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1-(2-Morpholinoethyl)-3-(3-phenylacryloyl)thiourea

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Key indicators

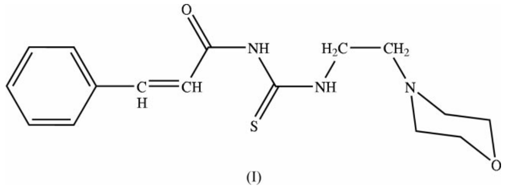
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.131
Data-to-parameter ratio = 16.4

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

The title compound, $C_{16}H_{21}N_3O_2S$, is a thiourea derivative with cinnamoyl and 2-morpholinoethyl groups attached at the terminal two N atoms. The groups lie *trans* and *cis*, respectively, to the S atom across the thiourea C—N bonds and the morpholine group adopts a chair conformation. The molecules are linked by N—H$\cdots$O hydrogen bonds, forming an infinite one-dimensional chain along the *b* axis.

Comment

Most carbonylthiourea derivatives of the type R^1C(O)NHC(*S*)NHR^2 have a *trans–cis* configuration with respect to the position of the R^1C(O) and R^2 groups across the thiourea C—N bonds, respectively, such as in *N*-benzoyl-*N*′-phenylthiourea (Yamin & Yusof, 2003*a*), *N*-benzoyl-*N*′(*p*-bromophenyl)thiourea (Yamin & Yusof, 2003*b*) and *N*-benzoyl-*N*′-(2-chlorophenyl)thiourea (Yusof & Yamin, 2004). Similarly, in the title compound, (I), the cinnamoyl and 2-morpholinoethyl groups are *trans* and *cis*, respectively, to the S atom across the thiourea C—N bonds (Fig. 1 and Table 1). The morpholine group adopts a chair conformation. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and in agreement with other thiourea derivatives. The C7—C8 bond [1.311 (3) Å] has double-bond character, with a *trans* configuration with respect to the H atoms at both C atoms.



(I)

The maximum deviation from the plane of the central fragment (S1/O1/N1/N2/C7/C8/C9/C10) is 0.049 (2) Å for atom O1. The dihedral angle between this plane and and the phenyl group (C1–C6) is 22.39 (9)°. Three intramolecular hydrogen bonds, *viz*. N2—H2*A*$\cdots$O1, N2—H2*A*$\cdots$N3 and C7—H7$\cdots$O1, are observed (Fig. 1 and Table 2). As a result, two pseudo-five-membered (C7—C8—C9—O1$\cdots$H and N3—C12—C11—N2—H2*A*) and one pseudo-six-membered (C9—N1—C10—N2—H2*A*$\cdots$O1) rings are formed. In the crystal structure, the molecule is stabilized by intermolecular N1—H1*A*$\cdots$O2^i [symmetry code: (i) *x*, 1 + *y*, *z*; Table 2] hydrogen bonds, forming one-dimensional chains extending along the *b* axis (Fig. 2).

Experimental

Ammonium thiocyanate (0.761 g, 10 mmol) was added to an acetone solution (20 ml) containing cinnamoyl chloride (1.666 g, 10 mmol). After stirring for 15 min, a solution (10 ml) of 2-morpholinoethylamine (1.301 g, 10 mmol) was added. The resulting solution was then refluxed for 1 h. After cooling, the solution was poured into a beaker containing some ice. The white precipitate was filtered off and washed with distilled water several times and then dried under vacuum (yield, 69%). Crystals were obtained from acetone; the melting point was 452.5 K.

Crystal data

$C_{16}H_{21}N_3O_2S$
$M_r = 319.42$
Triclinic, $P\overline{1}$
$a = 6.1452$ (14) Å
$b = 9.731$ (2) Å
$c = 14.690$ (3) Å
$\alpha = 98.711$ (4)°
$\beta = 93.971$ (4)°
$\gamma = 104.444$ (4)°
$V = 835.6$ (3) Å^3
$Z = 2$
$D_x = 1.270$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 775 reflections
$\theta = 1.4$–26.0°
$\mu = 0.20$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.41 × 0.34 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.921$, $T_{\max} = 0.960$
8635 measured reflections
3267 independent reflections
2748 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 26.0°$
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.131$
$S = 1.15$
3267 reflections
199 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.1504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C10	1.6675 (19)	N2—C10	1.314 (3)
O1—C9	1.219 (2)	N2—C11	1.449 (3)
O2—C15	1.425 (3)	N3—C16	1.450 (3)
O2—C14	1.426 (3)	N3—C13	1.453 (3)
N1—C9	1.377 (2)	N3—C12	1.460 (3)
N1—C10	1.389 (3)	C7—C8	1.311 (3)
C9—N1—C10	128.18 (17)	N2—C10—N1	116.43 (17)
C10—N2—C11	124.72 (17)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.95	2.642 (3)	136
N2—H2*A*···N3	0.86	2.39	2.758 (2)	107
C7—H7···O1	0.93	2.48	2.813 (3)	101
N1—H1*A*···O2$^{\rm i}$	0.86	2.27	3.114 (2)	169

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

After their location in a difference map, all H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) and 1.2$U_{\rm eq}$(N).

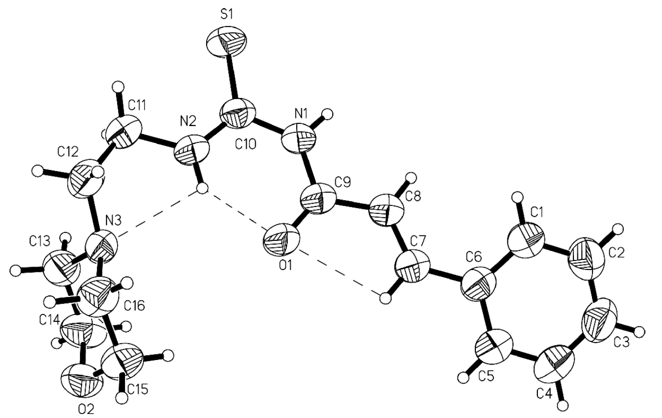


Figure 1
The molecular structure of the title compound, (I), with 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

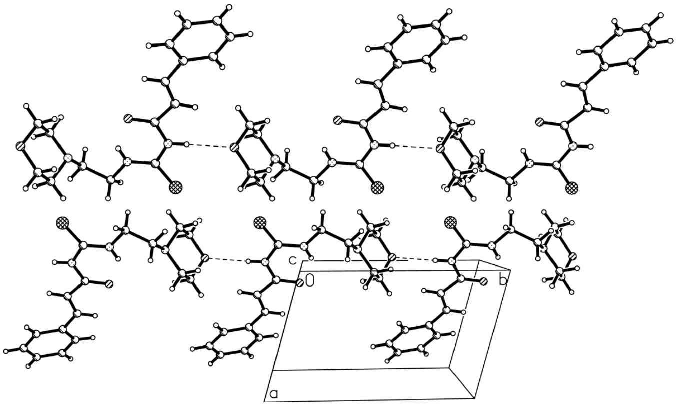


Figure 2
Packing diagram of the title compound, viewed down the *c* axis. The dashed lines denote N—H···O hydrogen bonds.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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