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Ethyl 2-[(5*Z*)-5-(4-methoxybenzylidene)-2,4-dioxo-1,3-thiazolidin-3-yl]acetate

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Keywords: crystal structure; thiazolidindione; C—H···O hydrogen bonds.

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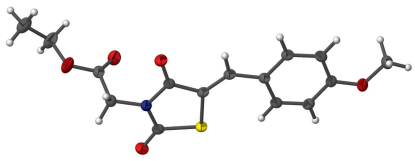
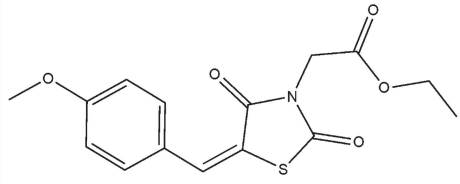
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{15}NO_5S$, the benzene and heterocyclic rings are close to being coplanar [dihedral angle = 1.49 (6)°]. In the crystal, pairwise C—H···O hydrogen bonds form dimers, which are arranged into 'stair-step' rows by way of C=O–π interactions between a carbonyl group and the benzene ring [O···π = 3.3837 (12) Å].

3D view



Chemical scheme



Structure description

As a continuation of our studies of the structures of thiazolidine-2,4-dione derivatives (Karrouchi *et al.*, 2016), we report on the N-alkylation of 5-(4 methoxybenzylidene)thiazolidine-2,4-dione with ethyl bromoacetate which gave title compound (Fig. 1) which was characterized by single-crystal X-ray diffraction.

The benzene ring and the heterocyclic ring (r.m.s. deviation = 0.018 Å) are close to being coplanar, the dihedral angle between their mean planes being 1.49 (6)°. In the crystal, the molecules form dimers *via* pairwise C8—H8···O2^{i} [symmetry code: (i) $-x$, $-y+1$, $-z+1$] hydrogen bonds (Table 1 and Fig. 2). These units are formed into 'stair-step' rows through C=O–π interactions between the C10=O2 carbonyl group and the benzene ring at ($\mp x$, y, z) [O···π = 3.370 (1) Å and C=O···π = 83.56 (8)°] (Fig. 3).

Synthesis and crystallization

To a solution of 5-(4-methoxybenzylidene)thiazolidine-2,4-dione (1 mmol) in acetone (30 ml) an excess of triethylamine (1.5 mmol) was added and the mixture was stirred for

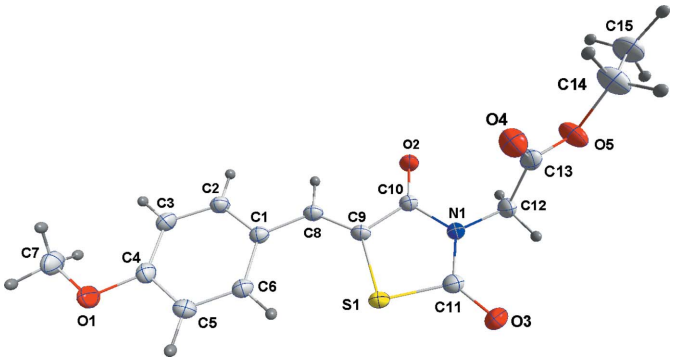


Figure 1
The title molecule, showing the atom-labelling scheme and 50% probability displacement ellipsoids.

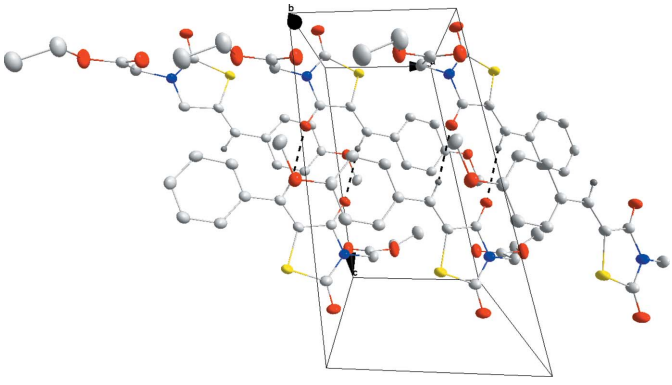


Figure 2
The packing, viewed along the *b* axis, with complementary C—H···O hydrogen bonds shown as dotted lines.

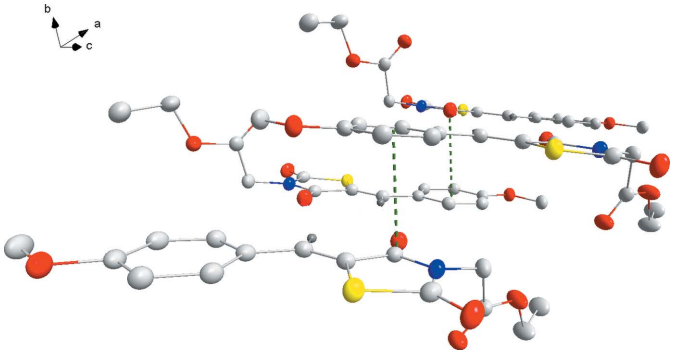


Figure 3
Detail of the complementary C═O–π stacking between one carbonyl group and the benzene ring in an adjacent molecule.

10 min at 25°C. Ethyl bromoacetate (1.5 mmol) was added slowly and the reaction mixture was refluxed for 10 h. The progress was monitored by TLC and, when complete, the reaction mixture was cooled to room temperature, filtered and the volatiles removed under reduced pressure. The residue was dissolved in ethanol, concentrated and cooled to afford crystals which were filtered off, washed with ethanol and recrystallized from ethanol solution. Yield 84%; m.p. 130–132°C.

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.95	2.42	3.3157 (15)	156

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{15}NO_5S$
M_r	321.34
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	6.1957 (2), 10.6460 (3), 12.2909 (3)
α, β, γ (°)	68.845 (1), 78.971 (1), 84.919 (1)
V (Å^3)	741.93 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.16
Crystal size (mm)	0.21 × 0.11 × 0.04
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.80, 0.92
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10428, 2884, 2690
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.079, 1.05
No. of reflections	2884
No. of parameters	202
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.23

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Refinement

Details of crystal data and refinement are presented in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161041 [https://doi.org/10.1107/S2414314616010415]

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Crystal data

$C_{15}H_{15}NO_5S$
M_r = 321.34
Triclinic, $P\overline{1}$
a = 6.1957 (2) Å
b = 10.6460 (3) Å
c = 12.2909 (3) Å
α = 68.845 (1)°
β = 78.971 (1)°
γ = 84.919 (1)°
V = 741.93 (4) Å^3
Z = 2
$F(000)$ = 336
D_x = 1.438 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 8632 reflections
θ = 3.9–72.0°
μ = 2.16 mm^{-1}
T = 150 K
Plate, colourless
0.21 × 0.11 × 0.04 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, 2016)
T_{min} = 0.80, T_{max} = 0.92
10428 measured reflections
2884 independent reflections
2690 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 72.1°, θ_{min} = 3.9°
h = −7→7
k = −12→13
l = −12→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.079
S = 1.05
2884 reflections
202 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
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.2407P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0071 (7)

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 - 0.99 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.34156 (5)	0.50355 (3)	0.12596 (3)	0.02452 (11)
O1	1.10838 (15)	0.14727 (10)	0.45592 (8)	0.0287 (2)
O2	−0.08424 (15)	0.60296 (10)	0.35429 (8)	0.0266 (2)
O3	0.09830 (17)	0.64516 (10)	−0.03482 (8)	0.0320 (2)
O4	−0.01087 (16)	0.89843 (10)	0.13494 (10)	0.0346 (2)
O5	−0.37526 (16)	0.92434 (10)	0.13243 (11)	0.0374 (3)
N1	−0.02628 (17)	0.63167 (11)	0.15778 (9)	0.0230 (2)
C1	0.5290 (2)	0.36877 (12)	0.38833 (11)	0.0214 (3)
C2	0.5793 (2)	0.32201 (13)	0.50296 (11)	0.0237 (3)
H2	0.4799	0.3421	0.5640	0.028*
C3	0.7695 (2)	0.24723 (13)	0.53029 (11)	0.0249 (3)
H3	0.7995	0.2163	0.6088	0.030*
C4	0.9158 (2)	0.21833 (13)	0.44081 (12)	0.0234 (3)
C5	0.8693 (2)	0.26387 (13)	0.32566 (12)	0.0247 (3)
H5	0.9693	0.2440	0.2648	0.030*
C6	0.6797 (2)	0.33731 (13)	0.30007 (11)	0.0234 (3)
H6	0.6499	0.3673	0.2215	0.028*
C7	1.1601 (2)	0.09656 (15)	0.57301 (13)	0.0314 (3)
H7A	1.3016	0.0475	0.5725	0.047*
H7B	1.0448	0.0356	0.6268	0.047*
H7C	1.1691	0.1719	0.5998	0.047*
C8	0.3265 (2)	0.44693 (12)	0.36890 (11)	0.0215 (3)
H8	0.2416	0.4593	0.4375	0.026*
C9	0.2398 (2)	0.50466 (12)	0.26949 (11)	0.0211 (3)
C10	0.0297 (2)	0.58171 (12)	0.27051 (11)	0.0211 (3)
C11	0.1182 (2)	0.60538 (13)	0.06821 (11)	0.0239 (3)
C12	−0.2200 (2)	0.71737 (13)	0.13586 (12)	0.0256 (3)
H12A	−0.3461	0.6765	0.1984	0.031*
H12B	−0.2561	0.7244	0.0588	0.031*
C13	−0.1837 (2)	0.85685 (13)	0.13435 (11)	0.0251 (3)
C14	−0.3745 (3)	1.06523 (15)	0.12095 (16)	0.0402 (4)
H14A	−0.3447	1.1233	0.0363	0.048*
H14B	−0.2590	1.0798	0.1605	0.048*
C15	−0.5960 (3)	1.09868 (16)	0.17802 (16)	0.0425 (4)

H15A	−0.6077	1.1957	0.1632	0.064*
H15B	−0.6160	1.0490	0.2635	0.064*
H15C	−0.7098	1.0734	0.1448	0.064*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.02626 (17)	0.02999 (18)	0.01769 (17)	0.00376 (12)	−0.00117 (11)	−0.01115 (12)
O1	0.0259 (5)	0.0305 (5)	0.0301 (5)	0.0060 (4)	−0.0074 (4)	−0.0111 (4)
O2	0.0257 (5)	0.0333 (5)	0.0208 (5)	0.0036 (4)	0.0003 (3)	−0.0125 (4)
O3	0.0410 (6)	0.0357 (5)	0.0186 (5)	0.0038 (4)	−0.0062 (4)	−0.0094 (4)
O4	0.0270 (5)	0.0307 (5)	0.0455 (6)	−0.0021 (4)	−0.0066 (4)	−0.0124 (4)
O5	0.0257 (5)	0.0259 (5)	0.0628 (7)	0.0040 (4)	−0.0082 (5)	−0.0190 (5)
N1	0.0247 (5)	0.0243 (5)	0.0201 (5)	0.0033 (4)	−0.0044 (4)	−0.0087 (4)
C1	0.0219 (6)	0.0219 (6)	0.0206 (6)	−0.0025 (5)	−0.0019 (5)	−0.0083 (5)
C2	0.0242 (6)	0.0266 (6)	0.0208 (6)	−0.0013 (5)	−0.0011 (5)	−0.0102 (5)
C3	0.0278 (6)	0.0263 (6)	0.0212 (6)	−0.0021 (5)	−0.0056 (5)	−0.0079 (5)
C4	0.0216 (6)	0.0212 (6)	0.0278 (7)	−0.0006 (5)	−0.0048 (5)	−0.0086 (5)
C5	0.0247 (6)	0.0257 (6)	0.0234 (6)	0.0005 (5)	0.0000 (5)	−0.0107 (5)
C6	0.0253 (6)	0.0250 (6)	0.0199 (6)	−0.0012 (5)	−0.0026 (5)	−0.0083 (5)
C7	0.0311 (7)	0.0308 (7)	0.0332 (7)	0.0034 (5)	−0.0123 (6)	−0.0099 (6)
C8	0.0215 (6)	0.0236 (6)	0.0198 (6)	−0.0023 (5)	0.0007 (5)	−0.0095 (5)
C9	0.0213 (6)	0.0228 (6)	0.0193 (6)	−0.0014 (5)	0.0010 (4)	−0.0095 (5)
C10	0.0228 (6)	0.0211 (6)	0.0196 (6)	−0.0019 (5)	−0.0023 (4)	−0.0078 (5)
C11	0.0293 (6)	0.0226 (6)	0.0202 (6)	−0.0023 (5)	−0.0022 (5)	−0.0086 (5)
C12	0.0241 (6)	0.0267 (7)	0.0276 (7)	0.0034 (5)	−0.0075 (5)	−0.0108 (5)
C13	0.0249 (6)	0.0263 (6)	0.0220 (6)	0.0013 (5)	−0.0023 (5)	−0.0074 (5)
C14	0.0355 (8)	0.0243 (7)	0.0595 (10)	0.0015 (6)	−0.0029 (7)	−0.0162 (7)
C15	0.0392 (8)	0.0338 (8)	0.0548 (10)	0.0057 (6)	−0.0022 (7)	−0.0204 (7)

Geometric parameters (Å, º)

S1—C9	1.7602 (12)	C4—C5	1.3989 (18)
S1—C11	1.7745 (13)	C5—C6	1.3755 (18)
O1—C4	1.3617 (15)	C5—H5	0.9500
O1—C7	1.4318 (16)	C6—H6	0.9500
O2—C10	1.2131 (15)	C7—H7A	0.9800
O3—C11	1.2089 (16)	C7—H7B	0.9800
O4—C13	1.1966 (17)	C7—H7C	0.9800
O5—C13	1.3315 (16)	C8—C9	1.3453 (18)
O5—C14	1.4553 (17)	C8—H8	0.9500
N1—C11	1.3757 (16)	C9—C10	1.4763 (17)
N1—C10	1.3937 (16)	C12—C13	1.5144 (18)
N1—C12	1.4482 (15)	C12—H12A	0.9900
C1—C2	1.4014 (18)	C12—H12B	0.9900
C1—C6	1.4070 (17)	C14—C15	1.493 (2)
C1—C8	1.4523 (17)	C14—H14A	0.9900
C2—C3	1.3888 (18)	C14—H14B	0.9900

C2—H2	0.9500	C15—H15A	0.9800
C3—C4	1.3924 (18)	C15—H15B	0.9800
C3—H3	0.9500	C15—H15C	0.9800

C9—S1—C11	92.26 (6)	C1—C8—H8	114.8
C4—O1—C7	117.16 (10)	C8—C9—C10	120.98 (11)
C13—O5—C14	117.70 (11)	C8—C9—S1	129.07 (10)
C11—N1—C10	117.36 (10)	C10—C9—S1	109.95 (9)
C11—N1—C12	122.13 (11)	O2—C10—N1	122.09 (11)
C10—N1—C12	120.30 (10)	O2—C10—C9	127.40 (11)
C2—C1—C6	117.48 (11)	N1—C10—C9	110.50 (10)
C2—C1—C8	117.80 (11)	O3—C11—N1	125.70 (12)
C6—C1—C8	124.71 (11)	O3—C11—S1	124.43 (10)
C3—C2—C1	122.14 (12)	N1—C11—S1	109.87 (9)
C3—C2—H2	118.9	N1—C12—C13	111.19 (10)
C1—C2—H2	118.9	N1—C12—H12A	109.4
C2—C3—C4	118.96 (12)	C13—C12—H12A	109.4
C2—C3—H3	120.5	N1—C12—H12B	109.4
C4—C3—H3	120.5	C13—C12—H12B	109.4
O1—C4—C3	124.62 (12)	H12A—C12—H12B	108.0
O1—C4—C5	115.40 (11)	O4—C13—O5	126.15 (13)
C3—C4—C5	119.98 (12)	O4—C13—C12	125.21 (12)
C6—C5—C4	120.38 (11)	O5—C13—C12	108.64 (11)
C6—C5—H5	119.8	O5—C14—C15	107.30 (12)
C4—C5—H5	119.8	O5—C14—H14A	110.3
C5—C6—C1	121.05 (12)	C15—C14—H14A	110.3
C5—C6—H6	119.5	O5—C14—H14B	110.3
C1—C6—H6	119.5	C15—C14—H14B	110.3
O1—C7—H7A	109.5	H14A—C14—H14B	108.5
O1—C7—H7B	109.5	C14—C15—H15A	109.5
H7A—C7—H7B	109.5	C14—C15—H15B	109.5
O1—C7—H7C	109.5	H15A—C15—H15B	109.5
H7A—C7—H7C	109.5	C14—C15—H15C	109.5
H7B—C7—H7C	109.5	H15A—C15—H15C	109.5
C9—C8—C1	130.43 (11)	H15B—C15—H15C	109.5
C9—C8—H8	114.8		

C6—C1—C2—C3	−0.04 (19)	C11—N1—C10—C9	−1.53 (15)
C8—C1—C2—C3	−179.58 (11)	C12—N1—C10—C9	−176.34 (10)
C1—C2—C3—C4	0.3 (2)	C8—C9—C10—O2	0.6 (2)
C7—O1—C4—C3	1.50 (19)	S1—C9—C10—O2	−179.72 (11)
C7—O1—C4—C5	−178.81 (11)	C8—C9—C10—N1	179.97 (11)
C2—C3—C4—O1	179.34 (12)	S1—C9—C10—N1	−0.33 (13)
C2—C3—C4—C5	−0.33 (19)	C10—N1—C11—O3	−177.26 (12)
O1—C4—C5—C6	−179.66 (11)	C12—N1—C11—O3	−2.6 (2)
C3—C4—C5—C6	0.0 (2)	C10—N1—C11—S1	2.64 (14)
C4—C5—C6—C1	0.3 (2)	C12—N1—C11—S1	177.35 (9)
C2—C1—C6—C5	−0.26 (19)	C9—S1—C11—O3	177.61 (12)

C8—C1—C6—C5	179.24 (12)	C9—S1—C11—N1	−2.30 (10)
C2—C1—C8—C9	179.20 (13)	C11—N1—C12—C13	−101.22 (14)
C6—C1—C8—C9	−0.3 (2)	C10—N1—C12—C13	73.34 (15)
C1—C8—C9—C10	−179.34 (12)	C14—O5—C13—O4	5.0 (2)
C1—C8—C9—S1	1.0 (2)	C14—O5—C13—C12	−175.08 (13)
C11—S1—C9—C8	−178.85 (13)	N1—C12—C13—O4	9.47 (19)
C11—S1—C9—C10	1.48 (9)	N1—C12—C13—O5	−170.46 (11)
C11—N1—C10—O2	177.90 (12)	C13—O5—C14—C15	−152.82 (14)
C12—N1—C10—O2	3.09 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.95	2.42	3.3157 (15)	156

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