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ISSN 2414-3146

(5*Z*)-5-(4-Chlorobenzylidene)-1,3-thiazolidine-2,4-dione

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Received 16 June 2016
Accepted 17 June 2016

Edited by D.-J. Xu, Zhejiang University (Yuquan Campus), China

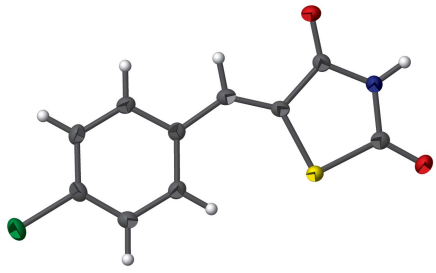
Keywords: crystal structure; 4-chlorophenyl ring; thiazolidine ring.

CCDC reference: 1486205

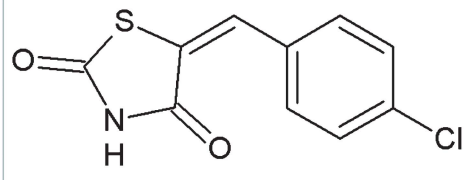
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{10}H_6ClNO_2S$, the dihedral angle between the planes of the 4-chlorophenyl and thiazolidine rings is 8.62 (9)°. In the crystal, molecules form undulating ribbons running approximately parallel to (101) through N—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

Thiazolidinedione scaffold compounds (TDZs), known as glitazones, are a class of medications used in the treatment of diabetes mellitus type 2. TZDs are an important heterocyclic ring system and have therapeutic importance when combined with other heterocyclic rings, as they can produce a wide range of biological activities, such as anti-inflammatory (Barros *et al.*, 2008), antitubercular (Pattan *et al.*, 2008), antimicrobial (Oya *et al.*, 2007) and cytotoxic (Shankar & Kallanagouda, 2012). In this context, we report here the synthesis and crystal structure of the title compound (Fig. 1).

The dihedral angle between the 4-chlorophenyl and thiazolidine rings is 8.62 (9)°. The molecules form undulating ribbons running approximately parallel to (101) through N1—H1···O2^{i} hydrogen bonds (Table 1 and Fig. 2).

Synthesis and crystallization

The title compound was obtained from a three components reaction between 2 mmol (234 mg) of thiazolidin-2,4-dione, 1 mmol (147.6 mg) of 4-chlorobenzaldehyde and 1 mmol (61 mg) of 2-aminoethanol in 30 ml ethanol. The reaction mixture was refluxed

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.91	1.87	2.782 (2)	175

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

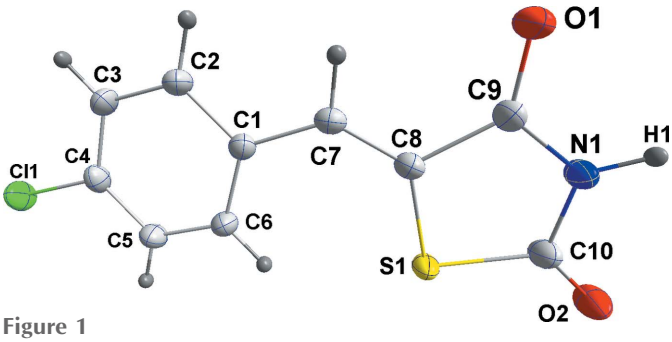


Figure 1
The title molecule, showing the atom labelling scheme and 50% probability ellipsoids.

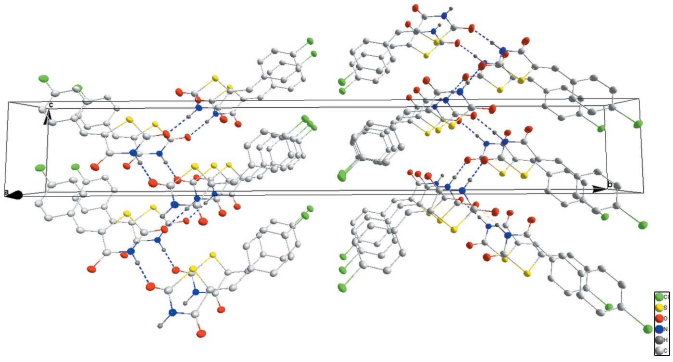


Figure 2
Packing viewed along the *a* axis, with N—H···O hydrogen bonds shown as dotted lines.

and monitored by TLC until completion. The resulting solid product was collected by filtration, dried under vacuum and recrystallized from ethanol to afford suitable quality crystals for X-ray diffraction.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{10}H_6ClNO_2S$
M_r	239.67
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	150
a, b, c (Å)	3.9098 (1), 40.7555 (15), 6.0917 (2)
β (°)	93.748 (1)
V (Å^3)	968.61 (5)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	5.33
Crystal size (mm)	0.36 × 0.13 × 0.10
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2015)
T_{min}, T_{max}	0.46, 0.63
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12027, 1885, 1815
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.088, 1.14
No. of reflections	1885
No. of parameters	136
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.37

Computer programs: *APEX2* and *SAINT* (Bruker, 2015), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

The support of NSF–MRI Grant No.1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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full crystallographic data

IUCrData (2016). **1**, x160988 [doi:10.1107/S2414314616009883]

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Crystal data

$C_{10}H_6ClNO_2S$
M_r = 239.67
Monoclinic, $P2_1/n$
a = 3.9098 (1) Å
b = 40.7555 (15) Å
c = 6.0917 (2) Å
β = 93.748 (1)°
V = 968.61 (5) Å^3
Z = 4
$F(000)$ = 488
D_x = 1.643 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9969 reflections
θ = 3.3–72.2°
μ = 5.33 mm^{-1}
T = 150 K
Column, colourless
0.36 × 0.13 × 0.10 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2015)
T_{min} = 0.46, T_{max} = 0.63
12027 measured reflections
1885 independent reflections
1815 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 72.2°, θ_{min} = 2.2°
h = −4→4
k = −50→50
l = −7→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.088
S = 1.14
1885 reflections
136 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 0.7319P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while that attached to nitrogen was placed in a location derived from a difference map and its coordinates adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.10718 (14)	0.47151 (2)	0.79460 (8)	0.03023 (16)
S1	0.42007 (12)	0.31099 (2)	0.30938 (7)	0.02236 (15)
O1	0.8931 (4)	0.33869 (3)	−0.1933 (2)	0.0282 (3)
O2	0.4488 (4)	0.25053 (3)	0.1464 (2)	0.0336 (4)
N1	0.6783 (4)	0.29170 (4)	−0.0508 (3)	0.0234 (4)
H1	0.7546	0.2778	−0.1536	0.028*
C1	0.4756 (5)	0.39611 (4)	0.3194 (3)	0.0199 (4)
C2	0.5011 (5)	0.42973 (5)	0.2761 (3)	0.0226 (4)
H2	0.5989	0.4367	0.1453	0.027*
C3	0.3868 (5)	0.45301 (5)	0.4197 (3)	0.0240 (4)
H3	0.4029	0.4758	0.3877	0.029*
C4	0.2489 (5)	0.44247 (5)	0.6107 (3)	0.0227 (4)
C5	0.2193 (5)	0.40946 (5)	0.6594 (3)	0.0231 (4)
H5	0.1236	0.4027	0.7916	0.028*
C6	0.3308 (5)	0.38626 (5)	0.5133 (3)	0.0219 (4)
H6	0.3088	0.3636	0.5450	0.026*
C7	0.6023 (5)	0.37354 (5)	0.1576 (3)	0.0210 (4)
H7	0.7114	0.3841	0.0420	0.025*
C8	0.5946 (5)	0.34092 (4)	0.1405 (3)	0.0197 (4)
C9	0.7404 (5)	0.32495 (5)	−0.0532 (3)	0.0215 (4)
C10	0.5164 (5)	0.27923 (5)	0.1228 (3)	0.0245 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0389 (3)	0.0259 (3)	0.0262 (3)	0.00532 (19)	0.0051 (2)	−0.00617 (17)
S1	0.0309 (3)	0.0182 (2)	0.0187 (3)	−0.00121 (17)	0.00752 (18)	0.00075 (15)
O1	0.0395 (8)	0.0235 (7)	0.0229 (7)	−0.0036 (6)	0.0129 (6)	0.0011 (5)
O2	0.0540 (10)	0.0182 (7)	0.0300 (8)	−0.0052 (6)	0.0147 (7)	0.0008 (6)
N1	0.0336 (9)	0.0177 (8)	0.0198 (8)	−0.0011 (6)	0.0080 (7)	−0.0013 (6)
C1	0.0221 (9)	0.0187 (9)	0.0187 (9)	0.0003 (7)	−0.0004 (7)	0.0006 (7)
C2	0.0277 (9)	0.0212 (9)	0.0192 (9)	−0.0019 (7)	0.0031 (7)	0.0024 (7)
C3	0.0317 (10)	0.0162 (9)	0.0239 (10)	−0.0008 (7)	0.0003 (8)	0.0008 (7)
C4	0.0236 (9)	0.0231 (9)	0.0211 (9)	0.0028 (7)	0.0001 (7)	−0.0035 (7)
C5	0.0254 (9)	0.0253 (9)	0.0191 (9)	−0.0005 (7)	0.0043 (7)	0.0014 (7)
C6	0.0262 (9)	0.0187 (9)	0.0209 (9)	−0.0006 (7)	0.0020 (7)	0.0021 (7)
C7	0.0231 (9)	0.0223 (9)	0.0178 (9)	−0.0009 (7)	0.0030 (7)	0.0031 (7)
C8	0.0217 (9)	0.0217 (9)	0.0159 (9)	−0.0002 (7)	0.0027 (7)	0.0011 (7)

C9	0.0248 (9)	0.0210 (9)	0.0188 (9)	0.0006 (7)	0.0024 (7)	−0.0003 (7)
C10	0.0309 (10)	0.0225 (10)	0.0203 (9)	−0.0011 (8)	0.0039 (8)	0.0010 (7)

Geometric parameters (Å, º)

Cl1—C4	1.7447 (19)	C2—C3	1.384 (3)
S1—C8	1.7619 (18)	C2—H2	0.9500
S1—C10	1.779 (2)	C3—C4	1.383 (3)
O1—C9	1.211 (2)	C3—H3	0.9500
O2—C10	1.210 (2)	C4—C5	1.384 (3)
N1—C10	1.365 (2)	C5—C6	1.388 (3)
N1—C9	1.377 (2)	C5—H5	0.9500
N1—H1	0.9099	C6—H6	0.9500
C1—C2	1.400 (3)	C7—C8	1.334 (3)
C1—C6	1.402 (3)	C7—H7	0.9500
C1—C7	1.458 (3)	C8—C9	1.493 (2)
C8—S1—C10	91.39 (9)	C4—C5—H5	120.3
C10—N1—C9	117.76 (16)	C6—C5—H5	120.3
C10—N1—H1	119.4	C5—C6—C1	120.44 (17)
C9—N1—H1	122.7	C5—C6—H6	119.8
C2—C1—C6	118.44 (17)	C1—C6—H6	119.8
C2—C1—C7	117.29 (17)	C8—C7—C1	132.41 (17)
C6—C1—C7	124.27 (17)	C8—C7—H7	113.8
C3—C2—C1	121.47 (18)	C1—C7—H7	113.8
C3—C2—H2	119.3	C7—C8—C9	119.25 (16)
C1—C2—H2	119.3	C7—C8—S1	130.70 (15)
C4—C3—C2	118.59 (17)	C9—C8—S1	110.01 (13)
C4—C3—H3	120.7	O1—C9—N1	123.94 (17)
C2—C3—H3	120.7	O1—C9—C8	125.93 (17)
C3—C4—C5	121.69 (17)	N1—C9—C8	110.12 (15)
C3—C4—Cl1	119.15 (15)	O2—C10—N1	124.62 (18)
C5—C4—Cl1	119.16 (15)	O2—C10—S1	124.78 (15)
C4—C5—C6	119.36 (18)	N1—C10—S1	110.60 (14)
C6—C1—C2—C3	0.0 (3)	C1—C7—C8—S1	0.7 (3)
C7—C1—C2—C3	179.82 (18)	C10—S1—C8—C7	174.7 (2)
C1—C2—C3—C4	0.8 (3)	C10—S1—C8—C9	−2.85 (15)
C2—C3—C4—C5	−0.8 (3)	C10—N1—C9—O1	177.09 (19)
C2—C3—C4—Cl1	179.45 (15)	C10—N1—C9—C8	−2.6 (2)
C3—C4—C5—C6	0.0 (3)	C7—C8—C9—O1	6.0 (3)
Cl1—C4—C5—C6	179.82 (15)	S1—C8—C9—O1	−176.14 (17)
C4—C5—C6—C1	0.7 (3)	C7—C8—C9—N1	−174.27 (17)
C2—C1—C6—C5	−0.7 (3)	S1—C8—C9—N1	3.6 (2)
C7—C1—C6—C5	179.44 (18)	C9—N1—C10—O2	−179.3 (2)
C2—C1—C7—C8	−174.5 (2)	C9—N1—C10—S1	0.5 (2)
C6—C1—C7—C8	5.3 (3)	C8—S1—C10—O2	−178.7 (2)
C1—C7—C8—C9	178.05 (19)	C8—S1—C10—N1	1.49 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.91	1.87	2.782 (2)	175

Symmetry code: (i) $x+1/2, -y+1/2, z-1/2$.