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(*Z*)-5-Benzylidene-3-phenethyl-2-thioxoimidazolidin-4-one

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Key indicators

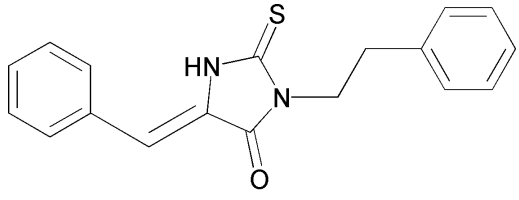
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.076
wR factor = 0.190
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{18}H_{16}N_2OS$, crystallizes in a triclinic unit cell with two independent molecules in the asymmetric unit. Intermolecular N—H···S, C—H···O and C—H···S hydrogen bonds link the molecules into ribbons, which form stacks with short intra- and intermolecular S···C [3.385 (5) Å] and C···O′ [3.162 (5) Å] contacts.

Comment

Imidazolinone derivatives exhibit various biological properties. Some of them have been reported to possess a broad spectrum of pharmacological activities, including anticonvulsant (Mehta *et al.*, 1981), antiviral (El-Barbary *et al.*, 1994) and antitumour activities (Khodair & Bertrand, 1998). As a result, considerable effort has been made to investigate imidazolinones (Heras *et al.*, 2001). The title compound, (I), is an intermediate in the preparation of 2-alkylthioimidazolones, which exhibit various fungicidal and herbicidal activities (Yang *et al.*, 2004).



(I)

The title compound, (I), exists in the *Z* isomeric form. In the crystal structure, there are two independent molecules in the asymmetric unit (Fig. 1). They have different conformations; for example, some of the torsion angles are different (Table 1).

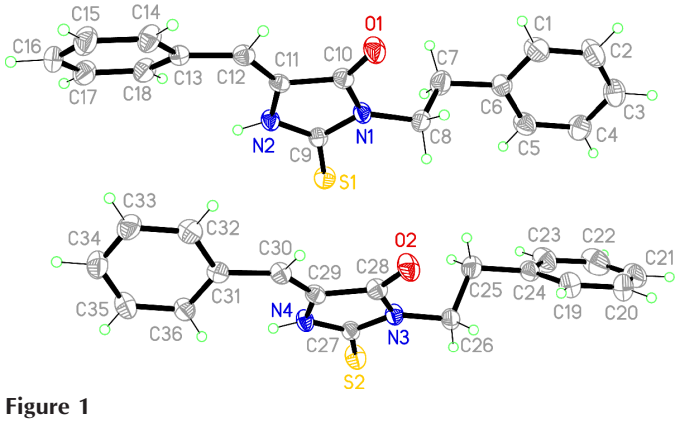


Figure 1
The asymmetric unit of (I), with the atom numbering, showing displacement ellipsoids at the 50% probability level.

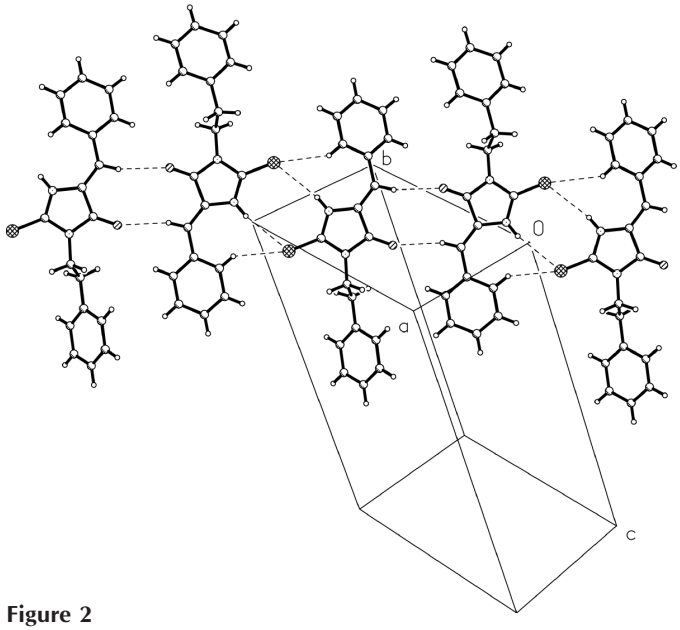


Figure 2
The hydrogen-bonded (dashed lines) ribbon in (I).

Intermolecular N—H···S, C—H···O and C—H···S hydrogen bonds link the molecules into ribbons (Fig. 2). Short intra- and intermolecular S1···C29 [3.385 (5) Å] and C28···O2^{iii} [3.162 (5) Å; symmetry code: (iii) $1-x, 1-y, -z$] contacts indicate π–π stacking interactions between the ribbons, forming an undulating sheet structure.

Experimental

To a solution of ethyl 3-phenyl-2-[(triphenylphosphoranylidene)-amino]prop-2-enoate (5 mmol) in dry dichloromethane was added carbon disulfide (50 mmol) and the mixture was refluxed for 29 h. The solvent was removed under pressure, and diethyl ether and petroleum ether (1:2, 60 ml) were added to precipitate triphenylphosphine oxide. The solution was filtered and the filtrate was concentrated *in vacuo* to give ethyl 3-phenyl-2-isothiocyanatoacrylate. To a solution of ethyl 3-phenyl-2-isothiocyanatoacrylate in dry acetonitrile (20 ml) was added phenylethylamine (5 mmol). The mixture was allowed to stand for 3 h, whereupon the title compound precipitated. Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethanol solution (m.p. 455–456 K). IR (KBr): 3246, 1727, 1653 cm^{-1}. ^{1}H NMR (chloroform-*d*): 9.00 (*s*, 1H), 7.22–7.45 (*m*, 10H), 6.73 (*s*, 1H), 4.13 (*t*, 2H, *J* = 7.5 Hz), 3.03 (*t*, 2H, *J* = 7.5 Hz) p.p.m. ^{13}C NMR (chloroform-*d*): 177.98, 163.39, 137.67, 132.79, 130.82, 130.50, 130.05, 129.59, 128.45, 128.09, 127.96, 127.49, 126.32, 125.64, 114.50, 112.44, 42.59, 33.66 p.p.m.

Crystal data

$C_{18}H_{16}N_2OS$
M_r = 308.39
Triclinic, $P\overline{1}$
a = 9.8221 (10) Å
b = 10.6654 (10) Å
c = 16.5207 (16) Å
α = 107.009 (2)°
β = 101.652 (2)°
γ = 100.645 (2)°
V = 1565.0 (3) Å^3
Z = 4
D_x = 1.309 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1368 reflections
θ = 2.7–24.1°
μ = 0.21 mm^{-1}
T = 298 (2) K
Block, colourless
0.23 × 0.18 × 0.15 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.953, T_{max} = 0.969
8414 measured reflections
5560 independent reflections
4149 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.2°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 10$
$l = -16 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.190
S = 1.13
5560 reflections
397 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2 + 0.2749P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C9	1.381 (4)	N3—C28	1.392 (4)
N1—C10	1.381 (4)	N3—C26	1.447 (4)
N1—C8	1.451 (4)	C7—C8	1.511 (5)
N3—C27	1.379 (4)	C25—C26	1.510 (5)
C9—N1—C8—C7	86.0 (4)	C27—N3—C26—C25	98.7 (4)
C10—N1—C8—C7	−95.7 (4)	C28—N3—C26—C25	−78.4 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···S1^{i}	0.86	2.71	3.549 (3)	165
N2—H2···S2^{i}	0.86	2.67	3.517 (3)	168
C12—H12···O2^{ii}	0.93	2.42	3.323 (4)	163
C18—H18···S2^{i}	0.93	2.71	3.415 (3)	133
C30—H30···O1^{ii}	0.93	2.44	3.336 (4)	163
C36—H36···S1^{i}	0.93	2.87	3.470 (3)	124

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

H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å and N—H = 0.86 Å, with $U_{iso} = 1.2U_{eq}$(C,N), and Csp^3—H = 0.96 Å, with $U_{iso} = 1.5U_{eq}$(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

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References

Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

El-Barbary, A. A., Khodair, A. I., Pedersen, E. B. & Nielsen, C. (1994). *J. Med. Chem.* **37**, 73–77.

Heras, M., Ventura, M., Linden, A. & Villalgordo, J. M. (2001). *Tetrahedron*, **57**, 4371–4388.
Khodair, A. I. & Bertrand, P. (1998). *Tetrahedron*, **54**, 4859–4872.
Mehta, N. B., Diuguid, C. A. R. & Soroko, F. E. (1981). *J. Med. Chem.* **24**, 465–468.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yang, F. L., Liu, Z. J., Huang, X. B. & Ding, M. W. (2004). *J. Heterocycl. Chem.* **41**, 77–83.