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(*E*)-5-{4-[2-(5-Ethylpyridin-2-yl)ethoxy]benzylidene}thiazolidine-2,4-dione

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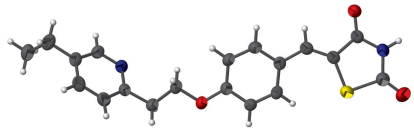
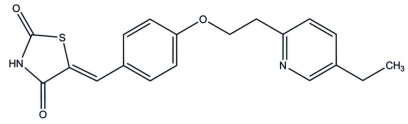
In the title compound, $C_{19}H_{18}N_2O_3S$, the thiazolidine ring makes dihedral angles of 46.97 (8) and 7.19 (9)° with the pyridine and benzene rings, respectively. The intramolecular structure is stabilized by a weak C—H···S hydrogen bond, which generates a $S(6)$ graph-set motif, and a weak C—H···O contact. In the crystal, N—H···N and C—H···O hydrogen bonds leads to infinite one-dimensional chains along (201) and generate an $R_2^2(7)$ ring-set motif. The crystal structure is further consolidated by weak π–π [centroid-to-centroid distance = 3.8204 (10) Å] interactions.

3D view



Chemical scheme



Structure description

Thiazolidinediones are known to sensitize tissues to insulin have been developed and clinically used as antidiabetic agents. They have been shown to reduce plasma glucose and lipid levels and are used for the treatment of type 2 diabetes (Day, 1999; Spiegelman, 1998). In view of this biological importance, the crystal structure of the title compound (Fig. 1) been determined and the results are presented here.

The geometric parameters for the title compound agree with those of reported similar structures (Vijayakumar *et al.*, 2012; Xiong *et al.*, 2011). The thiazolidine ring is planar [r.m.s. deviation = 0.007 (1) Å] and makes dihedral angles of 46.97 (8) and 7.19 (9)° with the pyridine and benzene rings, respectively. The intramolecular structure is stabilized by a weak C—H···S hydrogen bond, which generates an $S(6)$ graph-set motif (Fig. 1) and a weak C—H···O contact (Table 1).

In the crystal, N—H···N and C—H···O hydrogen bonds generate an $R_2^2(7)$ motif (Figs. 2 and 3) and lead to the formation of infinite chains along (201). The structure is

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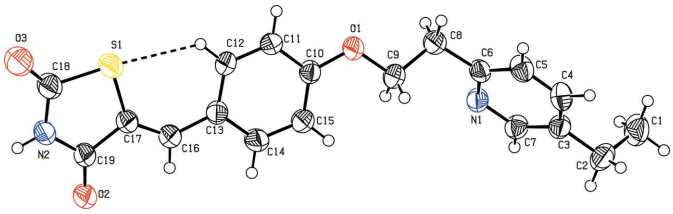

Figure 1
The molecular structure of the title compound, with the atom labelling and 30% probability displacement ellipsoids.

further consolidated by a weak π–π [centroid-to-centroid distance = 3.8204 (10) Å] interaction.

Synthesis and crystallization

4-[2-(5-Ethyl-2-pyridyl)ethoxy]benzaldehyde (600 mg, 2.32 mmol) and 2, 4- thiozolidindione (299 mg, 2.55 mmol) were dissolved in methanol (7 ml) together with a catalytic amount of piperidine (1.85 mmol). The yellow mixture was heated under reflux overnight. The suspension was acidified with acetic acid (140 mg, 2.3 mmol) and stirred for one additional hour after the addition of methanol (5 ml). The mixture was cooled in an ice bath, and the resulting solid was filtered, washed with methanol and dried under vacuum. Single crystals suitable for X-ray diffraction studies were obtained by slow evaporation of a solution of the title compound in dimethyl formamide at room temperature.

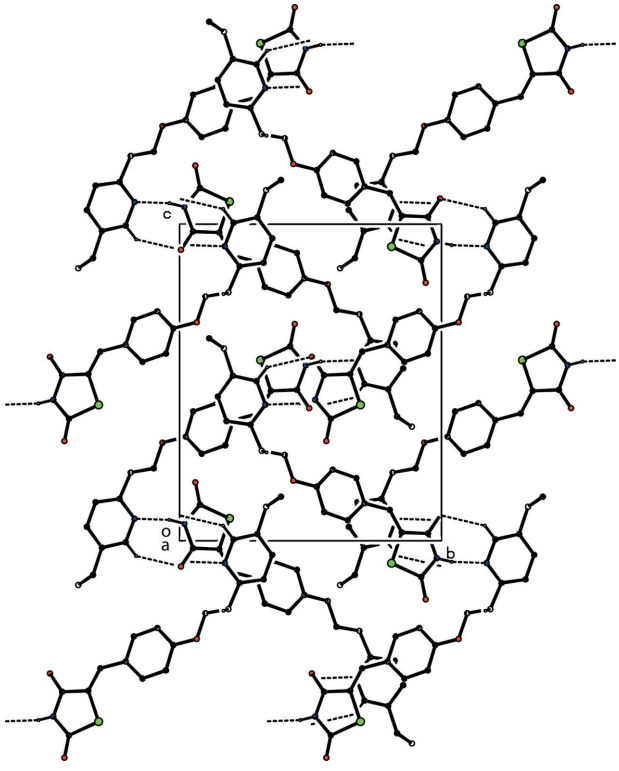

Figure 2
The crystal packing viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···S1	0.93	2.63	3.3166 (16)	131
C16—H16···O2	0.93	2.49	2.861 (2)	104
N2—H2···N1^{i}	0.86 (1)	2.00 (1)	2.8474 (19)	169 (2)
C7—H7···O2^{ii}	0.93	2.53	3.310 (2)	142

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{18}N_2O_3S$
M_r	354.41
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, b, c (Å)	7.6756 (2), 13.6762 (3), 17.6561 (4)
β (°)	110.442 (2)
V (Å^3)	1736.70 (7)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.83
Crystal size (mm)	0.32 × 0.28 × 0.24
Data collection	
Diffractometer	Bruker *APEX2* CCD Diffractometer
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.514, 0.668
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6076, 3314, 2894
R_{int}	0.013
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.616
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.115, 1.05
No. of reflections	3314
No. of parameters	231
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.22

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2004), *SHELXS2016/6* (Sheldrick, 2008), *SHELXL2016/6* (Sheldrick, 2015 and *PLATON* (Spek, 2009).

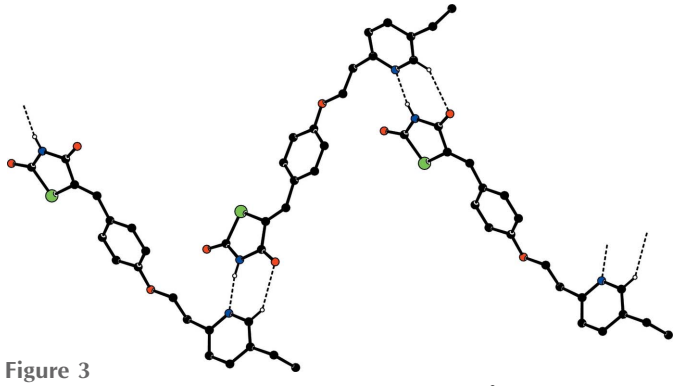

Figure 3
Partial packing of the crystal structure showing the $R_2^2(7)$ graph-set motif.

Refinement

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

References

Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Day, C. (1999). *Diabet. Med.* **16**, 179–192.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Spiegelman, B. M. (1998). *Diabetes*, **47**, 507–514.
Vijayakumar, S., Murugavel, S., Kannan, D. & Bakthadoss, M. (2012). *Acta Cryst.* E**68**, o156–o157.
Xiong, L.-Y., Wang, T.-F., Zheng, L.-P., Zhang, C. & Wang, F.-C. (2011). *Acta Cryst.* E**67**, o16.

full crystallographic data

IUCrData (2018). **3**, x171839 [https://doi.org/10.1107/S2414314617018399]

(*E*)-5-{4-[2-(5-Ethylpyridin-2-yl)ethoxy]benzylidene}thiazolidine-2,4-dione

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(*E*)-5-{4-[2-(5-Ethylpyridin-2-yl)ethoxy]benzylidene}thiazolidine- 2,4-dione

Crystal data

$C_{19}H_{18}N_2O_3S$
M_r = 354.41
Monoclinic, $P2_1/c$
a = 7.6756 (2) Å
b = 13.6762 (3) Å
c = 17.6561 (4) Å
β = 110.442 (2)°
V = 1736.70 (7) Å^3
Z = 4
$F(000)$ = 744
D_x = 1.355 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 2884 reflections
θ = 3.2–71.7°
μ = 1.83 mm^{-1}
T = 295 K
Needle, colourless
0.32 × 0.28 × 0.24 mm

Data collection

Bruker APEX2 CCD Diffractometer
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.514, T_{max} = 0.668
6076 measured reflections
3314 independent reflections
2894 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 71.7°, θ_{min} = 4.2°
h = −9→4
k = −16→16
l = −19→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 1.05
3314 reflections
231 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.3059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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. C-bound H atoms were placed in calculated positions and allowed to ride on their carrier atoms, with C—H = 0.93 Å (aromatic CH), 0.97 Å for CH_2, or 0.96 Å (methyl CH), and with U_{iso} = 1.5Ueq(methyl C) and U_{iso} = 1.2Ueq(aromatic and methylene C). H atom for NH group was located in difference-Fourier maps and refined with a distance restraint N—H = 0.86 (1) Å.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2212 (3)	0.88704 (17)	0.36053 (12)	0.0697 (6)
H1A	0.119617	0.845372	0.359311	0.105*
H1B	0.203120	0.909732	0.306865	0.105*
H1C	0.226556	0.942020	0.395081	0.105*
C2	0.4006 (3)	0.83058 (16)	0.39245 (11)	0.0587 (5)
H2A	0.501190	0.871525	0.389495	0.070*
H2B	0.392555	0.774043	0.358220	0.070*
C3	0.4456 (2)	0.79665 (12)	0.47862 (10)	0.0442 (4)
C4	0.5414 (2)	0.85396 (13)	0.54435 (11)	0.0508 (4)
H4	0.584151	0.915343	0.536308	0.061*
C5	0.5742 (2)	0.82076 (12)	0.62190 (11)	0.0494 (4)
H5	0.638544	0.859367	0.666206	0.059*
C6	0.5099 (2)	0.72919 (12)	0.63283 (9)	0.0420 (3)
C7	0.3879 (2)	0.70610 (12)	0.49544 (10)	0.0444 (4)
H7	0.323915	0.666059	0.452054	0.053*
C8	0.5444 (3)	0.68656 (13)	0.71561 (10)	0.0527 (4)
H8A	0.602405	0.735354	0.756531	0.063*
H8B	0.426978	0.667993	0.720636	0.063*
C9	0.6681 (3)	0.59875 (13)	0.72869 (10)	0.0500 (4)
H9A	0.605655	0.547305	0.691233	0.060*
H9B	0.781431	0.615675	0.719115	0.060*
C10	0.7955 (2)	0.47641 (11)	0.82902 (9)	0.0407 (3)
C11	0.8401 (2)	0.44784 (12)	0.90892 (9)	0.0441 (4)
H11	0.814895	0.489596	0.945378	0.053*
C12	0.9210 (2)	0.35853 (12)	0.93485 (9)	0.0447 (4)
H12	0.950844	0.340781	0.988757	0.054*
C13	0.9592 (2)	0.29401 (11)	0.88119 (10)	0.0416 (3)
C14	0.9161 (3)	0.32535 (13)	0.80174 (10)	0.0521 (4)
H14	0.941577	0.283999	0.765103	0.063*
C15	0.8372 (3)	0.41527 (13)	0.77520 (10)	0.0521 (4)
H15	0.812238	0.434561	0.721943	0.062*
C16	1.0401 (2)	0.19743 (11)	0.90231 (10)	0.0453 (4)
H16	1.042094	0.160700	0.858298	0.054*
C17	1.1124 (2)	0.15138 (11)	0.97345 (10)	0.0418 (3)
C18	1.2431 (3)	0.07767 (14)	1.11410 (11)	0.0561 (4)
C19	1.1908 (2)	0.05152 (12)	0.97676 (10)	0.0447 (4)
N1	0.41797 (19)	0.67230 (10)	0.57002 (8)	0.0433 (3)
N2	1.2580 (2)	0.01780 (10)	1.05478 (9)	0.0493 (3)
H2	1.310 (3)	−0.0389 (10)	1.0662 (14)	0.076 (7)*
O1	0.71184 (17)	0.56551 (9)	0.80991 (6)	0.0499 (3)
O2	1.1955 (2)	0.00511 (9)	0.91897 (8)	0.0638 (4)
O3	1.2938 (3)	0.06030 (13)	1.18489 (8)	0.0859 (6)
S1	1.13659 (7)	0.19066 (3)	1.07087 (3)	0.05382 (16)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0856 (15)	0.0638 (12)	0.0490 (10)	0.0104 (11)	0.0101 (10)	0.0119 (9)
C2	0.0670 (11)	0.0661 (12)	0.0466 (10)	0.0021 (9)	0.0245 (9)	0.0125 (9)
C3	0.0437 (8)	0.0469 (9)	0.0432 (8)	0.0048 (7)	0.0168 (7)	0.0076 (7)
C4	0.0541 (10)	0.0378 (8)	0.0571 (10)	−0.0033 (7)	0.0151 (8)	0.0061 (7)
C5	0.0530 (9)	0.0399 (8)	0.0458 (9)	0.0013 (7)	0.0051 (7)	−0.0032 (7)
C6	0.0472 (8)	0.0381 (8)	0.0378 (8)	0.0093 (6)	0.0110 (6)	0.0023 (6)
C7	0.0494 (9)	0.0443 (8)	0.0370 (8)	−0.0022 (7)	0.0121 (7)	−0.0008 (6)
C8	0.0703 (11)	0.0491 (10)	0.0365 (8)	0.0144 (8)	0.0160 (8)	0.0037 (7)
C9	0.0609 (10)	0.0511 (10)	0.0363 (8)	0.0125 (8)	0.0149 (7)	0.0087 (7)
C10	0.0448 (8)	0.0378 (8)	0.0372 (8)	0.0018 (6)	0.0113 (6)	0.0024 (6)
C11	0.0558 (9)	0.0418 (8)	0.0359 (7)	0.0060 (7)	0.0173 (7)	0.0004 (6)
C12	0.0544 (9)	0.0434 (8)	0.0369 (7)	0.0045 (7)	0.0168 (7)	0.0071 (6)
C13	0.0453 (8)	0.0383 (8)	0.0402 (8)	−0.0002 (6)	0.0138 (6)	0.0024 (6)
C14	0.0691 (11)	0.0472 (9)	0.0386 (8)	0.0113 (8)	0.0172 (8)	−0.0028 (7)
C15	0.0702 (11)	0.0507 (10)	0.0331 (8)	0.0125 (8)	0.0152 (7)	0.0039 (7)
C16	0.0545 (9)	0.0385 (8)	0.0446 (9)	0.0017 (7)	0.0194 (7)	−0.0004 (7)
C17	0.0463 (8)	0.0353 (8)	0.0442 (8)	−0.0008 (6)	0.0161 (7)	−0.0007 (6)
C18	0.0675 (11)	0.0511 (10)	0.0437 (9)	0.0124 (8)	0.0118 (8)	−0.0005 (8)
C19	0.0547 (9)	0.0357 (8)	0.0442 (8)	0.0010 (7)	0.0178 (7)	0.0005 (7)
N1	0.0523 (8)	0.0371 (7)	0.0393 (7)	−0.0003 (5)	0.0144 (6)	0.0014 (5)
N2	0.0638 (9)	0.0385 (7)	0.0431 (7)	0.0105 (6)	0.0157 (6)	0.0018 (6)
O1	0.0683 (8)	0.0447 (6)	0.0353 (6)	0.0153 (5)	0.0164 (5)	0.0064 (5)
O2	0.1034 (11)	0.0441 (7)	0.0466 (7)	0.0170 (7)	0.0294 (7)	0.0017 (6)
O3	0.1260 (14)	0.0805 (11)	0.0395 (7)	0.0390 (10)	0.0140 (8)	0.0028 (7)
S1	0.0694 (3)	0.0445 (3)	0.0435 (2)	0.01282 (19)	0.0146 (2)	−0.00351 (17)

Geometric parameters (Å, º)

C1—C2	1.506 (3)	C10—O1	1.3638 (19)
C1—H1A	0.9600	C10—C15	1.384 (2)
C1—H1B	0.9600	C10—C11	1.387 (2)
C1—H1C	0.9600	C11—C12	1.374 (2)
C2—C3	1.511 (2)	C11—H11	0.9300
C2—H2A	0.9700	C12—C13	1.398 (2)
C2—H2B	0.9700	C12—H12	0.9300
C3—C4	1.381 (2)	C13—C14	1.392 (2)
C3—C7	1.382 (2)	C13—C16	1.452 (2)
C4—C5	1.380 (2)	C14—C15	1.379 (2)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.384 (2)	C15—H15	0.9300
C5—H5	0.9300	C16—C17	1.340 (2)
C6—N1	1.337 (2)	C16—H16	0.9300
C6—C8	1.508 (2)	C17—C19	1.486 (2)
C7—N1	1.337 (2)	C17—S1	1.7488 (16)
C7—H7	0.9300	C18—O3	1.196 (2)

C8—C9	1.498 (2)	C18—N2	1.365 (2)
C8—H8A	0.9700	C18—S1	1.7897 (19)
C8—H8B	0.9700	C19—O2	1.213 (2)
C9—O1	1.4281 (19)	C19—N2	1.371 (2)
C9—H9A	0.9700	N2—H2	0.862 (10)
C9—H9B	0.9700		

C2—C1—H1A	109.5	H9A—C9—H9B	108.3
C2—C1—H1B	109.5	O1—C10—C15	124.86 (14)
H1A—C1—H1B	109.5	O1—C10—C11	115.53 (14)
C2—C1—H1C	109.5	C15—C10—C11	119.61 (15)
H1A—C1—H1C	109.5	C12—C11—C10	120.77 (15)
H1B—C1—H1C	109.5	C12—C11—H11	119.6
C1—C2—C3	113.09 (16)	C10—C11—H11	119.6
C1—C2—H2A	109.0	C11—C12—C13	120.81 (14)
C3—C2—H2A	109.0	C11—C12—H12	119.6
C1—C2—H2B	109.0	C13—C12—H12	119.6
C3—C2—H2B	109.0	C14—C13—C12	117.16 (15)
H2A—C2—H2B	107.8	C14—C13—C16	117.96 (15)
C4—C3—C7	116.41 (15)	C12—C13—C16	124.88 (15)
C4—C3—C2	122.65 (16)	C15—C14—C13	122.60 (16)
C7—C3—C2	120.92 (16)	C15—C14—H14	118.7
C5—C4—C3	120.41 (16)	C13—C14—H14	118.7
C5—C4—H4	119.8	C14—C15—C10	119.01 (15)
C3—C4—H4	119.8	C14—C15—H15	120.5
C4—C5—C6	119.07 (16)	C10—C15—H15	120.5
C4—C5—H5	120.5	C17—C16—C13	131.94 (16)
C6—C5—H5	120.5	C17—C16—H16	114.0
N1—C6—C5	121.44 (15)	C13—C16—H16	114.0
N1—C6—C8	116.24 (15)	C16—C17—C19	119.89 (15)
C5—C6—C8	122.30 (15)	C16—C17—S1	130.14 (13)
N1—C7—C3	124.28 (16)	C19—C17—S1	109.96 (11)
N1—C7—H7	117.9	O3—C18—N2	126.78 (18)
C3—C7—H7	117.9	O3—C18—S1	123.41 (15)
C9—C8—C6	110.28 (14)	N2—C18—S1	109.81 (13)
C9—C8—H8A	109.6	O2—C19—N2	123.91 (15)
C6—C8—H8A	109.6	O2—C19—C17	125.37 (15)
C9—C8—H8B	109.6	N2—C19—C17	110.72 (14)
C6—C8—H8B	109.6	C6—N1—C7	118.38 (14)
H8A—C8—H8B	108.1	C18—N2—C19	117.78 (14)
O1—C9—C8	108.90 (14)	C18—N2—H2	121.0 (16)
O1—C9—H9A	109.9	C19—N2—H2	121.2 (16)
C8—C9—H9A	109.9	C10—O1—C9	117.26 (13)
O1—C9—H9B	109.9	C17—S1—C18	91.72 (8)
C8—C9—H9B	109.9		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···S1	0.93	2.63	3.3166 (16)	131
C16—H16···O2	0.93	2.49	2.861 (2)	104
N2—H2···N1^{i}	0.86 (1)	2.00 (1)	2.8474 (19)	169 (2)
C7—H7···O2^{ii}	0.93	2.53	3.310 (2)	142

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