Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Hong-Shun Sun, Ye-Ming Xu, Wei He, Shi-Gui Tang and Cheng Guo*

Department of Applied Chemistry, College of Sciences, Nanjing University of Technolgy, Xinmofan Road No. 5, Nanjing 210009, People's Republic of China
Correspondence e-mail: guocheng@njut.edu.cn

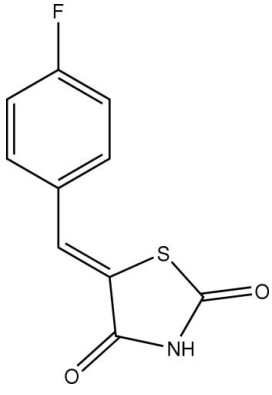
Received 30 November 2007; accepted 24 December 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; R factor = 0.054; wR factor = 0.121; data-to-parameter ratio = 7.8.

In the title compound, $C_{10}H_6FNO_2S$, the benzene and thiazolidine rings make a dihedral angle of 7.52 (3)°. Intramolecular C—H···O and C—H···S hydrogen bonds result in the formation of nearly planar five- and six-membered rings; the adjacent rings are nearly coplanar. 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 a related structure, see: Guo *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_6FNO_2S$
M_r = 223.22
Orthorhombic, *Fdd*2
a = 26.519 (5) Å
b = 36.509 (7) Å
c = 3.8490 (8) Å
V = 3726.6 (13) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 294 (2) K
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.905, T_{max} = 0.967
2087 measured reflections
1059 independent reflections
790 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.121
S = 1.04
1059 reflections
136 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Absolute structure: Flack (1983), no Friedel pairs
Flack parameter: 0.0 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O2^{i}	0.86	1.98	2.830 (5)	171
C5—H5A···S	0.93	2.54	3.241 (5)	133
C7—H7A···O2	0.93	2.50	2.870 (5)	104

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

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, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

The authors thank the Center for Testing and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2407).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Barreca, M. L., Balzarini, J., Chimirri, A., De Clercq, E., De Luca, L., Holtje, H. D., Holtje, M., Monforte, A. M., Monforte, P., Pannecouque, C., Rao, A. & Zappala, M. (2002). *J. Med. Chem.* **45**, 5410–5413.
Botti, P., Pallin, T. D. & Tam, J. P. (1996). *J. Am. Chem. Soc.* **118**, 10018–10024.
Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Guo, C., Zhang, D.-M., Tang, Q.-G. & Sun, H.-S. (2006). *Acta Cryst.* E**62**, o3994–o3995.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siemens (1996). *SHELXTL*. Version 5.0). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2008). E**64**, o524 [doi:10.1107/S1600536807068316]

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

Hong-Shun Sun, Ye-Ming Xu, Wei He, Shi-Gui Tang and Cheng Guo

S1. 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 ongoing studies in this area (Guo *et al.*, 2006), we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The intramolecular C—H···O and C—H···S hydrogen bonds (Table 1) result in the formations of nearly planar five- and six-membered rings; C (S/H5A/C4/C5/C7/C8) and D (O2/H7A/C7—C9). Rings A (C1—C6) and B (N/S/C8—C10) are, of course, planar and the dihedral angles between them are A/B = 7.52 (3)°, A/C = 4.73 (3)°, A/D = 6.90 (3)°, B/C = 3.63 (2)°, B/D = 2.39 (3)° and C/D = 4.56 (2)°. So, the adjacent rings are also nearly co-planar.

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

S2. Experimental

For the preparation of the title compound, thiazolidine-2,4-dione (10 mmol) and 4-fluorobenzaldehyde (10 mmol) were dissolved in ethanol (10 ml) in a round-bottomed flask (50 ml) and 5 drops of piperidine were added. The flask was heated in a modified domestic microwave oven at 300 W for 5 min. After cooling, the mixture was poured into water and the crude compound (I) filtered out. Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

S3. 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*).

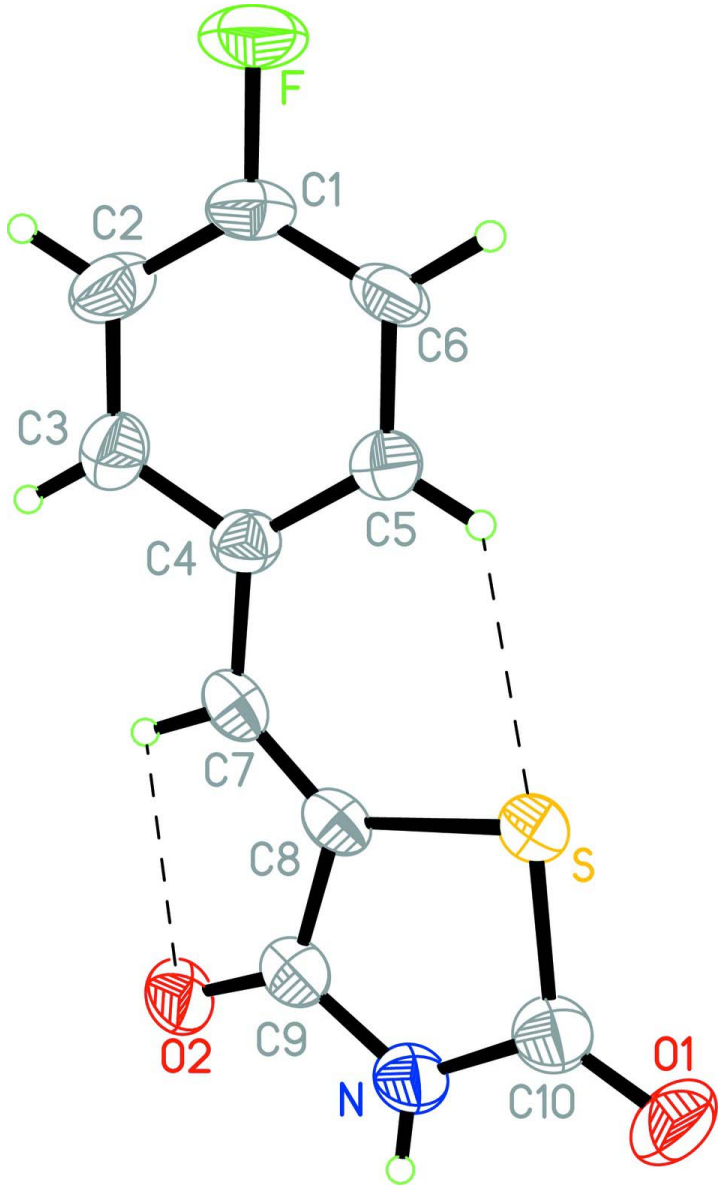


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines.

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

Crystal data

$C_{10}H_6FNO_2S$
$M_r = 223.22$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 26.519\ (5)$ Å
$b = 36.509\ (7)$ Å
$c = 3.8490\ (8)$ Å
$V = 3726.6\ (13)$ Å^3
$Z = 16$
$F(000) = 1824$
$D_x = 1.591$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.34$ mm^{-1}
$T = 294$ K
Block, colorles
0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.905, T_{max} = 0.967
2087 measured reflections
1059 independent reflections
790 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.0°, θ_{min} = 1.9°
h = 0→32
k = 0→44
l = −4→0
3 standard reflections every 120 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.121
S = 1.04
1059 reflections
136 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Absolute structure: Flack (1983), no Friedel pairs
Absolute structure parameter: 0.0 (2)

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 $F^2 > \sigma(F^2)$ 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}
S	0.08943 (4)	−0.00189 (3)	−0.0089 (5)	0.0379 (3)
F	−0.06109 (11)	0.13395 (8)	0.6207 (12)	0.0681 (12)
O1	0.13745 (13)	−0.05739 (9)	−0.3062 (14)	0.0619 (12)
O2	0.21942 (10)	0.03754 (8)	0.2142 (14)	0.0493 (10)
N	0.18501 (14)	−0.01228 (9)	−0.0627 (14)	0.0425 (11)
H0A	0.2136	−0.0219	−0.1150	0.051*
C1	−0.01787 (17)	0.11646 (12)	0.5430 (15)	0.0416 (12)
C2	0.02675 (18)	0.13443 (12)	0.5921 (16)	0.0484 (15)
H2A	0.0273	0.1584	0.6744	0.058*
C3	0.07078 (19)	0.11621 (11)	0.5166 (17)	0.0444 (13)
H3A	0.1015	0.1278	0.5559	0.053*
C4	0.07035 (16)	0.08082 (10)	0.3826 (13)	0.0346 (11)
C5	0.02378 (16)	0.06382 (12)	0.3314 (15)	0.0400 (12)
H5A	0.0225	0.0402	0.2421	0.048*

C6	−0.02016 (15)	0.08195 (12)	0.4125 (16)	0.0452 (14)
H6A	−0.0512	0.0707	0.3782	0.054*
C7	0.11827 (16)	0.06366 (11)	0.3095 (14)	0.0352 (10)
H7A	0.1462	0.0772	0.3782	0.042*
C8	0.12929 (15)	0.03151 (11)	0.1597 (14)	0.0317 (10)
C9	0.18224 (17)	0.02026 (11)	0.1107 (15)	0.0370 (12)
C10	0.14053 (17)	−0.02927 (12)	−0.1510 (17)	0.0450 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0303 (5)	0.0391 (5)	0.0444 (6)	−0.0005 (4)	0.0015 (7)	−0.0028 (6)
F	0.0400 (16)	0.0705 (18)	0.094 (3)	0.0212 (14)	0.003 (2)	−0.024 (2)
O1	0.058 (2)	0.0465 (17)	0.081 (3)	0.0105 (16)	0.000 (3)	−0.026 (2)
O2	0.0267 (16)	0.0444 (16)	0.077 (3)	0.0002 (12)	−0.0039 (19)	0.001 (2)
N	0.0295 (18)	0.0403 (18)	0.058 (3)	0.0068 (15)	0.001 (2)	0.001 (2)
C1	0.033 (2)	0.051 (2)	0.041 (3)	0.012 (2)	0.003 (2)	−0.009 (3)
C2	0.047 (3)	0.042 (2)	0.056 (4)	0.012 (2)	−0.009 (3)	−0.018 (3)
C3	0.047 (3)	0.036 (2)	0.050 (3)	−0.0028 (19)	−0.002 (3)	−0.007 (3)
C4	0.031 (2)	0.036 (2)	0.036 (3)	0.0030 (17)	−0.002 (2)	−0.002 (2)
C5	0.035 (2)	0.038 (2)	0.047 (3)	0.0012 (17)	−0.001 (2)	−0.006 (2)
C6	0.0209 (18)	0.056 (3)	0.058 (4)	−0.0019 (18)	0.007 (2)	−0.018 (3)
C7	0.030 (2)	0.042 (2)	0.033 (3)	−0.0057 (17)	0.000 (2)	0.008 (2)
C8	0.024 (2)	0.040 (2)	0.031 (3)	0.0007 (16)	−0.003 (2)	0.009 (2)
C9	0.032 (2)	0.037 (2)	0.042 (3)	0.0039 (18)	−0.001 (2)	0.011 (2)
C10	0.038 (2)	0.042 (2)	0.055 (4)	0.0065 (19)	−0.002 (3)	0.002 (3)

Geometric parameters (Å, º)

S—C8	1.739 (4)	C2—H2A	0.9300
S—C10	1.770 (5)	C3—C4	1.391 (6)
F—C1	1.346 (5)	C3—H3A	0.9300
O1—C10	1.191 (6)	C4—C5	1.396 (6)
O2—C9	1.237 (5)	C4—C7	1.445 (6)
N—C9	1.364 (6)	C5—C6	1.376 (6)
N—C10	1.375 (6)	C5—H5A	0.9300
N—H0A	0.8600	C6—H6A	0.9300
C1—C6	1.358 (6)	C7—C8	1.340 (6)
C1—C2	1.366 (7)	C7—H7A	0.9300
C2—C3	1.375 (6)	C8—C9	1.475 (6)
C8—S—C10	92.6 (2)	C6—C5—H5A	119.9
C9—N—C10	117.9 (4)	C4—C5—H5A	119.9
C9—N—H0A	121.1	C1—C6—C5	119.5 (4)
C10—N—H0A	121.1	C1—C6—H6A	120.3
F—C1—C6	119.0 (4)	C5—C6—H6A	120.3
F—C1—C2	118.6 (4)	C8—C7—C4	131.0 (4)
C6—C1—C2	122.4 (4)	C8—C7—H7A	114.5

C1—C2—C3	118.3 (4)	C4—C7—H7A	114.5
C1—C2—H2A	120.9	C7—C8—C9	120.4 (4)
C3—C2—H2A	120.9	C7—C8—S	129.9 (3)
C2—C3—C4	121.4 (4)	C9—C8—S	109.6 (3)
C2—C3—H3A	119.3	O2—C9—N	124.0 (4)
C4—C3—H3A	119.3	O2—C9—C8	125.1 (4)
C3—C4—C5	118.2 (4)	N—C9—C8	110.9 (4)
C3—C4—C7	117.9 (4)	O1—C10—N	124.9 (4)
C5—C4—C7	123.9 (4)	O1—C10—S	126.1 (4)
C6—C5—C4	120.2 (4)	N—C10—S	109.0 (3)
F—C1—C2—C3	179.1 (5)	C4—C7—C8—S	0.6 (9)
C6—C1—C2—C3	−2.2 (9)	C10—S—C8—C7	177.2 (5)
C1—C2—C3—C4	2.1 (9)	C10—S—C8—C9	−1.5 (4)
C2—C3—C4—C5	−1.0 (8)	C10—N—C9—O2	177.5 (5)
C2—C3—C4—C7	179.8 (5)	C10—N—C9—C8	−1.9 (6)
C3—C4—C5—C6	0.0 (8)	C7—C8—C9—O2	3.9 (8)
C7—C4—C5—C6	179.1 (5)	S—C8—C9—O2	−177.2 (4)
F—C1—C6—C5	179.8 (5)	C7—C8—C9—N	−176.7 (4)
C2—C1—C6—C5	1.2 (9)	S—C8—C9—N	2.2 (5)
C4—C5—C6—C1	−0.1 (9)	C9—N—C10—O1	178.9 (6)
C3—C4—C7—C8	−174.7 (5)	C9—N—C10—S	0.7 (6)
C5—C4—C7—C8	6.3 (9)	C8—S—C10—O1	−177.6 (6)
C4—C7—C8—C9	179.3 (5)	C8—S—C10—N	0.6 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0*A*···O2^{i}	0.86	1.98	2.830 (5)	171
C5—H5*A*···S	0.93	2.54	3.241 (5)	133
C7—H7*A*···O2	0.93	2.50	2.870 (5)	104

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