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(*E*)-Methyl 2-(3-cinnamoylthioureido)-acetate

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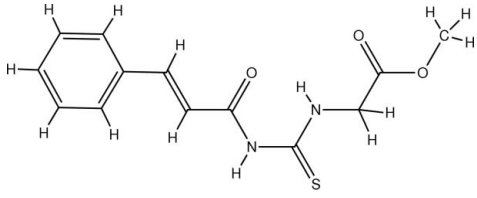
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.068; wR factor = 0.173; data-to-parameter ratio = 13.7.

In the title compound, $C_{13}H_{14}N_2O_3S$, the methyl 2-(3-formylthioureido)acetate fragment and the phenyl ring adopt an *E* configuration. The molecule exhibits an intramolecular N—H···O hydrogen bond, which completes a six-membered ring. The crystal packing is stabilized by intermolecular N—H···S contacts, generating a two-dimensional hydrogen-bonding network.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related structures, see: Yamin & Hassan (2004); Hassan *et al.* (2008*a*,*b*,*c*, 2009); Hung *et al.* (2010). For the preparation, see: Hassan *et al.* (2008*a*).



Experimental

Crystal data

$C_{13}H_{14}N_2O_3S$
M_r = 278.33
Triclinic, $P\overline{1}$
a = 4.992 (2) Å
b = 11.720 (5) Å
c = 12.542 (6) Å
α = 112.999 (7)°
β = 91.492 (7)°
γ = 96.258 (7)°
V = 669.6 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 298 K
0.38 × 0.32 × 0.13 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.912, T_{max} = 0.969
6562 measured reflections
2466 independent reflections
1689 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.173
S = 1.12
2466 reflections
180 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.86	1.88	2.610 (4)	142
N1—H1A···S1^{i}	0.85	2.61	3.463 (4)	176

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2273).

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supporting information

Acta Cryst. (2010). E**66**, o2242 [https://doi.org/10.1107/S1600536810030084]

(*E*)-Methyl 2-(3-cinnamoylthioureido)acetate

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S1. Comment

The title compound, I, is a methyl ester derivative of glycine thiourea analogoue to our previously reported molecules, methyl-2-(3-benzoylthioureido)acetate (II) (Hassan *et al.*, 2009), ethyl-2-(3-benzoylthioureido)acetate (III) (Hassan *et al.*, 2008*a*), propyl-2-(3-benzoylthioureido)acetate (IV) (Hassan *et al.*, 2008*b*) and butyl-2-(3-benzoylthioureido)acetate (V) (Hassan *et al.*, 2008*c*). Bond lengths and angles in the molecule are in normal ranges (Allen *et al.*, 1987) and comparable to those of the benzoyl derivatives II, III, IV and V. The methyl acetate, [O2/O3/C9/C10/C11] plane is inclined to the phenyl ring, [C1—C6] (A), with a dihedral angle of 26.78 (18)° and this angle is smaller than that of compound (II) [73.4 (2)°]. The phenyl ring and the thiourea fragment, [S1/N1/N2/C9/C10/C11] (B), are essentially planar. In the methyl acetate group, the maximum deviation from the mean plane is 0.037 (2)Å for the atom O3. The dihedral angles of the fragments A/B and B/C are 11.17 (14)° and 20.21 (15)°, respectively, whereas the dihedral angle of the A/C fragments is 73.4 (2)°. There is an intramolecular hydrogen bond, N2—H2A···O1 which completes a six-membered ring (N2/H2A/O1/C9/N1/C10) (Fig. 1) and an intermolecular N1—H1A···S1 hydrogen bond (Table 1) which generates a two-dimensional hydrogen bonding network.

S2. Experimental

The title compound was synthesised according to a previously reported procedure (Hassan *et al.*, 2008*a*). A colourless crystal, suitable for X-ray structuzre analysis was obtained by a slow evaporation from CH_2Cl_2 solution at room temperature (yield 73%).

S3. Refinement

H atoms of both C and N atoms were positioned geometrically and allowed to ride on their parent atoms, with U_{iso}=1.2U_{eq} (C) for aromatic 0.93 Å, U_{iso} = 1.2U_{eq} (C) for CH_2 0.97 Å, and U_{iso} = 1.5U_{eq} (C) for CH_3 0.96 Å. Hydrogen atoms attached to N were also positioned geometrically and allowed to ride on their parent atoms and with U_{iso}(H) = 1.2U_{eq}(N) for N–H 0.86 Å.

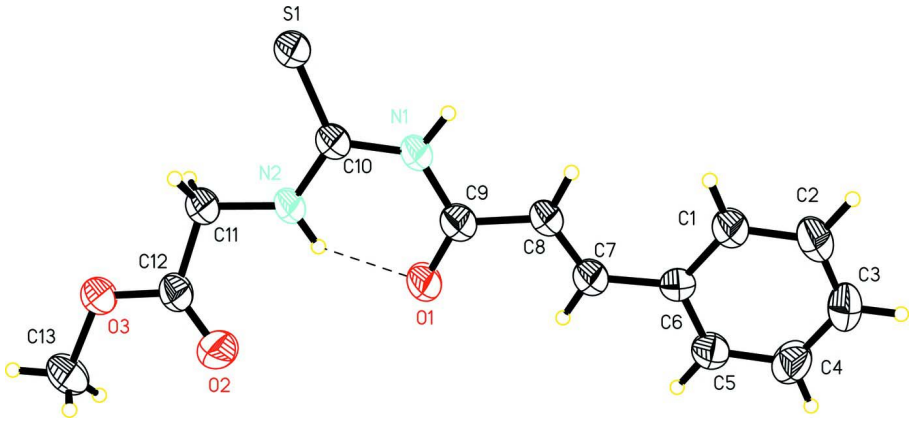

Figure 1

The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level. Intramolecular hydrogen bond is drawn by a dotted line.

(*E*)-Methyl 2-(3-cinnamoylthioureido)acetate

Crystal data

$C_{13}H_{14}N_2O_3S$
$M_r = 278.33$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.992$ (2) Å
$b = 11.720$ (5) Å
$c = 12.542$ (6) Å
$\alpha = 112.999$ (7)°
$\beta = 91.492$ (7)°
$\gamma = 96.258$ (7)°
$V = 669.6$ (5) Å^3
$Z = 2$
$F(000) = 292$
$D_x = 1.381$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1753 reflections
$\theta = 1.8$–25.5°
$\mu = 0.25$ mm^{-1}
$T = 298$ K
Block, colourless
0.38 × 0.32 × 0.13 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{min} = 0.912$, $T_{max} = 0.969$
6562 measured reflections
2466 independent reflections
1689 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 25.5°$, $\theta_{min} = 1.8°$
$h = -6 \rightarrow 6$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.173$
$S = 1.12$
2466 reflections
180 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0832P)^2 + 0.1736P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.2582 (2)	0.96891 (9)	0.84472 (8)	0.0546 (4)
O1	0.8785 (5)	1.3017 (3)	0.8950 (2)	0.0691 (9)
O2	0.6025 (5)	1.2077 (3)	0.5979 (2)	0.0728 (9)
O3	0.2006 (5)	1.1278 (2)	0.5020 (2)	0.0578 (7)
N1	0.6772 (6)	1.1430 (3)	0.9392 (2)	0.0423 (7)
N2	0.4711 (6)	1.1404 (3)	0.7718 (2)	0.0497 (8)
C1	1.5217 (7)	1.3497 (3)	1.2458 (3)	0.0541 (10)
H1B	1.4097	1.2766	1.2349	0.065*
C2	1.7365 (8)	1.3923 (4)	1.3298 (3)	0.0643 (11)
H2B	1.7663	1.3478	1.3753	0.077*
C3	1.9055 (8)	1.4992 (4)	1.3466 (3)	0.0596 (10)
H3A	2.0484	1.5274	1.4035	0.072*
C4	1.8621 (7)	1.5641 (3)	1.2790 (3)	0.0554 (10)
H4A	1.9769	1.6362	1.2891	0.066*
C5	1.6476 (7)	1.5223 (3)	1.1957 (3)	0.0477 (9)
H5A	1.6196	1.5672	1.1504	0.057*
C6	1.4733 (6)	1.4153 (3)	1.1780 (3)	0.0400 (8)
C7	1.2554 (7)	1.3734 (3)	1.0864 (3)	0.0453 (8)
H7A	1.2414	1.4237	1.0452	0.054*
C8	1.0742 (6)	1.2719 (3)	1.0546 (3)	0.0428 (8)
H8A	1.0749	1.2193	1.0942	0.051*
C9	0.8735 (7)	1.2429 (3)	0.9572 (3)	0.0479 (9)
C10	0.4759 (6)	1.0896 (3)	0.8501 (3)	0.0394 (8)
C11	0.2769 (7)	1.0976 (3)	0.6722 (3)	0.0496 (9)
H11A	0.1038	1.1253	0.6961	0.059*
H11B	0.2522	1.0070	0.6355	0.059*
C12	0.3849 (7)	1.1516 (3)	0.5889 (3)	0.0431 (8)
C13	0.2708 (9)	1.1823 (4)	0.4194 (3)	0.0680 (12)
H13A	0.1251	1.1594	0.3605	0.102*
H13B	0.4312	1.1520	0.3841	0.102*
H13C	0.3027	1.2716	0.4588	0.102*
H2A	0.591 (6)	1.204 (2)	0.787 (3)	0.076 (14)*
H1A	0.694 (8)	1.112 (3)	0.990 (3)	0.070 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0615 (6)	0.0558 (6)	0.0520 (6)	−0.0166 (4)	−0.0175 (4)	0.0350 (5)
O1	0.0748 (18)	0.0797 (19)	0.0662 (17)	−0.0313 (15)	−0.0313 (14)	0.0561 (16)
O2	0.0507 (17)	0.109 (2)	0.0733 (19)	−0.0241 (16)	−0.0172 (14)	0.0620 (18)
O3	0.0570 (15)	0.0750 (18)	0.0525 (15)	−0.0159 (13)	−0.0175 (12)	0.0450 (14)
N1	0.0475 (16)	0.0468 (17)	0.0386 (16)	−0.0049 (13)	−0.0107 (13)	0.0270 (14)
N2	0.0589 (19)	0.0551 (19)	0.0407 (16)	−0.0160 (15)	−0.0173 (14)	0.0324 (15)
C1	0.054 (2)	0.058 (2)	0.058 (2)	−0.0086 (18)	−0.0137 (18)	0.0368 (19)
C2	0.066 (3)	0.077 (3)	0.061 (2)	−0.005 (2)	−0.019 (2)	0.046 (2)
C3	0.053 (2)	0.070 (3)	0.050 (2)	−0.004 (2)	−0.0173 (18)	0.023 (2)
C4	0.050 (2)	0.048 (2)	0.061 (2)	−0.0087 (17)	−0.0087 (18)	0.0193 (19)
C5	0.051 (2)	0.047 (2)	0.051 (2)	−0.0028 (17)	−0.0041 (17)	0.0278 (18)
C6	0.0392 (18)	0.0427 (19)	0.0408 (18)	−0.0029 (15)	0.0001 (15)	0.0217 (16)
C7	0.047 (2)	0.049 (2)	0.0449 (19)	−0.0022 (17)	−0.0054 (16)	0.0267 (17)
C8	0.0451 (19)	0.050 (2)	0.0400 (19)	−0.0011 (16)	−0.0061 (15)	0.0271 (16)
C9	0.053 (2)	0.050 (2)	0.045 (2)	−0.0034 (17)	−0.0062 (17)	0.0270 (18)
C10	0.0424 (19)	0.0425 (19)	0.0379 (18)	0.0021 (15)	−0.0024 (15)	0.0221 (16)
C11	0.051 (2)	0.059 (2)	0.045 (2)	−0.0092 (18)	−0.0124 (17)	0.0330 (17)
C12	0.046 (2)	0.048 (2)	0.0401 (19)	0.0011 (17)	−0.0079 (16)	0.0246 (17)
C13	0.083 (3)	0.083 (3)	0.056 (2)	−0.009 (2)	−0.012 (2)	0.052 (2)

Geometric parameters (Å, º)

S1—C10	1.666 (3)	C3—H3A	0.9300
O1—C9	1.226 (4)	C4—C5	1.380 (5)
O2—C12	1.186 (4)	C4—H4A	0.9300
O3—C12	1.329 (4)	C5—C6	1.383 (4)
O3—C13	1.445 (4)	C5—H5A	0.9300
N1—C10	1.381 (4)	C6—C7	1.456 (5)
N1—C9	1.383 (4)	C7—C8	1.330 (5)
N1—H1A	0.859 (10)	C7—H7A	0.9300
N2—C10	1.333 (4)	C8—C9	1.466 (5)
N2—C11	1.447 (4)	C8—H8A	0.9300
N2—H2A	0.859 (10)	C11—C12	1.502 (4)
C1—C6	1.383 (5)	C11—H11A	0.9700
C1—C2	1.387 (5)	C11—H11B	0.9700
C1—H1B	0.9300	C13—H13A	0.9600
C2—C3	1.371 (6)	C13—H13B	0.9600
C2—H2B	0.9300	C13—H13C	0.9600
C3—C4	1.370 (5)		
C12—O3—C13	116.2 (3)	C8—C7—H7A	116.1
C10—N1—C9	127.9 (3)	C6—C7—H7A	116.1
C10—N1—H1A	120 (3)	C7—C8—C9	120.0 (3)
C9—N1—H1A	112 (3)	C7—C8—H8A	120.0
C10—N2—C11	124.3 (3)	C9—C8—H8A	120.0

C10—N2—H2A	114 (3)	O1—C9—N1	121.8 (3)
C11—N2—H2A	122 (3)	O1—C9—C8	122.9 (3)
C6—C1—C2	120.3 (3)	N1—C9—C8	115.3 (3)
C6—C1—H1B	119.8	N2—C10—N1	116.0 (3)
C2—C1—H1B	119.8	N2—C10—S1	123.9 (2)
C3—C2—C1	120.8 (4)	N1—C10—S1	120.2 (2)
C3—C2—H2B	119.6	N2—C11—C12	107.6 (3)
C1—C2—H2B	119.6	N2—C11—H11A	110.2
C4—C3—C2	119.4 (3)	C12—C11—H11A	110.2
C4—C3—H3A	120.3	N2—C11—H11B	110.2
C2—C3—H3A	120.3	C12—C11—H11B	110.2
C3—C4—C5	119.8 (3)	H11A—C11—H11B	108.5
C3—C4—H4A	120.1	O2—C12—O3	124.0 (3)
C5—C4—H4A	120.1	O2—C12—C11	125.7 (3)
C4—C5—C6	121.7 (3)	O3—C12—C11	110.3 (3)
C4—C5—H5A	119.2	O3—C13—H13A	109.5
C6—C5—H5A	119.2	O3—C13—H13B	109.5
C1—C6—C5	117.9 (3)	H13A—C13—H13B	109.5
C1—C6—C7	122.8 (3)	O3—C13—H13C	109.5
C5—C6—C7	119.2 (3)	H13A—C13—H13C	109.5
C8—C7—C6	127.8 (3)	H13B—C13—H13C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.88	2.610 (4)	142
N1—H1*A*···S1^{i}	0.85	2.61	3.463 (4)	176

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