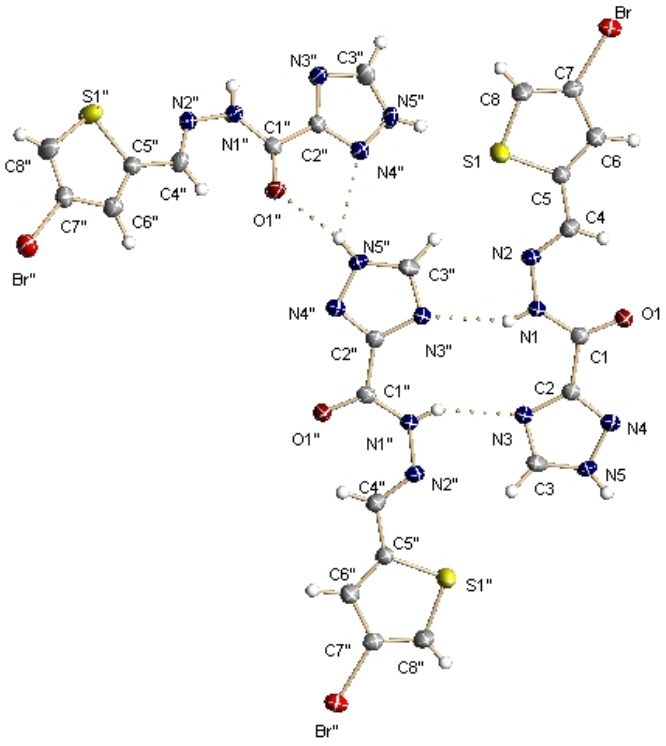


Figure 2
The packing of (I), showing the intermolecular hydrogen bonds as dotted lines.

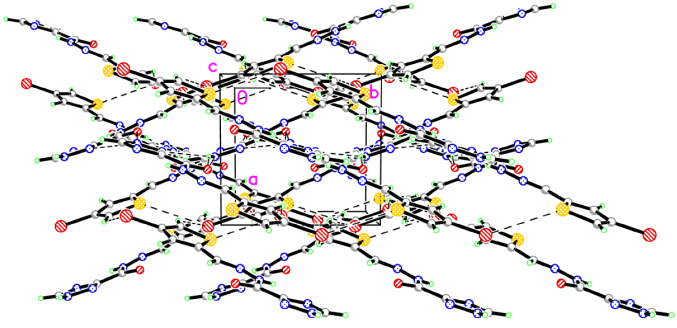


Figure 3
The packing of (I), viewed down the *c* axis, showing hydrogen-bonded chains. Hydrogen bonds are shown as dashed lines.

and N—H···N hydrogen bonds are observed, linking the ring NH group with the keto group and ring N atom of an adjacent molecule. Another N—H···N hydrogen bond links the chain NH group with a ring N atom, forming a ten-membered ring (Fig. 2 and Table 2). Symmetry-related molecules are linked along the *c*-axis direction *via* N—H···O and N—H···N hydrogen bonds (Fig. 3), forming a chain.

Experimental

(1*H*-1,2,4-triazol-3-ylcarbonyl)hydrazine (0.02 mol, 2.54 g) was dissolved in anhydrous ethanol (50 ml) at room temperature. 4-Bromo-2-thiophenecarboxaldehyde (0.02 mol, 3.82 g) was added and the mixture was refluxed for 2 h, yielding a precipitate which was collected by filtration and washed with ethanol. The product was recrystallized from ethanol and dried under reduced pressure to give the title compound. The latter compound, of which 2.5 mmol (0.75 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 30 d, yielding colorless block-shaped single crystals which were washed with distilled water.

Crystal data

$C_8H_6BrN_5OS$
M_r = 300.15
Monoclinic, $P2_1/c$
a = 7.3236 (10) Å
b = 7.6535 (10) Å
c = 18.761 (3) Å
β = 100.312 (2)°
V = 1034.6 (2) Å^3
Z = 4
D_x = 1.927 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2196 reflections
θ = 4.4–54.0°
μ = 4.16 mm^{-1}
T = 293 (2) K
Block, colorless
0.48 × 0.22 × 0.19 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.253, T_{max} = 0.459
5711 measured reflections
2242 independent reflections
1698 reflections with $I > 2\sigma(I)$
R_{int} = 0.142
θ_{max} = 27.0°
$h = -8 \rightarrow 9$
$k = -8 \rightarrow 9$
$l = -23 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.128
S = 0.97
2242 reflections
169 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.90 e Å^{-3}
$\Delta\rho_{min}$ = −1.00 e Å^{-3}

Table 1
Selected bond lengths (Å).

Br—C7	1.880 (4)	N3—C2	1.368 (5)
S1—C8	1.711 (5)	N4—C2	1.298 (5)
S1—C5	1.726 (4)	N5—C3	1.319 (6)
O1—C1	1.238 (5)	C4—C5	1.441 (5)
N1—C1	1.332 (5)	C5—C6	1.349 (6)
N1—N2	1.380 (4)	C6—C7	1.417 (5)
N2—C4	1.275 (5)	C7—C8	1.329 (6)
N3—C3	1.315 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···N4^{i}	0.815 (19)	2.47 (4)	3.038 (5)	128 (4)
N5—H5···O1^{i}	0.815 (19)	2.14 (3)	2.897 (4)	154 (5)
N1—H1···N3^{ii}	0.81 (4)	2.30 (4)	3.047 (5)	152 (4)

Symmetry codes: (i) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$; (ii) $1-x, 1-y, 2-z$.

All H atoms were located in a difference map and their parameters were freely refined. The N—H distances are 0.81 (4) and 0.815 (19) Å, and the C—H distances lie in the range 0.86 (4)–1.01 (5) Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Zhejiang Provincial Natural Science Foundation of China (No. M203115).

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