Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-[4-Chloro-2-(4-chlorobenzylsulfanyl)-thiazol-5-ylmethylene]-3-(2,4-dichlorobenzyl)thiazolidine-2,4-dione

Özen Özgen,[a]* Oya Bozdağ-Dündar,[b] Rahmiye Ertan[b] and Engin Kendi[a]

[a]Department of Physics Engineering, Hacettepe University, Beytepe 06800, Ankara, Turkey, and [b]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Ankara University, Tandõgan, Ankara, Turkey

Correspondence e-mail:
ozgen_ozen@yahoo.com

Received 27 March 2007
Accepted 28 March 2007

There are two molecules in the asymmetric unit of the title compound, $C_{21}H_{12}Cl_4N_2O_2S_3$, which differ in the orientation of the chlorobenzene ring.

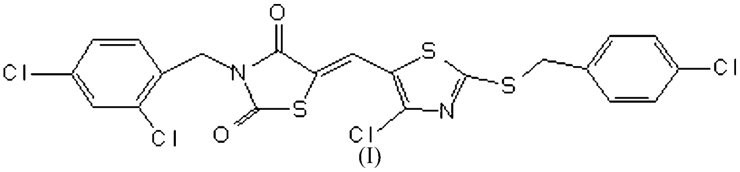
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
R factor = 0.076
wR factor = 0.221
Data-to-parameter ratio = 15.2

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

Comment

The title compound, (I), was synthesized to test its anti-hyperglycemic and AR (aldose reductase) inhibitory activity (Costantino *et al.*, 2000).



(I)

There are two molecules in the asymmetric unit of (I) (Fig. 1), denoted 1 (containing C1) and 2 (containing C1′): each contains a thiazolidine (ring *A*), thiazole (ring *B*), and two substituted [C1–C6 (ring *C*) and C16–C21 (ring *D*)] benzene rings.

Ring *A* is planar in molecule 2, but in molecule 1 atom S1 deviates by 0.058 (3) Å from the C9/C8/N1/C10 plane. Rings *A* and *B* are twisted slightly with respect to each other, making dihedral angles of 5.5 (3)° and 6.3 (2)° in 1 and 2, respectively. The C8—C9—C11—C12 torsion angles [−177.8 (9)° in 1 and −179.6 (8)° in 2] indicate that both molecules adopt a *cis* configuration.

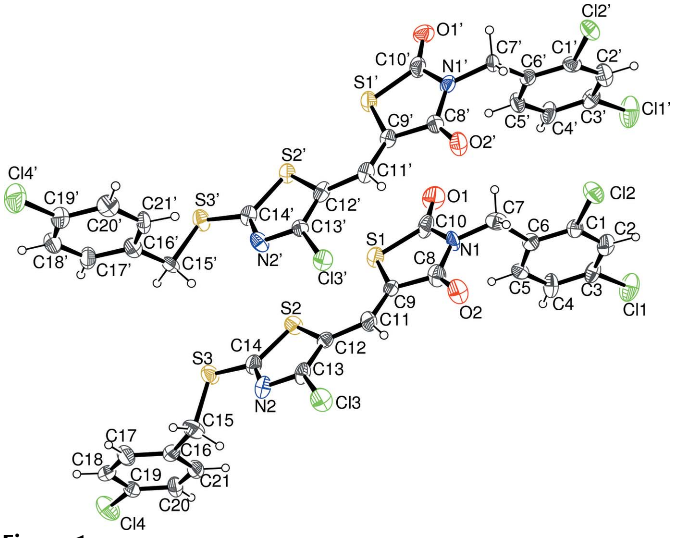


Figure 1
The asymmetric unit of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms).

doi:10.1107/S1600536807015139

The bond lengths and angles in ring *A* are very similar to the values reported for 3-(4-chlorobenzyl)-5-(4-oxo-4*H*-chromen-3-yl-methylene)-1,3-thiazolidine-2,4-dione (Özgen *et al.*, 2005) and 3-methyl-5-(4-oxo-4*H*-chromen-3-yl-methylene)-1,3-thiazolidine-2,4-dione (Aslantaş *et al.*, 2006). In both molecules, the C14—S3 bond length [1.700 (9) and 1.728 (8) Å in 1 and 2, respectively] is shorter than the standard Csp^2—S single bond (1.76 Å), whereas the C15—S3 bond lengths [1.813 (8) in 1 and 1.813 (9) Å in 2] are much longer than the reference value. The orientation of ring *D* attached to *B* is quite different in the two molecules, as reflected in the C14—S3—C15—C16 torsion angles of −153.9 (7)° in 1 and 96.8 (7)° in 2.

In the packing of (I), the molecules aggregate as layers parallel to (001), as shown in Fig. 2.

Experimental

A mixture of 4-chloro-2-(4-chlorobenzylsulfanyl)thiazole-5-carbaldehyde (0.5 mmol) and 3-(2,4-dichlorobenzyl)thiazolidine-2,4-dione (Lo & Shropshire, 1957) (0.5 mmol) was heated at 373 K in the presence of 0.5 ml glacial acetic acid and sodium acetate (0.5 mmol). The reaction mixture was extracted with $CHCl_3$ (3 × 25 ml) and the organic layer was washed with water, dried over anhydrous Na_2SO_4 and evaporated to dryness. The residue was purified by column chromatography using silica gel 60 (230–400 mesh ASTM) as the adsorbent and hexane–dicholoromethane (1:1 *v*/*v*) as the eluent. Yellow prisms of (I) were recrystallized from dimethylformamide/isopropanol (1:9 *v*/*v*) (yield 0.432 g, 61.31%; m.p. 427–428 K). Analysis calculated for $C_{21}H_{12}Cl_4N_2O_2S_3$: C 44.85, H 2.15, N 4.98, S 17.11%; found C 44.41, H 2.39, N 5.03, S 17.07%.

Crystal data

$C_{21}H_{12}Cl_4N_2O_2S_3$
$M_r = 562.31$
Triclinic, $P\overline{1}$
$a = 8.0420$ (7) Å
$b = 14.359$ (2) Å
$c = 21.2641$ (15) Å
$\alpha = 100.386$ (8)°
$\beta = 99.594$ (7)°
$\gamma = 104.237$ (10)°
$V = 2283.0$ (4) Å^3
$Z = 4$
Cu *Kα* radiation
$\mu = 7.49$ mm^{-1}
$T = 293$ (2) K
0.27 × 0.24 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.237$, $T_{max} = 0.346$ (expected range = 0.178–0.260)
9143 measured reflections
8769 independent reflections
3186 reflections with $I > 2\sigma(I)$
$R_{int} = 0.111$
3 standard reflections frequency: 120 min intensity decay: −14%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.221$
$S = 1.03$
8769 reflections
577 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

H atoms were placed in idealized geometries (C—H = 0.93–0.97 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

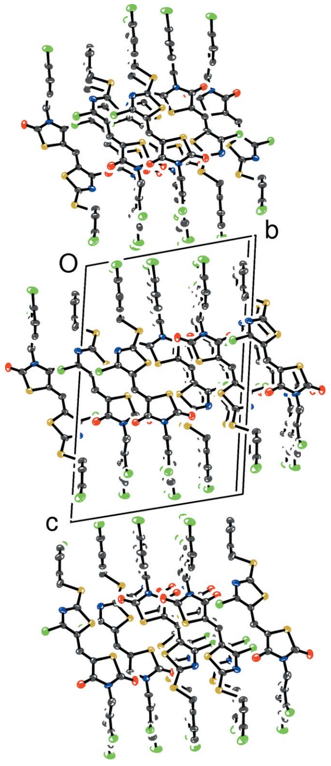


Figure 2
The packing for (I), viewed down the *a* axis, with H atoms omitted for clarity.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *WinGX* (Farrugia, 1999).

The authors thank the Research Organization of Ankara University, Turkey (No. 2005–0803048) for support of this work.

References

Aslantaş, M., Ceylan-Ünlüsoy, M., Ertan, R., Kendi, E. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o1471–o1473.
Costantino, L., Rastelli, G., Gamberoni, M. C. & Barlocco, D. (2000). *Exp. Opin. Ther. Patents*, **10**, 1245–1262.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). J. Appl. Cryst. 32, 837-838.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Lo, C.-P. & Shropshire, E. (1957). *J. Org. Chem.* **22**, 999–1001.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Özgen, Ö., Ceylan-Ünlüsoy, M., Bozdağ-Dündar, O., Ertan, R. & Kendi, E. (2005). *Acta Cryst.* E**61**, o870–o872.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.