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## Key indicators

Single-crystal X-ray study

T = 273 K

Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$ 

R factor = 0.038

wR factor = 0.111

Data-to-parameter ratio = 16.0

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

## 1-[2-(1,3-Benzothiazol-2-ylimino)imidazolidin-1-yl]ethanone

With the exception of seven H atoms, the molecule of the title compound,  $\text{C}_{12}\text{H}_{12}\text{N}_4\text{OS}$ , is planar. Two intramolecular  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds are observed in the crystal structure.

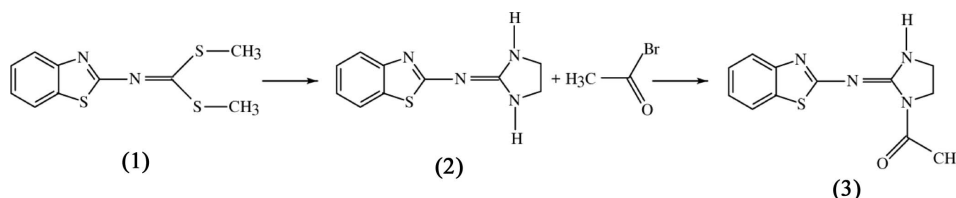
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## Comment

It is well known that the 2-arylamino-2-imidazoline pharmacophore is an important structural element in medicinal chemistry and that it shows a broad spectrum of pharmacological activities (Dardonville *et al.*, 2000; Matosiuk *et al.*, 2001). Several compounds from this class have been used as antihistaminic, antiparasitic and antiviral agents. A hypotensive action has been described for benzoyl derivatives of 2-arylamino-2-imidazoline, especially for 1-benzoyl-2-(2',6'-dichlorophenylamino)-2-imidazoline, in the depressant action on the central nervous system. Thus the sedative action is substantially less pronounced with this compound (Rudolf, 1975; Anastassiadou *et al.*, 2001).

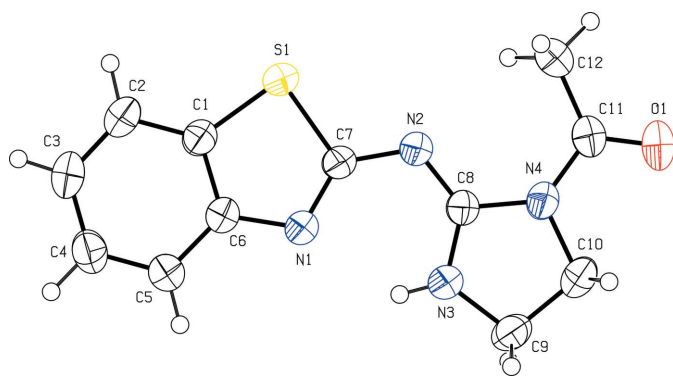
The atom-numbering scheme of the title compound, (3), is shown in Fig. 1. With the exception of seven H atoms, (3) is planar. The  $\text{S1}-\text{C1}$  [1.7338 (15) Å] and  $\text{S1}-\text{C7}$  [1.7654 (13) Å] bond distances are in agreement with those in similar compounds (Aydın *et al.*, 2002; Akkurt *et al.*, 2005). The  $\text{N2}=\text{C8}$  bond distance [1.3053 (16) Å] and the  $\text{N2}-\text{C7}$  bond length [1.3589 (17) Å] are normal for  $\text{N}=\text{Csp}^2$  and  $\text{N}-\text{Csp}^2$  bond lengths in related compounds (Cambridge Structural Database; Version 5.27; Allen, 2002).



The structure of (3) is stabilized by weak intramolecular  $\text{N}-\text{H}\cdots\text{N}$  and  $\text{C}-\text{H}\cdots\text{N}$  hydrogen bonds (Table 1).

## Experimental

Many methods have been reported for the synthesis of 2-arylamino-2-imidazoline compounds (Genç & Servi, 2005). In the present study, the starting material was dimethyl *N*-(1,3-benzothiazol-2-yl)dithioimidocarbonate, (1). To a solution of 2-amino-1,3-benzothiazole (0.1 mol) in dimethylformamide (75 ml), aqueous 20 M sodium hydroxide (5.5 ml, 0.11 mol) was added with stirring at room temperature. After 10 min, carbon disulfide (3.3 ml, 0.055 mol) was



**Figure 1**  
An ORTEP-3 (Farrugia, 1997) plot of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

added and stirring was continued for 30 min. Aqueous 20 M sodium hydroxide (3 ml, 0.06 mol) and carbon disulfide (1.8 ml, 0.0275 mol) were then added. This operation was finally repeated 10 min later. After 30 min, the reaction mixture was placed in an ice bath, methyl iodide (12.5 ml, 0.2 mol) was added dropwise and stirring was continued for 2 h. The mixture was then poured into ice-cooled water, and the resulting precipitate was filtered off, washed with water and dried; compound (1) was recrystallized from ethanol as a yellow powder (yield 69%; m.p. 374–375 K). Analysis (calculated/found) for  $C_{10}H_{10}N_2S_3$ : C 47.21/48.39, H 3.96/4.06, N 11.01/11.34, S 37.81/38.77%. IR (KBr) ( $t$ ,  $cm^{-1}$ ): 2995 (aliphatic C–H stretching), 1592 (C=C stretching), 1510 (C=N stretching), 833 (S–CH<sub>3</sub> stretching). <sup>1</sup>H NMR ( $CHCl_3$ -*d*, 90 MHz, p.p.m.):  $\delta$  2.59 (*s*, 6H, SCH<sub>3</sub>), 6.90–7.30 (*m*, 2H, Ar–H), 7.50–7.86 (*m*, 2H, Ar–H).

Synthesis of the 2-arylmino-2-imidazoline (2) was carried out both by conventional heating and by microwave irradiation, according to literature procedures (Servi *et al.*, 2005; Servi, 2002). Its *N*-acetyl derivative, (3), was synthesized from the reaction of (2) and acetyl bromide. *N*-(1,3-Benzothiazol-2-yl)-*N*-(4,5-dihydro-1H-imidazol-2-yl)amine, (2) (1 mmol, 0.218 g), and tetrahydrofuran (40 ml) were placed in a 250 ml one-necked flask with a reflux condenser. Acetyl bromide (1.1 mmol) was added and the mixture was refluxed for 16 h. The reaction mixture was cooled and then neutralized with NH<sub>3</sub> solution. The resulting precipitate was filtered off and washed with water. The residue was filtered off and recrystallized from acetone. The product, (3), was obtained in 79% yield (m.p. 575 K). IR ( $cm^{-1}$ ): 3194 (N–H stretching), 3060 (aromatic C–H stretching), 2981–2927 (C–H stretching), 1678 (C=O stretching), 1630 (C=C stretching).

#### Crystal data

$C_{12}H_{12}N_4OS$   
 $M_r = 260.33$   
Monoclinic,  $P2_1/n$   
 $a = 7.3124$  (1) Å  
 $b = 7.9312$  (1) Å  
 $c = 21.0085$  (3) Å  
 $\beta = 93.271$  (1)°  
 $V = 1216.43$  (3) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.421$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation  
 $\mu = 0.26$  mm<sup>-1</sup>  
 $T = 273$  (2) K  
Block, colorless  
 $0.57 \times 0.30 \times 0.14$  mm

#### Data collection

Siemens SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: none  
17496 measured reflections

3197 independent reflections  
2639 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.025$   
 $\theta_{max} = 29.0^\circ$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.111$   
 $S = 1.05$   
3197 reflections  
200 parameters

$w = 1/[^2(F_o^2) + (0.059P)^2 + 0.2011P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} = 0.002$   
 $\Delta\rho_{max} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.21$  e Å<sup>-3</sup>

H atoms treated by a mixture of independent and constrained refinement

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H13N \cdots N1$	0.82 (2)	2.06 (2)	2.6731 (16)	131 (2)
$C12-H12C \cdots N2$	0.96	2.34	2.872 (2)	115

Methyl H atoms were positioned geometrically and treated as riding, with C–H = 0.96 Å and  $U_{iso}(H) = 1.5U_{eq}(C)$ . Other H atoms were located in a difference Fourier map and refined freely [C–H = 0.91 (2)–1.00 (2) Å and N–H = 0.82 (2) Å].

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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