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N-(3-Butyl-4-oxo-1,3-thiazolidin-2-ylidene)benzamide

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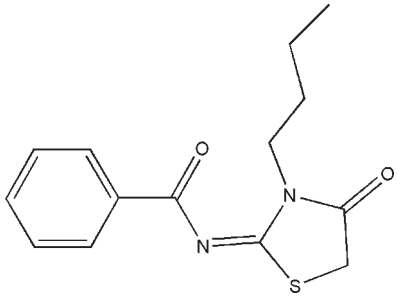
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.044; wR factor = 0.129; data-to-parameter ratio = 14.5.

In the title compound, $C_{14}H_{16}N_2O_2S$, the thiazolidine ring is planar [maximum atomic deviation = 0.0080 (14) Å] and twisted slightly with respect to the phenyl ring, making a dihedral angle of 4.46 (14)°. The butyl group displays an extended conformation, with a torsion angle of 169.4 (4)°. In the crystal structure, weak intermolecular C—H···O hydrogen bonds link the molecules, forming supramolecular chains.

Related literature

For the pharmaceutical applications of thiazolidinones, see: Amin *et al.* (2008); Ramla *et al.* (2007). For the synthesis, see: Peng *et al.* (2004).



Experimental

Crystal data

$C_{14}H_{16}N_2O_2S$
M_r = 276.35
Monoclinic, $P2_1/n$
a = 5.4690 (1) Å
b = 30.5591 (8) Å
c = 8.6032 (2) Å
β = 99.895 (3)°
V = 1416.44 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 K
0.50 × 0.14 × 0.07 mm

Data collection

Oxford Diffraction Nova A diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2008)
T_{min} = 0.895, T_{max} = 0.984
7446 measured reflections
2530 independent reflections
1987 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.06
2530 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O2^{i}	0.93	2.39	3.309 (3)	172

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2008); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *OLEX2* (Dolomanov *et al.*, 2009).; software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2789).

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supporting information

Acta Cryst. (2010). E**66**, o1803 [doi:10.1107/S1600536810020556]

N-(3-Butyl-4-oxo-1,3-thiazolidin-2-ylidene)benzamide

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S1. Comment

Thiazolidinones have wide applications as anticonvulsant (Amin *et al.*, 2008) and anti-neoplastic drugs (Ramla *et al.*, 2007). We report here the structure of a new thiazolidinone derivative, I, Fig. 1.

The thiazolidinyl ring and phenyl ring are almost co-planar with the dihedral angle of 4.46 (14)°. The C11—C12—C13—C14 torsion angle is 169.4 (4)°, showing an extended conformation for the butyl substituent. The N1═C8 bond distance of 1.291 (3) Å indicates a typical double bond. In the crystal structure, weak intermolecular C—H···O hydrogen bonds, Table 1, link the molecules to form one-dimensional supra-molecular chains, Fig. 2.

S2. Experimental

The title compound was prepared according to the procedure reported by Peng *et al.* (2004). A 50 ml flask equipped with a dropping funnel was charged with NH_4SCN (0.152 g, 2 mmol) and [bmim][PF_6] (2 ml) and was cooled in an ice-water bath. Freshly distilled benzoyl chloride(0.284 g, 2 mmol) was added dropwise and stirred for a further 20 min (disappearance of the starting material was monitored by TLC). n-Butylamine (2 mmol) was then added to the same reaction vessel at room temperature and the mixture was stirred for 20 min more. On completion, ethyl chloroacetate (2.4 mmol) and anhydrous sodium acetate (0.196 g, 2.4 mmol) was added to the flask, and the mixture was heated at 80°C for 2-3 h. After consumption of *N*-benzoyl-*N*′-butylthiourea as indicated by by TLC monitoring, the salts were firstly leached with water (5 ml×2), and the crude product was collected by filtration. Recrystallization from ethanol gave pure product as a yellow crystalline solid.

S3. Refinement

H atoms were placed in calculated positions with C—H = 0.93 (aromatic), 0.96 (methyl) and 0.97 Å (methine). The torsion angle of methyl group was refined to fit the electron density, with U_{iso}(H) = 1.5U_{ea}(C). For the other H atoms, U_{iso}(H) = 1.2U_{eq}(C).

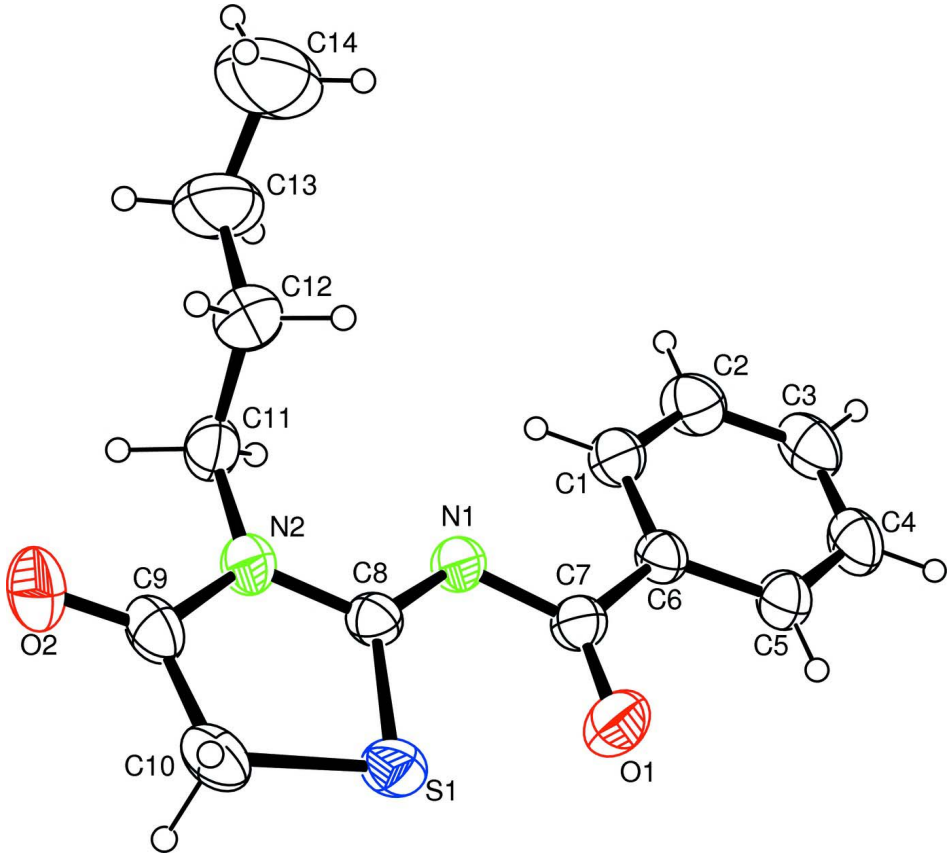

Figure 1

The molecular structure of the title compound with 40% probability displacement ellipsoids.

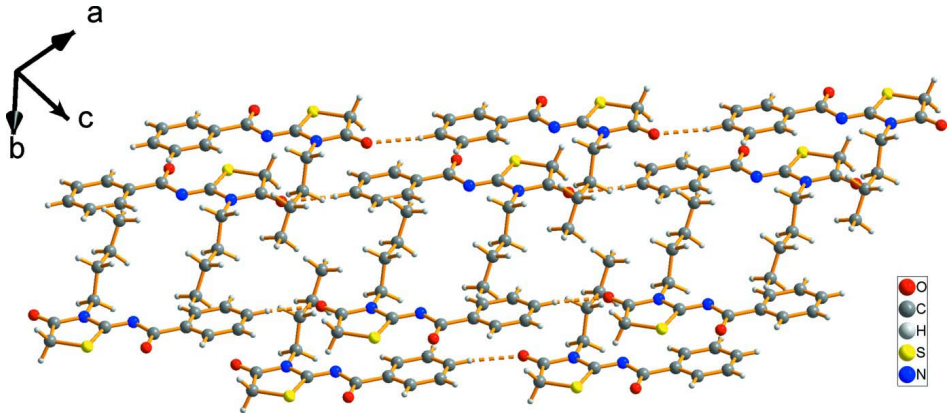

Figure 2

Crystal packing for I viewed down the *a* axis.

N-(3-Butyl-4-oxo-1,3-thiazolidin-2-ylidene)benzamide

Crystal data

$C_{14}H_{16}N_2O_2S$
$M_r = 276.35$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 5.4690\ (1)$ Å
$b = 30.5591\ (8)$ Å

c = 8.6032 (2) Å
β = 99.895 (3)°
V = 1416.44 (6) Å^3
Z = 4
F(000) = 584
D_x = 1.296 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 3174 reflections
θ = 2.8–25.0°
μ = 0.23 mm^{-1}
T = 294 K
Platelet, yellow
0.50 × 0.14 × 0.07 mm

Data collection

Oxford Diffraction Nova A diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2008)
T_{min} = 0.895, T_{max} = 0.984

7446 measured reflections
2530 independent reflections
1987 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.1°, θ_{min} = 1.3°
h = −6→3
k = −36→36
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.129
S = 1.06
2530 reflections
174 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
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.3856P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0113 (18)

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 $F^2 > \sigma(F^2)$ 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}
S1	0.28923 (11)	0.210301 (19)	0.34733 (8)	0.0680 (2)
N1	0.0399 (3)	0.14912 (6)	0.1466 (2)	0.0568 (5)
N2	0.4097 (3)	0.12988 (6)	0.3021 (2)	0.0604 (5)
O1	−0.1307 (3)	0.21815 (6)	0.1475 (2)	0.0846 (6)
O2	0.7761 (3)	0.11970 (7)	0.4680 (2)	0.0903 (6)
C1	−0.3441 (4)	0.12483 (8)	−0.0946 (3)	0.0675 (6)
H1	−0.2120	0.1058	−0.0637	0.081*
C2	−0.5428 (5)	0.11225 (10)	−0.2086 (3)	0.0834 (8)

H2	−0.5435	0.0846	−0.2540	0.100*
C3	−0.7382 (5)	0.14015 (11)	−0.2550 (3)	0.0850 (8)
H3	−0.8711	0.1313	−0.3310	0.102*
C4	−0.7377 (4)	0.18069 (11)	−0.1902 (3)	0.0798 (8)
H4	−0.8697	0.1996	−0.2228	0.096*
C5	−0.5429 (4)	0.19391 (8)	−0.0765 (3)	0.0674 (6)
H5	−0.5441	0.2217	−0.0322	0.081*
C6	−0.3447 (4)	0.16583 (7)	−0.0278 (2)	0.0555 (5)
C7	−0.1369 (4)	0.18080 (7)	0.0965 (3)	0.0588 (5)
C8	0.2273 (4)	0.16005 (7)	0.2522 (2)	0.0543 (5)
C9	0.6056 (4)	0.14333 (9)	0.4145 (3)	0.0669 (6)
C10	0.5774 (4)	0.19006 (9)	0.4582 (3)	0.0740 (7)
H10A	0.7154	0.2071	0.4338	0.089*
H10B	0.5756	0.1925	0.5704	0.089*
C11	0.4005 (4)	0.08576 (8)	0.2365 (3)	0.0693 (6)
H11A	0.5683	0.0746	0.2452	0.083*
H11B	0.3289	0.0870	0.1253	0.083*
C12	0.2517 (5)	0.05489 (9)	0.3177 (4)	0.0900 (8)
H12A	0.0871	0.0669	0.3160	0.108*
H12B	0.3303	0.0516	0.4271	0.108*
C13	0.2296 (8)	0.01017 (11)	0.2380 (6)	0.1436 (17)
H13A	0.1831	0.0146	0.1251	0.172*
H13B	0.3921	−0.0035	0.2564	0.172*
C14	0.0574 (10)	−0.01942 (15)	0.2872 (8)	0.194 (3)
H14A	0.0878	−0.0214	0.4002	0.291*
H14B	0.0767	−0.0478	0.2430	0.291*
H14C	−0.1084	−0.0091	0.2515	0.291*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0674 (4)	0.0599 (4)	0.0716 (4)	−0.0109 (3)	−0.0022 (3)	−0.0083 (3)
N1	0.0544 (10)	0.0541 (10)	0.0564 (10)	−0.0011 (8)	−0.0061 (8)	0.0013 (8)
N2	0.0529 (10)	0.0640 (11)	0.0592 (10)	0.0002 (8)	−0.0045 (8)	0.0004 (9)
O1	0.0866 (12)	0.0620 (10)	0.0931 (13)	0.0110 (8)	−0.0190 (10)	−0.0116 (9)
O2	0.0606 (10)	0.1150 (15)	0.0852 (12)	0.0086 (10)	−0.0155 (9)	0.0012 (11)
C1	0.0633 (13)	0.0679 (14)	0.0656 (14)	−0.0014 (11)	−0.0054 (11)	−0.0005 (11)
C2	0.0786 (17)	0.0852 (18)	0.0781 (17)	−0.0140 (14)	−0.0104 (13)	−0.0076 (14)
C3	0.0620 (15)	0.115 (2)	0.0689 (16)	−0.0187 (15)	−0.0132 (12)	0.0112 (16)
C4	0.0534 (13)	0.102 (2)	0.0792 (17)	0.0026 (13)	−0.0040 (12)	0.0219 (16)
C5	0.0577 (13)	0.0705 (14)	0.0714 (14)	0.0035 (11)	0.0035 (11)	0.0101 (12)
C6	0.0495 (11)	0.0596 (12)	0.0548 (11)	−0.0026 (9)	0.0021 (9)	0.0075 (10)
C7	0.0584 (12)	0.0539 (12)	0.0606 (12)	0.0001 (10)	0.0001 (10)	0.0009 (10)
C8	0.0534 (11)	0.0540 (12)	0.0531 (11)	−0.0059 (9)	0.0027 (9)	0.0013 (9)
C9	0.0506 (12)	0.0865 (17)	0.0590 (13)	−0.0085 (11)	−0.0035 (10)	0.0037 (12)
C10	0.0615 (14)	0.0884 (18)	0.0669 (14)	−0.0221 (12)	−0.0039 (11)	−0.0040 (13)
C11	0.0608 (13)	0.0674 (14)	0.0751 (15)	0.0091 (11)	−0.0013 (11)	−0.0030 (12)
C12	0.0860 (19)	0.0689 (16)	0.114 (2)	0.0021 (14)	0.0156 (17)	−0.0020 (16)

C13	0.144 (3)	0.0650 (19)	0.234 (5)	−0.012 (2)	0.068 (3)	−0.017 (3)
C14	0.179 (5)	0.108 (3)	0.313 (8)	−0.038 (3)	0.093 (5)	−0.040 (4)

Geometric parameters (Å, º)

S1—C8	1.746 (2)	C5—C6	1.390 (3)
S1—C10	1.805 (2)	C5—H5	0.9300
N1—C8	1.291 (3)	C6—C7	1.492 (3)
N1—C7	1.384 (3)	C9—C10	1.491 (4)
N2—C8	1.372 (3)	C10—H10A	0.9700
N2—C9	1.377 (3)	C10—H10B	0.9700
N2—C11	1.459 (3)	C11—C12	1.495 (4)
O1—C7	1.221 (3)	C11—H11A	0.9700
O2—C9	1.206 (3)	C11—H11B	0.9700
C1—C6	1.379 (3)	C12—C13	1.525 (5)
C1—C2	1.387 (3)	C12—H12A	0.9700
C1—H1	0.9300	C12—H12B	0.9700
C2—C3	1.372 (4)	C13—C14	1.421 (5)
C2—H2	0.9300	C13—H13A	0.9700
C3—C4	1.359 (4)	C13—H13B	0.9700
C3—H3	0.9300	C14—H14A	0.9600
C4—C5	1.377 (3)	C14—H14B	0.9600
C4—H4	0.9300	C14—H14C	0.9600
C8—S1—C10	91.59 (11)	N2—C9—C10	111.2 (2)
C8—N1—C7	117.76 (19)	C9—C10—S1	108.30 (15)
C8—N2—C9	117.08 (19)	C9—C10—H10A	110.0
C8—N2—C11	121.66 (17)	S1—C10—H10A	110.0
C9—N2—C11	121.24 (19)	C9—C10—H10B	110.0
C6—C1—C2	119.3 (2)	S1—C10—H10B	110.0
C6—C1—H1	120.4	H10A—C10—H10B	108.4
C2—C1—H1	120.4	N2—C11—C12	112.8 (2)
C3—C2—C1	120.7 (3)	N2—C11—H11A	109.0
C3—C2—H2	119.7	C12—C11—H11A	109.0
C1—C2—H2	119.7	N2—C11—H11B	109.0
C4—C3—C2	120.0 (2)	C12—C11—H11B	109.0
C4—C3—H3	120.0	H11A—C11—H11B	107.8
C2—C3—H3	120.0	C11—C12—C13	111.3 (3)
C3—C4—C5	120.4 (2)	C11—C12—H12A	109.4
C3—C4—H4	119.8	C13—C12—H12A	109.4
C5—C4—H4	119.8	C11—C12—H12B	109.4
C4—C5—C6	120.1 (2)	C13—C12—H12B	109.4
C4—C5—H5	120.0	H12A—C12—H12B	108.0
C6—C5—H5	120.0	C14—C13—C12	116.2 (4)
C1—C6—C5	119.5 (2)	C14—C13—H13A	108.2
C1—C6—C7	121.44 (19)	C12—C13—H13A	108.2
C5—C6—C7	119.0 (2)	C14—C13—H13B	108.2
O1—C7—N1	124.6 (2)	C12—C13—H13B	108.2

O1—C7—C6	121.0 (2)	H13A—C13—H13B	107.4
N1—C7—C6	114.46 (19)	C13—C14—H14A	109.5
N1—C8—N2	119.52 (19)	C13—C14—H14B	109.5
N1—C8—S1	128.62 (17)	H14A—C14—H14B	109.5
N2—C8—S1	111.86 (14)	C13—C14—H14C	109.5
O2—C9—N2	123.1 (2)	H14A—C14—H14C	109.5
O2—C9—C10	125.7 (2)	H14B—C14—H14C	109.5
C6—C1—C2—C3	0.1 (4)	C11—N2—C8—N1	1.3 (3)
C1—C2—C3—C4	0.5 (4)	C9—N2—C8—S1	−0.2 (3)
C2—C3—C4—C5	−0.7 (4)	C11—N2—C8—S1	−178.67 (17)
C3—C4—C5—C6	0.3 (4)	C10—S1—C8—N1	−179.2 (2)
C2—C1—C6—C5	−0.5 (4)	C10—S1—C8—N2	0.77 (18)
C2—C1—C6—C7	179.3 (2)	C8—N2—C9—O2	179.6 (2)
C4—C5—C6—C1	0.3 (4)	C11—N2—C9—O2	−1.9 (4)
C4—C5—C6—C7	−179.5 (2)	C8—N2—C9—C10	−0.7 (3)
C8—N1—C7—O1	−1.5 (4)	C11—N2—C9—C10	177.8 (2)
C8—N1—C7—C6	178.87 (18)	O2—C9—C10—S1	−179.1 (2)
C1—C6—C7—O1	175.0 (2)	N2—C9—C10—S1	1.2 (3)
C5—C6—C7—O1	−5.1 (3)	C8—S1—C10—C9	−1.12 (19)
C1—C6—C7—N1	−5.3 (3)	C8—N2—C11—C12	−85.8 (3)
C5—C6—C7—N1	174.51 (19)	C9—N2—C11—C12	95.8 (3)
C7—N1—C8—N2	−178.42 (19)	N2—C11—C12—C13	175.6 (3)
C7—N1—C8—S1	1.5 (3)	C11—C12—C13—C14	−169.4 (4)
C9—N2—C8—N1	179.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.39	3.309 (3)	172

Symmetry code: (i) *x*−2, *y*, *z*−1.