Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-5-(2-Chlorobenzylidene)thiazolidine-2,4-dione

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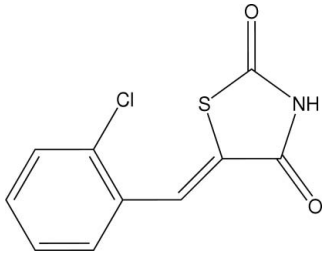
Received 7 October 2007; accepted 10 October 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.068; wR factor = 0.173; data-to-parameter ratio = 14.3.

In the title compound, $C_{10}H_6ClNO_2S$, the benzene and thiazolidine rings are oriented at a dihedral angle of 14.22 (5)°. Intramolecular C—H···O, C—H···Cl and C—H···S hydrogen bonds result in the formation of two nearly planar five-membered rings and one nonplanar six-membered ring, the five-membered rings being also nearly coplanar with the adjacent rings. In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules.

Related literature

For general background, see: Barreca *et al.* (2002); Botti *et al.* (1996). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_6ClNO_2S$
M_r = 239.67
Monoclinic, $P2_1/a$
a = 7.2680 (8) Å
b = 7.5916 (11) Å
c = 18.038 (2) Å
β = 91.25 (3)°
V = 995.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.57 mm^{-1}
T = 294 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.848, T_{max} = 0.945
2109 measured reflections
1942 independent reflections
1102 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.173
S = 1.01
1942 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O2^{i}	0.86	2.00	2.833 (6)	164
C4—H4A···S	0.93	2.63	3.326 (5)	132
C7—H7A···Cl	0.93	2.54	3.010 (6)	112
C7—H7A···O1	0.93	2.50	2.866 (7)	104

Symmetry code: (i) $-x-\frac{1}{2}, y-\frac{1}{2}, -z+1$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; 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: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2342).

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 doi:10.1107/S1600536807049756

supplementary materials

Acta Cryst. (2007). E**63**, o4325 [**doi:10.1107/S1600536807049756**]

(*Z*)-5-(2-Chlorobenzylidene)thiazolidine-2,4-dione

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Comment

Thiazolidines are an important class of heteroaromatic compounds and have widespread applications from pharmaceuticals (Barreca *et al.*, 2002) to materials (Botti *et al.*, 1996). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The intramolecular C—H···O, C—H···Cl and C—H···S hydrogen bonds (Table 1) cause to the formation of the nearly planar five-membered rings; C (Cl/C5—C7/H7A) and D (O1/C7—C9/H7A), in which they are also nearly co-planar with the adjacent rings A (C1—C6) and B (S/N/C8—C10), and non-planar six-membered ring E (S/C4/C5/C7/C8/H4A). The dihedral angles between them are A/C = 5.13 (3)° and B/D = 1.99 (3)°. The planar rings A (C1—C6) and B (S/N/C8—C10) are oriented at a dihedral angle of 14.22 (5)°.

In the crystal structure, intermolecular N—H···O hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they seem to be effective in the stabilization of the structure.

Experimental

Thiazolidine-2,4-dione (10 mmol) and 2-chlorobenzaldehyde (10 mmol) were dissolved in ethanol (10 ml) in a 50 ml round-bottomed flask and piperidine (5 d) was added. The flask was heated in a modified domestic microwave oven at 300 W for 5 min. After being cooled down, the mixture was poured into water and the crude compound (I) was filtered. Crystals of (I) suitable for X-ray analysis were obstained by slow evaporation of an ethanol solution.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C,*N*),

Figures

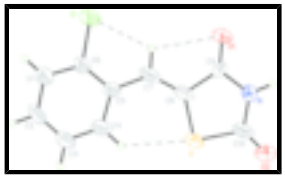

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

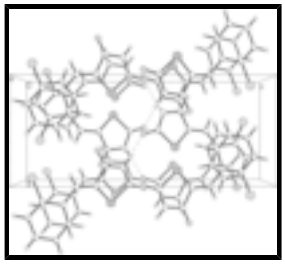

Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

(*Z*)-5-(2-Chlorobenzylidene)thiazolidine-2,4-dione

Crystal data

$C_{10}H_6ClNO_2S$	F_{000} = 488
M_r = 239.67	D_x = 1.600 Mg m^{-3}
Monoclinic, $P2_1/a$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yab	Cell parameters from 25 reflections
a = 7.2680 (8) Å	θ = 10–13º
b = 7.5916 (11) Å	μ = 0.57 mm^{-1}
c = 18.038 (2) Å	T = 294 (2) K
β = 91.25 (3)º	Block, colorless
V = 995.1 (2) Å^3	0.30 × 0.20 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.035
Radiation source: fine-focus sealed tube	θ_{max} = 26.0º
Monochromator: graphite	θ_{min} = 2.9º
T = 294(2) K	h = −8→8
ω/2θ scans	k = 0→9
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→22
T_{min} = 0.848, T_{max} = 0.945	3 standard reflections
2109 measured reflections	every 120 min
1942 independent reflections	intensity decay: none
1102 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.068	H-atom parameters constrained
$wR(F^2)$ = 0.173	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 2P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.01	$(\Delta/\sigma)_{max} < 0.001$
1942 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
136 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl	0.1217 (3)	−0.08790 (19)	0.09226 (9)	0.0707 (6)
S	−0.1266 (2)	0.15255 (17)	0.37609 (8)	0.0503 (4)
O1	0.0030 (7)	−0.3234 (5)	0.3296 (2)	0.0682 (13)
O2	−0.2614 (7)	0.0265 (5)	0.5003 (3)	0.0711 (13)
N	−0.1285 (6)	−0.1687 (6)	0.4216 (2)	0.0450 (11)
H0A	−0.1416	−0.2594	0.4495	0.054*
C1	0.1318 (9)	0.2601 (7)	0.0772 (3)	0.0544 (15)
H1A	0.1663	0.2377	0.0288	0.065*
C2	0.1109 (9)	0.4315 (7)	0.1044 (3)	0.0598 (17)
H2A	0.1286	0.5263	0.0727	0.072*
C3	0.0653 (8)	0.4634 (7)	0.1764 (3)	0.0516 (15)
H3A	0.0512	0.5783	0.1932	0.062*
C4	0.0410 (7)	0.3267 (6)	0.2224 (3)	0.0455 (13)
H4A	0.0111	0.3509	0.2712	0.055*
C5	0.0578 (7)	0.1521 (6)	0.2017 (3)	0.0398 (12)
C6	0.0975 (8)	0.1249 (6)	0.1277 (3)	0.0444 (13)
C7	0.0357 (9)	0.0019 (7)	0.2503 (3)	0.0536 (16)
H7A	0.0773	−0.1044	0.2312	0.064*
C8	−0.0358 (7)	−0.0091 (6)	0.3192 (3)	0.0405 (13)
C9	−0.0471 (7)	−0.1845 (7)	0.3543 (2)	0.0385 (12)
C10	−0.1892 (9)	−0.0062 (7)	0.4440 (4)	0.0508 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.1288 (16)	0.0311 (7)	0.0529 (9)	−0.0065 (9)	0.0132 (9)	−0.0105 (7)
S	0.0786 (10)	0.0240 (6)	0.0483 (8)	0.0039 (7)	0.0009 (7)	−0.0024 (6)

O1	0.125 (4)	0.026 (2)	0.053 (2)	0.017 (2)	0.017 (2)	−0.0015 (18)
O2	0.115 (4)	0.039 (2)	0.059 (3)	0.006 (2)	0.017 (3)	−0.012 (2)
N	0.065 (3)	0.032 (2)	0.038 (2)	0.004 (2)	0.014 (2)	0.0063 (19)
C1	0.084 (4)	0.037 (3)	0.042 (3)	−0.012 (3)	0.010 (3)	0.000 (3)
C2	0.094 (5)	0.026 (3)	0.060 (4)	−0.014 (3)	0.002 (3)	0.012 (3)
C3	0.073 (4)	0.026 (3)	0.056 (4)	−0.003 (3)	0.000 (3)	0.000 (2)
C4	0.060 (3)	0.029 (3)	0.047 (3)	0.002 (3)	−0.006 (3)	−0.007 (2)
C5	0.058 (3)	0.027 (2)	0.034 (3)	−0.001 (3)	−0.002 (2)	−0.003 (2)
C6	0.066 (4)	0.029 (3)	0.038 (3)	−0.012 (3)	0.003 (3)	−0.001 (2)
C7	0.089 (5)	0.021 (3)	0.050 (3)	0.004 (3)	−0.017 (3)	−0.004 (2)
C8	0.051 (3)	0.021 (2)	0.049 (3)	0.006 (2)	−0.010 (3)	−0.005 (2)
C9	0.053 (3)	0.040 (3)	0.023 (2)	0.004 (3)	0.014 (2)	0.000 (2)
C10	0.070 (4)	0.023 (3)	0.059 (4)	0.002 (3)	−0.015 (3)	−0.009 (3)

Geometric parameters (Å, °)

Cl—C6	1.747 (5)	C2—C3	1.369 (8)
S—C8	1.739 (5)	C2—H2A	0.9300
S—C10	1.785 (6)	C3—C4	1.343 (7)
O1—C9	1.204 (5)	C3—H3A	0.9300
O2—C10	1.179 (7)	C4—C5	1.383 (6)
N—C9	1.366 (6)	C4—H4A	0.9300
N—C10	1.374 (6)	C5—C6	1.387 (7)
N—H0A	0.8600	C5—C7	1.449 (7)
C1—C6	1.398 (7)	C7—C8	1.360 (8)
C1—C2	1.400 (8)	C7—H7A	0.9300
C1—H1A	0.9300	C8—C9	1.478 (7)
C8—S—C10	92.0 (3)	C4—C5—C7	125.3 (5)
C9—N—C10	119.3 (4)	C6—C5—C7	119.6 (5)
C9—N—H0A	120.4	C5—C6—C1	124.1 (5)
C10—N—H0A	120.4	C5—C6—Cl	120.9 (4)
C6—C1—C2	115.7 (5)	C1—C6—Cl	114.9 (4)
C6—C1—H1A	122.2	C8—C7—C5	130.6 (5)
C2—C1—H1A	122.2	C8—C7—H7A	114.7
C3—C2—C1	121.8 (5)	C5—C7—H7A	114.7
C3—C2—H2A	119.1	C7—C8—C9	118.3 (4)
C1—C2—H2A	119.1	C7—C8—S	130.8 (4)
C4—C3—C2	119.2 (5)	C9—C8—S	110.9 (4)
C4—C3—H3A	120.4	O1—C9—N	123.1 (5)
C2—C3—H3A	120.4	O1—C9—C8	127.6 (4)
C3—C4—C5	124.1 (5)	N—C9—C8	109.3 (4)
C3—C4—H4A	118.0	O2—C10—N	126.5 (5)
C5—C4—H4A	118.0	O2—C10—S	125.0 (4)
C4—C5—C6	115.1 (5)	N—C10—S	108.4 (4)
C6—C1—C2—C3	1.9 (10)	C5—C7—C8—S	−0.6 (10)
C1—C2—C3—C4	0.5 (10)	C10—S—C8—C7	175.9 (6)
C2—C3—C4—C5	−0.5 (10)	C10—S—C8—C9	−2.4 (4)
C3—C4—C5—C6	−1.7 (9)	C10—N—C9—O1	−177.0 (5)
C3—C4—C5—C7	178.8 (6)	C10—N—C9—C8	2.1 (7)

C4—C5—C6—C1	4.4 (9)	C7—C8—C9—O1	1.3 (9)
C7—C5—C6—C1	−176.2 (6)	S—C8—C9—O1	179.8 (5)
C4—C5—C6—Cl	−179.6 (4)	C7—C8—C9—N	−177.7 (5)
C7—C5—C6—Cl	−0.1 (8)	S—C8—C9—N	0.8 (6)
C2—C1—C6—C5	−4.5 (9)	C9—N—C10—O2	−180.0 (6)
C2—C1—C6—Cl	179.3 (5)	C9—N—C10—S	−3.8 (7)
C4—C5—C7—C8	13.4 (10)	C8—S—C10—O2	179.6 (6)
C6—C5—C7—C8	−166.0 (6)	C8—S—C10—N	3.4 (4)
C5—C7—C8—C9	177.5 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0A···O2^{i}	0.86	2.00	2.833 (6)	164
C4—H4A···S	0.93	2.63	3.326 (5)	132
C7—H7A···Cl	0.93	2.54	3.010 (6)	112
C7—H7A···O1	0.93	2.50	2.866 (7)	104

Symmetry codes: (i) −*x*−1/2, *y*−1/2, −*z*+1.

Fig. 1

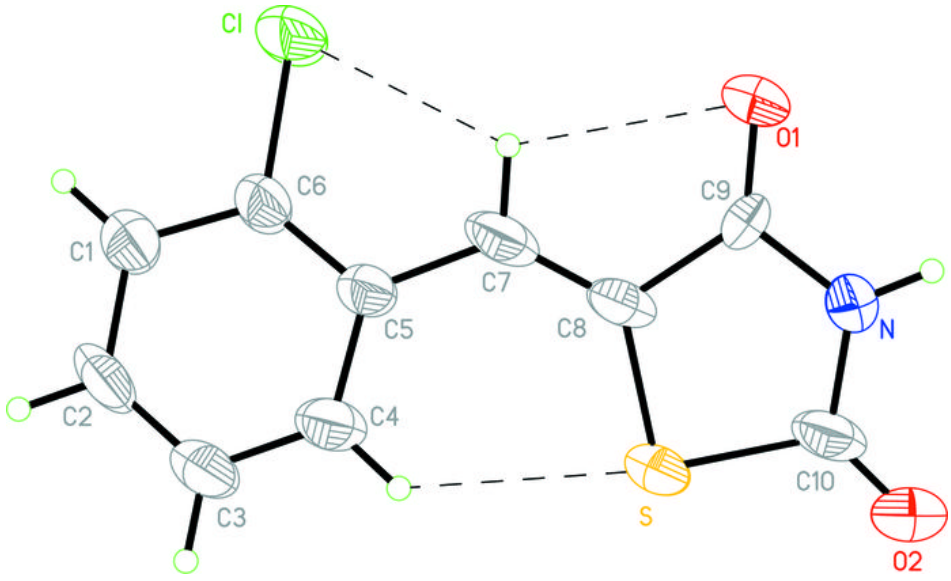

Fig. 2