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(1,3-Benzothiazol-2-yl)[1-(2-chlorobenzyl)-imidazolidin-2-ylidene]amine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.135
Data-to-parameter ratio = 33.7

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

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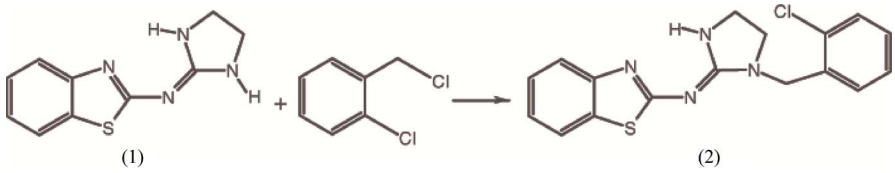
In the crystal structure of the title compound, $C_{17}H_{15}ClN_4S$, the benzothiazole, imidazolidine and benzene rings are twisted relative to each other.

Comment

The imidazole ring system is an important nucleus for drug discovery and represents the core structure of a number of biologically important molecules. 2-Arylamino-2-imidazolines are effective pharmacophores in medicinal chemistry (Dardonville *et al.*, 2000). The most important of such compounds are clonidine, moxonidine and phentolamine compounds. These compounds show α_1 and α_2 adrenoceptor activities. Phentolamine, which contains an imidazoline ring, is a known α_1-adrenergic antagonist (Dardonville *et al.*, 2000; Matosiuk *et al.*, 2001).



(1) (2)

In the crystal structure of the title compound, (2), the benzothiazole ring system is almost planar, the greatest deviation from the least-squares plane being 0.015 (1) Å for C7. The imidazolidine ring is also almost planar [deviations from planarity: N3 0.064 (1), N4 0.055 (1), C8 0.006 (1), C9 0.088 (2) and C10 − 0.084 (1) Å]. The dihedral angle between the benzothiazole and benzene rings is 70.00 (6)° and that between the benzothiazole and imidazolidine rings is 7.88 (6)°. The dihedral angle between the benzene and imidazolidine rings is 77.41 (7)°. The chlorobenzene unit is nearly planar, with Cl1 displaced 0.030 (1) Å from the mean plane of the benzene ring.

The bond lengths and angles are comparable with those found in related structures, for example 2-hydroxybenzothiazole (Flakus *et al.*, 2002) and methyl 3-[5-chloro-2-oxo-2*H*-1,3-benzothiazol-3-yl]propanoate (Aydin *et al.*, 2002).

Experimental

The synthesis of *N*-(1,3-benzothiazol-2-yl)-N-(4,5-dihydro-1*H*-imidazol-2-yl)amine, (1), was performed according to literature procedures (Genç & Servi, 2005; Servi *et al.*, 2005; Servi, 2002). For the synthesis of the title compound, (2), *N*-(1,3-benzothiazol-2-yl)-*N*-(4,5-dihydro-1*H*-imidazol-2-yl)amine (0.69 g, 3.17 mmol) and finely powdered NaOH (0.5 g, 12.5 mmol) were suspended in DMSO (7 ml) and afterwards 2-chlorobenzyl chloride (0.35 ml, d = 1.274 Mg m^{-3},

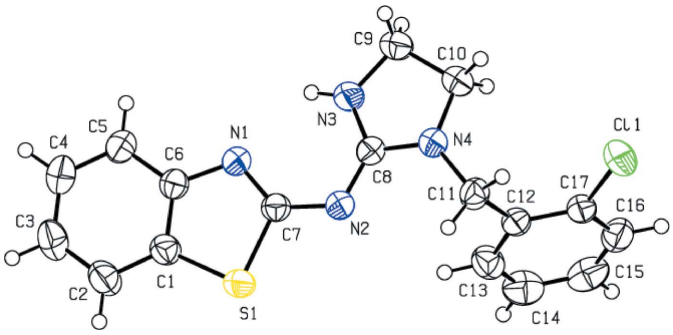


Figure 1
The molecular structure of the title compound, (2), showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

3.45 mmol) was added dropwise. The resulting solution was stirred at 308–313 K for 1 h. Afterwards water was added to the reaction mixture and the solid that precipitated was filtered off and dissolved in ethanol. Crystals of (2) formed by slow evaporation of the solvent (yield 76%; m.p. 421 K). IR (cm^{-1}): 3312 (N—H stretching), 3085 (aromatic C—H stretching), 2951–2856 (C—H stretching), 1679 (C═C stretching), 1589 (C═N stretching), 525 (C—Cl).

Crystal data

$C_{17}H_{15}ClN_4S$
M_r = 342.85
Monoclinic, $C2/c$
a = 20.0595 (3) Å
b = 10.6269 (2) Å
c = 15.0647 (2) Å
β = 97.572 (1)°
V = 3183.34 (9) Å^3
Z = 8
D_x = 1.431 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 293 K
Block, colourless
0.76 × 0.45 × 0.44 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
34371 measured reflections
7012 independent reflections
4725 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 35.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.135
S = 1.03
7012 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 1.0812P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C17	1.7436 (14)	N2—C8	1.3205 (15)
S1—C1	1.7367 (13)	N3—C8	1.3471 (15)
S1—C7	1.7644 (12)	N3—C9	1.4508 (19)
N1—C6	1.3854 (16)	N4—C8	1.3492 (16)
N1—C7	1.3150 (15)	N4—C10	1.4543 (17)
N2—C7	1.3558 (15)	N4—C11	1.4495 (16)
C1—S1—C7	89.50 (6)	S1—C7—N1	114.78 (9)
C6—N1—C7	110.72 (10)	S1—C7—N2	115.65 (8)
C7—N2—C8	118.18 (10)	N1—C7—N2	129.56 (11)
C8—N3—C9	111.39 (10)	N2—C8—N3	129.05 (11)
C8—N4—C10	111.66 (10)	N3—C8—N4	109.13 (10)
C8—N4—C11	124.43 (11)	N2—C8—N4	121.82 (10)
C10—N4—C11	122.68 (10)	N3—C9—C10	103.00 (11)
S1—C1—C6	109.01 (9)	N4—C10—C9	102.44 (10)
S1—C1—C2	129.55 (10)	N4—C11—C12	112.19 (10)
N1—C6—C1	115.97 (11)	Cl1—C17—C12	119.90 (10)
N1—C6—C5	124.70 (11)	Cl1—C17—C16	118.25 (11)

All H atoms were positioned with idealized geometry (C–H = 0.93–0.97 Å) and were refined using a riding-model, with U_{iso}(H) = 1.2U_{eq}(parent atom).

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* (Farrugia, 1997); software used to prepare material for publication: Win *GX* (Farrugia, 1999).

References

Aydin, A., Arici, C., Akkurt, M., Akkoç, Y. & Sahin, M. F. (2002). *Anal. Sci.* **18**, 1401–1402.
Dardonville, C., Goya, P., Rozas, I., Alsasua, A., Martin, M. I. & Borrego, M. (2000). *Bioorg. Med. Chem.* **8**, 1567–1577.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flakus, H. T., Miros, A. & Jones, P. G. (2002). *J. Mol. Struct.* **604**, 29–44.
Genç, M. & Servi, S. (2005). *Heteroatom Chem.* **16**, 142–147.
Matosiuk, D., Fidecka, S., Michaluk, L. A., Dybala, I. & Koziol, A. E. (2001). *Eur. J. Med. Chem.* **36**, 783–797.
Servi, S. (2002). *S. Afr. J. Chem.* **55**, 119–123.
Servi, S., Genç, M., Gür, S. & Koca, M. (2005). *Eur. J. Med. Chem.* **40**, 687–693.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.