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

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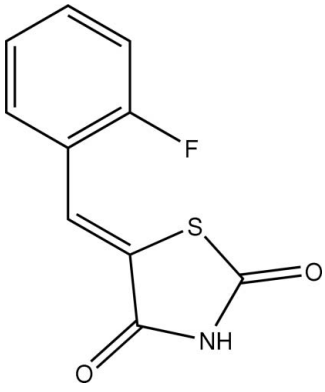
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.060; wR factor = 0.173; data-to-parameter ratio = 13.6.

In the title compound, $C_{10}H_6FNO_2S$, the benzene and thiazolidine rings are oriented at a dihedral angle of 8.90 (3)°. Intramolecular C—H···O, C—H···F and C—H···S hydrogen bonds result in the formation of two nearly planar five-membered rings and one non-planar six-membered ring, the five-membered rings being also nearly coplanar with the adjacent rings. In the crystal structure, intermolecular N—H···O and C—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
Monoclinic, $P2_1/c$
a = 5.120 (1) Å
b = 21.189 (4) Å
c = 9.0310 (18) Å
β = 105.49 (3)°
V = 944.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 294 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.878, T_{max} = 0.967
2060 measured reflections
1853 independent reflections
1283 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.173
S = 1.00
1853 reflections
136 parameters
48 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O1^{i}	0.86	2.00	2.846 (5)	170
C2—H2A···O2^{ii}	0.93	2.57	3.243 (6)	130
C5—H5A···S	0.93	2.56	3.248 (5)	131
C6—H6A···O2^{iii}	0.93	2.51	3.417 (6)	165
C7—H7A···F	0.93	2.33	2.717 (6)	105
C7—H7A···O1	0.93	2.54	2.889 (6)	102

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z+1$; (iii) $x-1, -y+\frac{3}{2}, z+\frac{1}{2}$.

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2351).

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supplementary materials

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(*Z*)-5-(2-Fluorobenzylidene)-1,3-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 (Guo *et al.*, 2006), we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The intramolecular C—H···O, C—H···F and C—H···S hydrogen bonds (Table 1) result in the formation of the nearly planar five-membered rings; C (F/C3/C4/C7/H7A) and D (O1/C7–C9/H7A), and one non-planar six-membered ring E (S/C4/C5/C7/C8/H5A), the five-membered rings being also nearly co-planar with the adjacent rings A (C1–C6) and B (S/N/C8–C10). The dihedral angles between them are A/C = 2.08 (2)° and B/D = 1.18 (3)°. The planar rings A (C1–C6) and B (S/N/C8–C10) are oriented at a dihedral angle of 8.90 (3)°.

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

Experimental

Thiazolidine-2,4-dione (10 mmol) and 2-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, the crude compound filtered out, and recrystallized from ethanol. Crystals of (I) suitable for X-ray analysis were obtained 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

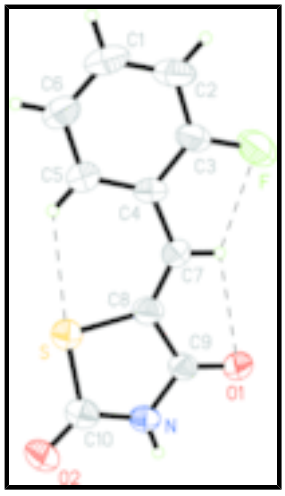

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines.

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

Crystal data

$C_{10}H_6FNO_2S$	F_{000} = 456
M_r = 223.22	D_x = 1.570 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
a = 5.120 (1) Å	θ = 9–13º
b = 21.189 (4) Å	μ = 0.33 mm^{-1}
c = 9.0310 (18) Å	T = 294 (2) K
β = 105.49 (3)º	Block, colourless
V = 944.2 (3) Å^3	0.40 × 0.10 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.043
Radiation source: fine-focus sealed tube	θ_{max} = 25.9º
Monochromator: graphite	θ_{min} = 1.9º
T = 294(2) K	h = −6→6
ω/2θ scans	k = 0→26
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→11
T_{min} = 0.878, T_{max} = 0.967	3 standard reflections
2060 measured reflections	every 120 min
1853 independent reflections	intensity decay: none
1283 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
$R[F^2 > 2\sigma(F^2)] = 0.060$	H-atom parameters constrained
$wR(F^2) = 0.173$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 3P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
1853 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
136 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
48 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
S	0.4369 (2)	0.65416 (5)	0.48345 (13)	0.0472 (3)
N	0.7813 (7)	0.57028 (17)	0.4442 (4)	0.0435 (9)
H0A	0.8957	0.5506	0.4065	0.052*
F	0.2877 (8)	0.53827 (18)	0.9920 (4)	0.0975 (13)
O1	0.8214 (7)	0.50293 (14)	0.6463 (4)	0.0489 (8)
C1	−0.0740 (10)	0.6837 (3)	0.9594 (6)	0.0605 (13)
H1A	−0.1900	0.7048	1.0060	0.073*
O2	0.6738 (7)	0.64701 (16)	0.2582 (4)	0.0585 (9)
C2	0.0270 (10)	0.6253 (3)	1.0131 (5)	0.0589 (13)
H2A	−0.0176	0.6068	1.0965	0.071*
C3	0.1953 (10)	0.5952 (2)	0.9402 (5)	0.0527 (12)
C4	0.2695 (8)	0.6197 (2)	0.8152 (5)	0.0389 (9)
C5	0.1655 (10)	0.6795 (2)	0.7646 (5)	0.0520 (11)
H5A	0.2109	0.6984	0.6819	0.062*
C6	−0.0040 (11)	0.7108 (2)	0.8368 (6)	0.0591 (13)
H6A	−0.0711	0.7504	0.8021	0.071*
C7	0.4484 (8)	0.5833 (2)	0.7487 (5)	0.0408 (10)

H7A	0.5179	0.5470	0.8029	0.049*
C8	0.5305 (8)	0.5934 (2)	0.6216 (5)	0.0400 (9)
C9	0.7224 (8)	0.55010 (19)	0.5755 (5)	0.0374 (9)
C10	0.6489 (9)	0.6235 (2)	0.3753 (5)	0.0462 (10)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0537 (7)	0.0454 (6)	0.0477 (6)	0.0082 (5)	0.0226 (5)	0.0061 (5)
N	0.048 (2)	0.046 (2)	0.046 (2)	0.0069 (16)	0.0288 (17)	0.0035 (16)
F	0.124 (3)	0.101 (3)	0.094 (3)	0.053 (2)	0.075 (2)	0.051 (2)
O1	0.061 (2)	0.0406 (16)	0.0552 (19)	0.0089 (14)	0.0323 (16)	0.0024 (14)
C1	0.054 (3)	0.074 (3)	0.057 (3)	0.011 (2)	0.022 (2)	−0.020 (2)
O2	0.073 (2)	0.062 (2)	0.0510 (19)	0.0049 (18)	0.0348 (17)	0.0121 (16)
C2	0.055 (3)	0.085 (3)	0.045 (2)	0.008 (3)	0.027 (2)	−0.003 (2)
C3	0.062 (3)	0.063 (3)	0.041 (2)	0.012 (2)	0.027 (2)	0.007 (2)
C4	0.037 (2)	0.047 (2)	0.037 (2)	0.0007 (18)	0.0169 (17)	−0.0062 (17)
C5	0.064 (3)	0.047 (2)	0.052 (3)	0.009 (2)	0.027 (2)	0.000 (2)
C6	0.070 (3)	0.049 (3)	0.066 (3)	0.008 (2)	0.031 (3)	−0.010 (2)
C7	0.044 (2)	0.039 (2)	0.042 (2)	0.0083 (18)	0.0166 (19)	0.0022 (18)
C8	0.042 (2)	0.042 (2)	0.040 (2)	−0.0025 (18)	0.0171 (18)	−0.0029 (18)
C9	0.039 (2)	0.038 (2)	0.040 (2)	−0.0034 (18)	0.0188 (18)	−0.0008 (18)
C10	0.044 (2)	0.054 (3)	0.042 (2)	−0.001 (2)	0.0143 (19)	0.000 (2)

Geometric parameters (Å, °)

S—C10	1.767 (5)	C2—C3	1.374 (6)
S—C8	1.767 (4)	C2—H2A	0.9300
N—C9	1.368 (5)	C3—C4	1.385 (6)
N—C10	1.376 (6)	C4—C5	1.403 (6)
N—H0A	0.8600	C4—C7	1.444 (5)
F—C3	1.333 (6)	C5—C6	1.385 (6)
O1—C9	1.221 (5)	C5—H5A	0.9300
C1—C6	1.377 (7)	C6—H6A	0.9300
C1—C2	1.378 (7)	C7—C8	1.341 (6)
C1—H1A	0.9300	C7—H7A	0.9300
O2—C10	1.206 (5)	C8—C9	1.483 (6)
C10—S—C8	91.8 (2)	C6—C5—H5A	119.7
C9—N—C10	116.9 (3)	C4—C5—H5A	119.7
C9—N—H0A	121.6	C1—C6—C5	120.6 (5)
C10—N—H0A	121.6	C1—C6—H6A	119.7
C6—C1—C2	120.2 (4)	C5—C6—H6A	119.7
C6—C1—H1A	119.9	C8—C7—C4	130.5 (4)
C2—C1—H1A	119.9	C8—C7—H7A	114.8
C3—C2—C1	118.2 (5)	C4—C7—H7A	114.8
C3—C2—H2A	120.9	C7—C8—C9	121.6 (4)
C1—C2—H2A	120.9	C7—C8—S	129.2 (3)
F—C3—C2	117.5 (4)	C9—C8—S	109.2 (3)

F—C3—C4	118.4 (4)	O1—C9—N	123.6 (4)
C2—C3—C4	124.0 (5)	O1—C9—C8	125.0 (4)
C3—C4—C5	116.2 (4)	N—C9—C8	111.4 (4)
C3—C4—C7	118.7 (4)	O2—C10—N	125.5 (4)
C5—C4—C7	125.1 (4)	O2—C10—S	123.9 (4)
C6—C5—C4	120.7 (4)	N—C10—S	110.6 (3)
C6—C1—C2—C3	−0.7 (8)	C4—C7—C8—S	−2.9 (8)
C1—C2—C3—F	−178.9 (5)	C10—S—C8—C7	−179.8 (4)
C1—C2—C3—C4	−0.3 (8)	C10—S—C8—C9	−1.2 (3)
F—C3—C4—C5	179.6 (5)	C10—N—C9—O1	178.5 (4)
C2—C3—C4—C5	1.0 (7)	C10—N—C9—C8	−2.9 (5)
F—C3—C4—C7	−1.5 (7)	C7—C8—C9—O1	−0.3 (7)
C2—C3—C4—C7	179.9 (5)	S—C8—C9—O1	−179.0 (4)
C3—C4—C5—C6	−0.8 (7)	C7—C8—C9—N	−178.8 (4)
C7—C4—C5—C6	−179.6 (4)	S—C8—C9—N	2.4 (4)
C2—C1—C6—C5	0.9 (8)	C9—N—C10—O2	−178.8 (4)
C4—C5—C6—C1	−0.1 (8)	C9—N—C10—S	1.9 (5)
C3—C4—C7—C8	173.4 (5)	C8—S—C10—O2	−179.6 (4)
C5—C4—C7—C8	−7.8 (8)	C8—S—C10—N	−0.3 (3)
C4—C7—C8—C9	178.7 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0A···O1^{i}	0.86	2.00	2.846 (5)	170
C2—H2A···O2^{ii}	0.93	2.57	3.243 (6)	130
C5—H5A···S	0.93	2.56	3.248 (5)	131
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Symmetry codes: (i) −*x*+2, −*y*+1, −*z*+1; (ii) *x*−1, *y*, *z*+1; (iii) *x*−1, −*y*+3/2, *z*+1/2.

Fig. 1