Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(2-Bromo-4,5-dimethoxybenzyl)thiazolidine-2,4-dione

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.032
wR factor = 0.078
Data-to-parameter ratio = 12.2

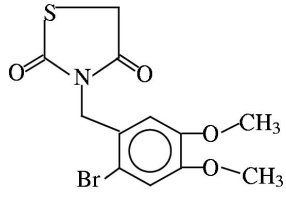
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 27 January 2005
Accepted 7 February 2005
Online 12 February 2005

The title compound, $C_{12}H_{12}BrNO_4S$, belongs to the class of substituted thiazolidine-2,4-diones. Compounds of this type are important starting materials in pharmaceutical chemistry. The thiazolidine-2,4-dione ring is disordered about a twofold rotation axis. Geometric parameters, other than those of the disordered groups, are in the normal ranges.

Comment

Thiazolidine-2,4-dione is used as a starting material for the synthesis of drugs with antihyperglycemic activity (Zask *et al.*, 1990). In heterocyclic chemistry, thiazolidine-2,4-diones are particularly important as a therapeutic agents and have been thoroughly investigated as PPAR-γ agonists that led to the development of several insulin-sensitizing drugs for the treatment of type-2 diabetes (Blanchet & Zhu, 2004). Diverse biological activities have been found to be associated with thiazolidine derivatives (Singh *et al.*, 1981). The present communication reports the synthesis and crystal structure of a new thiazolidine-2,4-dione derivative, *viz*. 3-(2-bromo-4,5-dimethoxybenzyl)thiazolidine-2,4-dione, (I).



(I)

A perspective view of (I) is shown in Fig. 1. Since the thiazolidine-2,4-dione ring is disordered, it is not appropriate to discuss its geometric parameters. The dihedral angles between the two rings are 70.86 (12) and 68.3 (2)° for the major and minor occupied sites, respectively. The Br atom is displaced away from the neighbouring carbonyl group [Br12···O2 = 4.126 (4) Å and Br12···O2′ = 3.549 (14) Å], as can be seen by comparing the bond angles involving the C—Br bond (Table 1). Whereas one of the methoxy groups is almost coplanar with the aromatic ring, the other one is slightly displaced from the ring plane.

Experimental

An equimolar mixture of thiazolidine-2,4-dione (1.17 g, 10 mmol), 1-bromo-2-bromomethyl-4,5-dimethoxybenzene (3.1 g, 10 mmol) and anhydrous K_2CO_3 (1.38 g, 10 mmol) was stirred at room temperature in dimethylformamide (10 ml) for 6 h. The product formed was crystallized from ethanol. The title compound melts at 420 K. IR

(KBr, ν cm^{-1}): 3396 (*w*), 2939 (*s*), 2837 (*w*), 1740 (*s*), 1668 (*s*), 1508 (*s*), 1380 (*m*), 1213 (*m*); ^{1}H NMR ($CDCl_3$, p.p.m.): 3.8 (*s*, 6H, OCH_3–), 3.92 (*s*, 2H, CH_2–), 4.89 (*s*, 2H, CH_2–), 6.49 (*s*, 1H, ArH–), 6.92 (*s*, 1H, ArH–); ^{13}C NMR ($CDCl_3$, p.p.m.): 39.2 (*t*, C2, CH_2–), 42.3 (*t*, C4, CH_2–), 59.1 (*q*, C42, C52, OCH_3–), 116 (*s*, C11, C—C–), 117.2 (*d*, C13, ArCH–), 119.4 (*d*, C16, ArCH–), 141.1 (*s*, C12, C—Br–), 145.1 (*s*, C15, C—O–), 146.8 (*s*, C14, C—O–), 167.9 (*s*, C3, C═C–), 168.2 (*s*, C1, C═O). Analysis calculated for $C_{12}H_{12}BrNO_4S$: C 41.63, H 3.49, N 4.05%; found: C 41.67, H 3.48, N 4.06%.

Crystal data

$C_{12}H_{12}BrNO_4S$
M_r = 346.20
Monoclinic, $P2_1/c$
a = 8.6677 (10) Å
b = 18.5082 (18) Å
c = 8.9746 (10) Å
β = 111.950 (9)°
V = 1335.4 (3) Å^3
Z = 4
D_x = 1.722 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7895 reflections
θ = 4.2–25.6°
μ = 3.24 mm^{-1}
T = 173 (2) K
Block, colourless
0.36 × 0.32 × 0.29 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.334, T_{max} = 0.390
6240 measured reflections
2486 independent reflections
2242 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.7°
$h = -9 \rightarrow 10$
$k = -19 \rightarrow 22$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.078
S = 1.16
2486 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.5152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.79 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0107 (12)

Table 1
Selected geometric parameters (Å, °).

Br12—C12	1.911 (2)		
C11—C12—Br12	121.66 (19)	C13—C12—Br12	116.37 (18)
C42—O41—C14—C13	0.6 (3)	C52—O51—C15—C16	−7.1 (4)

The thiazolidine-2,4-dione ring is disordered about an approximate twofold rotation axis running through atom N1 and the CH_2—S bond. The ratio of site-occupation factors of the disordered atoms refined to 0.740 (5):0.260 (7). The atoms of the minor component were refined isotropically. Corresponding bond lengths and angles in the two disordered groups were restrained to be equal. H atoms (excluding those of the methylene group in the thiazolidine-2,4-dione ring) were located in a difference map. All H atoms were geometrically positioned and refined with fixed individual displacement parameters [set at $1.2U_{eq}$ of the parent atom ($1.5U_{eq}$ for methyl groups)] using a riding model, with C—H distances ranging from 0.95 to 0.99 Å. In addition, the torsion angles about the methyl groups were refined.

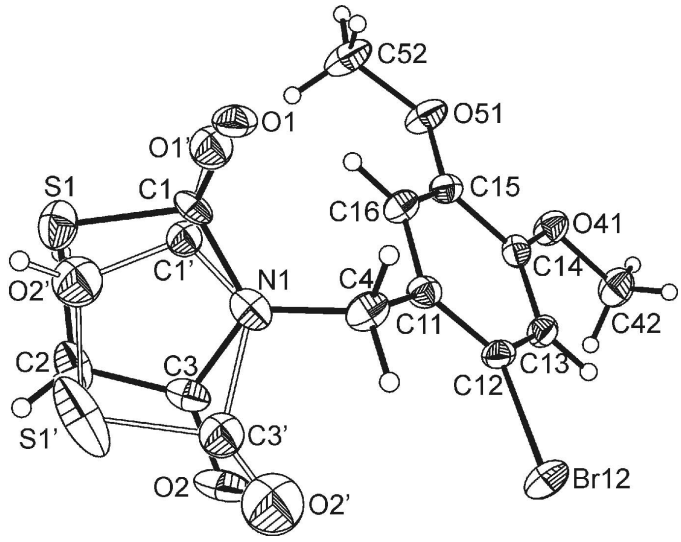


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

References

Blanchet, J. & Zhu, J. (2004). *Tetrahedron Lett.* **45**, 4449–4452.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Singh, S. P., Parmar, S. S., Raman, K. & Stenberg, V. I. (1981). *Chem. Rev.* **81**, 175–203.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
Zask, A., Jirkovsky, I., Nowicki, J. W. & McCaleb, M. L. (1990). *J. Med. Chem.* **33**, 1418–1423.