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(Benzoyl)[2-(cyanoimino)-1,3-thiazolidin-2-yl]methyl acetate

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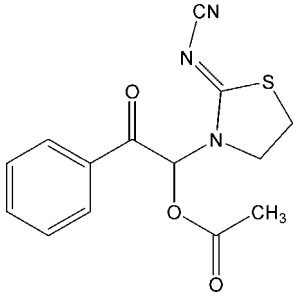
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.119; data-to-parameter ratio = 13.7.

The title compound, $C_{14}H_{13}N_3O_3S$, has been synthesized as a potent fungicidal agent. The 1,3-thiazolidine ring is approximately planar and makes a dihedral angle of 84.9 (2)° with the phenyl ring. There are weak intramolecular C—H···O and C—H···N hydrogen bonds, which stabilize the molecular structure.

Related literature

For related literature, see: Brackmann *et al.* (2005); Ezer *et al.* (1984); Ford & Casida (2006); Liu *et al.* (2006); Mota-Sanchez *et al.* (2006); Ogawa *et al.* (1992); Schmuck (2001); Shiokawa *et al.* (1990); Yoneda *et al.* (2001); Zhang *et al.* (2000).



Experimental

Crystal data

$C_{14}H_{13}N_3O_3S$
M_r = 303.33
Monoclinic, $P2_1/n$
a = 8.4679 (19) Å
b = 8.713 (2) Å
c = 20.147 (5) Å
β = 91.565 (4)°
V = 1486.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 294 (2) K
0.22 × 0.18 × 0.12 mm

Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.963
7441 measured reflections
2620 independent reflections
1807 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.119
S = 1.00
2620 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O3	0.98	2.22	2.649 (4)	105
C8—H8···N2	0.98	2.38	2.796 (3)	105

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); 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: IS2211).

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supplementary materials

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(Benzoyl)[2-(cyanoimino)-1,3-thiazolidin-2-yl]methyl acetate

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Comment

Recently, compounds containing 2-cyanoimino-thiazolidine group have attracted much interest because they exhibit diverse biological activities, such as antiulcer, vasodilator, antihypertensive and insecticidal activities (Ezer *et al.*, 1984; Shiokawa *et al.*, 1990; Ogawa *et al.*, 1992; Zhang *et al.*, 2000). They are widely applied in the fields of medication and plant protection (Yoneda *et al.*, 2001; Schmuck, 2001; Ford & Casida, 2006). For example, the chloronicotinyl insecticide Thiacloprid has high insecticidal activity with a favorable ecobiological profile and safety to bees, it is very useful in horticulture as well as in modern crop protection systems (Brackmann *et al.*, 2005; Mota-Sanchez *et al.*, 2006). In a search for more biologically active 2-cyanoimino-thiazolidine derivatives, the title compound was synthesized and its crystal structure was determined (Fig. 1). The molecule is non-planar, the benzene ring and the 1,3-thiazolidine ring making a dihedral of 84.9 (2) °.

Experimental

[3-(2-Oxo-2-phenylethyl)thiazolidin-2-ylideneamino]formonitrile (Liu *et al.*, 2006; 5 mmol) was dissolved in acetic acid (20 ml), and sodium acetate (6 mmol) was added. Then bromine (6 mmol) was dropwise added with stirring at 343 K, the reaction was maintained for about 3 h, until the mixture was turned into light yellow. Then water (50 ml) and chloroform (40 ml) were added. The organic layer was washed with saturated brine (3 × 30 ml), the combined organic layer was dried over anhydrous Na_2SO_4. After removal of the solvent, the residue was separated by column chromatography on silica gel, with petroleum ether/ethyl acetate (2:1 *v*/*v*) as eluent, and recrystallized from ethyl acetate to give a colorless crystal (yield 65%).

Refinement

H atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and were refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

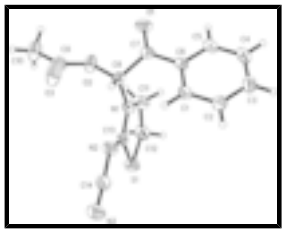

Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

(Benzoyl)[2-(cyanoimino)-1,3-thiazolidin-2-yl]methyl acetate

Crystal data

$C_{14}H_{13}N_3O_3S$ $F_{000} = 632$

M_r = 303.33	D_x = 1.356 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2292 reflections
a = 8.4679 (19) Å	θ = 2.6–25.4º
b = 8.713 (2) Å	μ = 0.23 mm^{-1}
c = 20.147 (5) Å	T = 294 (2) K
β = 91.565 (4)º	Monoclinic, colorless
V = 1486.0 (6) Å^3	0.22 × 0.18 × 0.12 mm
Z = 4	

Data collection

Bruker SMART 1000 diffractometer	2620 independent reflections
Radiation source: fine-focus sealed tube	1807 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.032
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	h = −9→10
T_{min} = 0.951, T_{max} = 0.963	k = −10→10
7441 measured reflections	l = −23→20

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.119	$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.8973P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{max}$ = 0.001
2620 reflections	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
191 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 calculat-

ing *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	0.48762 (9)	0.77682 (9)	0.05323 (4)	0.0621 (3)
O1	0.0277 (2)	1.0382 (2)	0.23069 (9)	0.0649 (6)
O3	0.0994 (4)	1.2840 (3)	0.05419 (12)	0.1099 (10)
N1	0.2699 (2)	0.9246 (2)	0.11271 (9)	0.0397 (5)
N2	0.2269 (3)	0.9170 (3)	−0.00064 (10)	0.0523 (6)
N3	0.3142 (4)	0.8453 (4)	−0.11270 (13)	0.0963 (10)
C1	−0.0593 (3)	0.7490 (3)	0.10658 (12)	0.0450 (6)
H1	−0.0287	0.8022	0.0690	0.054*
C2	−0.1267 (3)	0.6056 (3)	0.10017 (14)	0.0545 (7)
H2	−0.1413	0.5623	0.0583	0.065*
C3	−0.1727 (3)	0.5259 (3)	0.15584 (15)	0.0590 (7)
H3	−0.2163	0.4283	0.1515	0.071*
C4	−0.1537 (3)	0.5914 (3)	0.21766 (14)	0.0558 (7)
H4	−0.1869	0.5388	0.2550	0.067*
C5	−0.0863 (3)	0.7335 (3)	0.22468 (12)	0.0476 (6)
H5	−0.0732	0.7765	0.2667	0.057*
C6	−0.0370 (3)	0.8143 (3)	0.16918 (11)	0.0383 (6)
C7	0.0381 (3)	0.9666 (3)	0.17954 (11)	0.0408 (6)
C8	0.1404 (3)	1.0279 (3)	0.12343 (12)	0.0411 (6)
H8	0.0763	1.0392	0.0825	0.049*
O2	0.2034 (2)	1.1730 (2)	0.14353 (9)	0.0576 (5)
C9	0.1813 (3)	1.2918 (3)	0.10238 (14)	0.0537 (7)
C10	0.2675 (4)	1.4304 (3)	0.12631 (18)	0.0838 (10)
H10A	0.2424	1.5155	0.0976	0.126*
H10B	0.3791	1.4114	0.1262	0.126*
H10C	0.2367	1.4541	0.1707	0.126*
C11	0.3752 (3)	0.8805 (4)	0.16776 (13)	0.0665 (9)
H11A	0.3194	0.8153	0.1983	0.080*
H11B	0.4108	0.9713	0.1917	0.080*
C12	0.5133 (3)	0.7969 (4)	0.14161 (14)	0.0631 (8)
H12A	0.5217	0.6964	0.1621	0.076*
H12B	0.6096	0.8532	0.1520	0.076*
C13	0.3130 (3)	0.8817 (3)	0.05162 (12)	0.0409 (6)
C14	0.2788 (4)	0.8745 (3)	−0.05949 (15)	0.0638 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0543 (4)	0.0690 (5)	0.0636 (5)	0.0039 (4)	0.0152 (3)	−0.0048 (4)
O1	0.0687 (13)	0.0744 (14)	0.0521 (11)	−0.0061 (10)	0.0119 (9)	−0.0255 (10)
O3	0.199 (3)	0.0569 (14)	0.0711 (16)	−0.0233 (17)	−0.0545 (18)	0.0135 (12)
N1	0.0361 (10)	0.0495 (13)	0.0335 (11)	−0.0032 (9)	0.0015 (8)	0.0027 (9)

N2	0.0626 (14)	0.0613 (15)	0.0329 (12)	−0.0084 (11)	0.0015 (10)	−0.0033 (10)
N3	0.151 (3)	0.094 (2)	0.0454 (16)	−0.030 (2)	0.0220 (17)	−0.0161 (15)
C1	0.0454 (14)	0.0501 (16)	0.0398 (14)	−0.0043 (12)	0.0062 (11)	0.0022 (11)
C2	0.0575 (17)	0.0517 (17)	0.0543 (16)	−0.0074 (14)	0.0049 (13)	−0.0062 (13)
C3	0.0544 (17)	0.0488 (17)	0.074 (2)	−0.0090 (13)	0.0055 (14)	0.0084 (15)
C4	0.0463 (15)	0.0654 (19)	0.0559 (18)	−0.0030 (14)	0.0057 (13)	0.0208 (14)
C5	0.0379 (13)	0.0680 (18)	0.0372 (13)	0.0017 (13)	0.0044 (10)	0.0049 (12)
C6	0.0303 (11)	0.0488 (15)	0.0359 (13)	0.0020 (11)	0.0022 (10)	0.0015 (11)
C7	0.0377 (13)	0.0492 (15)	0.0354 (13)	0.0044 (11)	−0.0008 (10)	−0.0028 (12)
C8	0.0453 (14)	0.0374 (14)	0.0401 (13)	−0.0044 (11)	−0.0068 (11)	−0.0006 (11)
O2	0.0730 (12)	0.0415 (11)	0.0570 (12)	−0.0124 (9)	−0.0203 (9)	0.0054 (9)
C9	0.0671 (18)	0.0477 (17)	0.0462 (16)	−0.0078 (14)	0.0018 (13)	0.0003 (13)
C10	0.098 (3)	0.0502 (19)	0.102 (3)	−0.0221 (18)	−0.015 (2)	0.0055 (18)
C11	0.0541 (17)	0.098 (2)	0.0466 (16)	0.0191 (16)	−0.0078 (13)	−0.0024 (16)
C12	0.0464 (16)	0.078 (2)	0.0654 (19)	0.0064 (15)	0.0018 (13)	0.0114 (16)
C13	0.0435 (13)	0.0403 (14)	0.0391 (14)	−0.0138 (11)	0.0066 (11)	0.0010 (11)
C14	0.085 (2)	0.0584 (19)	0.0480 (18)	−0.0203 (16)	0.0042 (15)	−0.0025 (14)

Geometric parameters (Å, °)

S1—C13	1.738 (3)	C4—H4	0.9300
S1—C12	1.796 (3)	C5—C6	1.395 (3)
O1—C7	1.210 (3)	C5—H5	0.9300
O3—C9	1.179 (3)	C6—C7	1.484 (3)
N1—C13	1.346 (3)	C7—C8	1.538 (3)
N1—C8	1.439 (3)	C8—O2	1.427 (3)
N1—C11	1.456 (3)	C8—H8	0.9800
N2—C13	1.301 (3)	O2—C9	1.336 (3)
N2—C14	1.328 (3)	C9—C10	1.485 (4)
N3—C14	1.149 (4)	C10—H10A	0.9600
C1—C2	1.378 (4)	C10—H10B	0.9600
C1—C6	1.392 (3)	C10—H10C	0.9600
C1—H1	0.9300	C11—C12	1.487 (4)
C2—C3	1.384 (4)	C11—H11A	0.9700
C2—H2	0.9300	C11—H11B	0.9700
C3—C4	1.376 (4)	C12—H12A	0.9700
C3—H3	0.9300	C12—H12B	0.9700
C4—C5	1.369 (4)		
C13—S1—C12	92.71 (12)	O2—C8—H8	110.1
C13—N1—C8	122.5 (2)	N1—C8—H8	110.1
C13—N1—C11	116.6 (2)	C7—C8—H8	110.1
C8—N1—C11	120.26 (19)	C9—O2—C8	117.73 (19)
C13—N2—C14	117.7 (2)	O3—C9—O2	122.4 (3)
C2—C1—C6	120.1 (2)	O3—C9—C10	125.9 (3)
C2—C1—H1	120.0	O2—C9—C10	111.7 (2)
C6—C1—H1	120.0	C9—C10—H10A	109.5
C1—C2—C3	120.3 (3)	C9—C10—H10B	109.5
C1—C2—H2	119.9	H10A—C10—H10B	109.5
C3—C2—H2	119.9	C9—C10—H10C	109.5

C4—C3—C2	119.8 (3)	H10A—C10—H10C	109.5
C4—C3—H3	120.1	H10B—C10—H10C	109.5
C2—C3—H3	120.1	N1—C11—C12	109.3 (2)
C5—C4—C3	120.5 (2)	N1—C11—H11A	109.8
C5—C4—H4	119.8	C12—C11—H11A	109.8
C3—C4—H4	119.8	N1—C11—H11B	109.8
C4—C5—C6	120.4 (2)	C12—C11—H11B	109.8
C4—C5—H5	119.8	H11A—C11—H11B	108.3
C6—C5—H5	119.8	C11—C12—S1	108.89 (19)
C1—C6—C5	119.0 (2)	C11—C12—H12A	109.9
C1—C6—C7	122.7 (2)	S1—C12—H12A	109.9
C5—C6—C7	118.4 (2)	C11—C12—H12B	109.9
O1—C7—C6	122.7 (2)	S1—C12—H12B	109.9
O1—C7—C8	120.2 (2)	H12A—C12—H12B	108.3
C6—C7—C8	117.1 (2)	N2—C13—N1	120.9 (2)
O2—C8—N1	108.44 (19)	N2—C13—S1	126.66 (19)
O2—C8—C7	108.22 (19)	N1—C13—S1	112.39 (18)
N1—C8—C7	109.86 (19)	N3—C14—N2	174.3 (4)
C6—C1—C2—C3	0.1 (4)	O1—C7—C8—N1	115.7 (2)
C1—C2—C3—C4	1.2 (4)	C6—C7—C8—N1	−60.7 (3)
C2—C3—C4—C5	−1.5 (4)	N1—C8—O2—C9	113.8 (2)
C3—C4—C5—C6	0.5 (4)	C7—C8—O2—C9	−127.1 (2)
C2—C1—C6—C5	−1.1 (4)	C8—O2—C9—O3	7.7 (4)
C2—C1—C6—C7	178.6 (2)	C8—O2—C9—C10	−173.9 (2)
C4—C5—C6—C1	0.8 (3)	C13—N1—C11—C12	1.0 (4)
C4—C5—C6—C7	−178.9 (2)	C8—N1—C11—C12	−170.1 (2)
C1—C6—C7—O1	163.7 (2)	N1—C11—C12—S1	−2.8 (3)
C5—C6—C7—O1	−16.7 (3)	C13—S1—C12—C11	3.1 (2)
C1—C6—C7—C8	−20.0 (3)	C14—N2—C13—N1	178.2 (2)
C5—C6—C7—C8	159.7 (2)	C14—N2—C13—S1	−2.7 (3)
C13—N1—C8—O2	−106.5 (2)	C8—N1—C13—N2	−8.6 (3)
C11—N1—C8—O2	64.1 (3)	C11—N1—C13—N2	−179.5 (2)
C13—N1—C8—C7	135.4 (2)	C8—N1—C13—S1	172.23 (17)
C11—N1—C8—C7	−54.0 (3)	C11—N1—C13—S1	1.4 (3)
O1—C7—C8—O2	−2.5 (3)	C12—S1—C13—N2	178.3 (2)
C6—C7—C8—O2	−178.97 (18)	C12—S1—C13—N1	−2.61 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O3	0.98	2.22	2.649 (4)	105
C8—H8···N2	0.98	2.38	2.796 (3)	105

Fig. 1