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(*E*)-1-(4-Methylphenyl)-3-[(1-phenylethylidene)amino]thiourea

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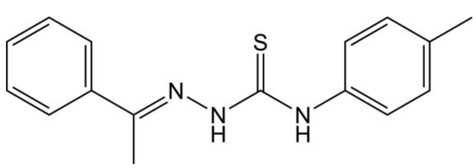
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 15.4.

In the title compound, $C_{16}H_{17}N_3S$, the aminothiourea unit is nearly planar (r.m.s. deviation = 0.0425 Å), and is twisted with respect to the tolyl and phenyl rings by 57.84 (7) and 15.88 (14)°, respectively; the tolyl and phenyl rings are twisted by 65.64 (11)° to each other. Intermolecular N—H⋯S and weak C—H⋯S hydrogen bonds are present in the crystal structure.

Related literature

The title compound is a derivative of thiosemicarbazone. For applications of thiosemicarbazones in the biological field, see: Hu *et al.* (2006).



Experimental

Crystal data

$C_{16}H_{17}N_3S$
M_r = 283.39
Monoclinic, $P2_1/c$
a = 10.5881 (3) Å
b = 5.7355 (2) Å
c = 26.9746 (7) Å
β = 108.670 (2)°
V = 1551.91 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 1.79 mm^{-1}
T = 291 K
0.25 × 0.18 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.60, T_{max} = 0.73
15835 measured reflections
2945 independent reflections
2587 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.06
2945 reflections
191 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2⋯S1^{i}	0.90 (2)	2.86 (2)	3.7456 (16)	167.8 (19)
C16—H16B⋯S1^{i}	0.96	2.74	3.471 (2)	133

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5214).

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supplementary materials

Acta Cryst. (2011). E**67**, o1547 [**doi:10.1107/S1600536811019404**]

(*E*)-1-(4-Methylphenyl)-3-[(1-phenylethylidene)amino]thiourea

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Comment

Thiosemicarbazones have attracted our attention because of their biological applications (Hu *et al.*, 2006). A few single-crystal structures were reported. For understanding their anticancer activity, it is necessary to have detailed information on their geometries.

The molecular structure of (I) is shown in Fig 1. The molecules reveal an E configuration. The dihedral angles formed by the tolyl and phenyl rings with the almost planar aminothiourea unit (r.m.s. deviation = 0.0425 Å) are 57.84 (7) and 15.88 (14)°, respectively. Intermolecular N—H···S and C—H···S interactions occur in the crystal structure (Table 1).

Experimental

N-(*p*-Tolyl)thiosemicarbazide (2.7 g, 15 mmol) and acetophenone (1.8 g, 15 mmol) was dissolved in 95% ethanol (20 ml), and the solution was refluxed for 3 h. Fine colorless crystals appeared on cooling. They were filtered and washed by 95% ethanol to give 2.9 g of the title compound in 69.0% yield. Single crystals suitable for X-ray measurements were obtained from ether by slow evaporation at room temperature.

Refinement

Imino H atoms were located in a difference Fourier map and refined isotropically. Other H atoms were placed in calculated positions with C—H = 0.93–0.96 and refined using a riding model, U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for the others.

Figures

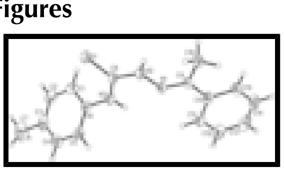

Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at 30% probability level.

(*E*)-1-(4-Methylphenyl)-3-[(1-phenylethylidene)amino]thiourea

Crystal data

$C_{16}H_{17}N_3S$	*F*(000) = 600
M_r = 283.39	D_x = 1.213 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu *K*α radiation, λ = 1.5418 Å
a = 10.5881 (3) Å	Cell parameters from 6724 reflections
b = 5.7355 (2) Å	θ = 3.5–70.9°

c = 26.9746 (7) Å	μ = 1.79 mm^{-1}
β = 108.670 (2)°	T = 291 K
V = 1551.91 (8) Å^3	Prismatic, colorless
Z = 4	0.25 × 0.18 × 0.18 mm

Data collection

Oxford Diffraction Xcalibur Eos Gemini diffractometer	2945 independent reflections
Radiation source: Enhance (Cu) X-ray Source	2587 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.041
Detector resolution: 16.2312 pixels mm^{-1}	θ_{max} = 70.9°, θ_{min} = 3.5°
ω scans	$h = -12 \rightarrow 12$
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	$k = -7 \rightarrow 6$
T_{min} = 0.60, T_{max} = 0.73	$l = -28 \rightarrow 31$
15835 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.044	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.126	H atoms treated by a mixture of independent and constrained refinement
S = 1.06	$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 0.2489P]$ where $P = (F_o^2 + 2F_c^2)/3$
2945 reflections	$(\Delta/\sigma)_{max}$ = 0.001
191 parameters	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.24 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}

S1	1.08154 (5)	0.02598 (9)	0.436028 (18)	0.05334 (18)
N1	0.82552 (14)	0.5213 (3)	0.42960 (6)	0.0465 (3)
N2	0.90859 (14)	0.3341 (3)	0.44695 (6)	0.0499 (4)
N3	0.99833 (16)	0.4238 (3)	0.38311 (6)	0.0510 (4)
C1	1.06088 (19)	0.2091 (4)	0.31610 (7)	0.0559 (5)
H1	1.0044	0.0876	0.3179	0.067*
C2	1.1318 (2)	0.1997 (4)	0.28098 (7)	0.0656 (6)
H2A	1.1229	0.0699	0.2595	0.079*
C3	1.2149 (2)	0.3777 (5)	0.27725 (7)	0.0651 (6)
C4	1.2278 (2)	0.5681 (4)	0.30964 (8)	0.0630 (5)
H4	1.2841	0.6896	0.3077	0.076*
C5	1.15789 (19)	0.5810 (4)	0.34519 (7)	0.0520 (4)
H5	1.1672	0.7109	0.3667	0.062*
C6	1.07490 (16)	0.4014 (3)	0.34853 (6)	0.0443 (4)
C7	1.2928 (3)	0.3658 (8)	0.23886 (12)	0.1137 (13)
H7A	1.2491	0.4593	0.2087	0.171*
H7B	1.2971	0.2070	0.2283	0.171*
H7C	1.3814	0.4237	0.2553	0.171*
C8	0.99248 (15)	0.2719 (3)	0.42007 (6)	0.0440 (4)
C9	0.74014 (16)	0.5714 (3)	0.45287 (7)	0.0437 (4)
C10	0.64955 (15)	0.7680 (3)	0.43048 (6)	0.0452 (4)
C11	0.6772 (2)	0.9235 (4)	0.39573 (7)	0.0584 (5)
H11	0.7547	0.9058	0.3869	0.070*
C12	0.5910 (3)	1.1032 (5)	0.37429 (9)	0.0761 (7)
H12	0.6102	1.2057	0.3510	0.091*
C13	0.4749 (2)	1.1320 (5)	0.38739 (9)	0.0783 (8)
H13	0.4162	1.2526	0.3726	0.094*
C14	0.4478 (2)	0.9828 (5)	0.42192 (12)	0.0773 (7)
H14	0.3707	1.0024	0.4309	0.093*
C15	0.53386 (19)	0.8031 (4)	0.44367 (10)	0.0665 (6)
H15	0.5146	0.7037	0.4675	0.080*
C16	0.7269 (2)	0.4397 (4)	0.49895 (8)	0.0570 (5)
H16A	0.6858	0.5377	0.5182	0.086*
H16B	0.8137	0.3932	0.5212	0.086*
H16C	0.6728	0.3038	0.4869	0.086*
H2	0.904 (2)	0.230 (4)	0.4714 (8)	0.058 (6)*
H3	0.960 (2)	0.548 (4)	0.3831 (8)	0.053 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0594 (3)	0.0488 (3)	0.0626 (3)	0.01893 (19)	0.0347 (2)	0.01199 (19)
N1	0.0428 (7)	0.0442 (9)	0.0593 (8)	0.0093 (6)	0.0257 (6)	0.0052 (6)
N2	0.0502 (8)	0.0488 (9)	0.0607 (8)	0.0150 (7)	0.0319 (6)	0.0117 (7)
N3	0.0550 (8)	0.0492 (10)	0.0578 (8)	0.0193 (7)	0.0305 (7)	0.0099 (7)
C1	0.0614 (10)	0.0575 (12)	0.0464 (8)	0.0029 (9)	0.0138 (7)	−0.0037 (8)
C2	0.0828 (13)	0.0722 (15)	0.0404 (8)	0.0195 (11)	0.0177 (8)	−0.0086 (8)
C3	0.0717 (12)	0.0870 (17)	0.0436 (9)	0.0292 (12)	0.0282 (8)	0.0116 (9)

C4	0.0648 (11)	0.0709 (14)	0.0624 (11)	0.0067 (10)	0.0333 (9)	0.0157 (10)
C5	0.0624 (10)	0.0480 (11)	0.0518 (9)	0.0075 (8)	0.0268 (8)	0.0018 (7)
C6	0.0450 (8)	0.0503 (10)	0.0395 (7)	0.0137 (7)	0.0162 (6)	0.0063 (6)
C7	0.130 (3)	0.159 (3)	0.0798 (16)	0.037 (2)	0.0733 (18)	0.0119 (19)
C8	0.0398 (7)	0.0478 (10)	0.0487 (8)	0.0052 (7)	0.0201 (6)	0.0017 (7)
C9	0.0392 (7)	0.0441 (10)	0.0535 (8)	0.0035 (7)	0.0229 (6)	0.0005 (7)
C10	0.0375 (7)	0.0506 (10)	0.0490 (8)	0.0058 (7)	0.0159 (6)	−0.0033 (7)
C11	0.0623 (11)	0.0632 (13)	0.0556 (10)	0.0202 (9)	0.0273 (8)	0.0082 (9)
C12	0.0911 (16)	0.0768 (17)	0.0577 (11)	0.0305 (13)	0.0201 (10)	0.0146 (11)
C13	0.0655 (12)	0.0806 (18)	0.0706 (13)	0.0360 (12)	−0.0040 (10)	−0.0071 (12)
C14	0.0433 (10)	0.0823 (18)	0.1062 (18)	0.0183 (10)	0.0237 (11)	−0.0094 (14)
C15	0.0480 (10)	0.0686 (15)	0.0930 (14)	0.0100 (9)	0.0367 (10)	0.0057 (12)
C16	0.0595 (10)	0.0586 (12)	0.0638 (10)	0.0104 (9)	0.0351 (8)	0.0095 (9)

Geometric parameters (Å, °)

S1—C8	1.6746 (18)	C7—H7A	0.9600
N1—N2	1.372 (2)	C7—H7B	0.9600
N1—C9	1.287 (2)	C7—H7C	0.9600
N2—C8	1.362 (2)	C9—C10	1.478 (2)
N2—H2	0.90 (2)	C9—C16	1.499 (2)
N3—C6	1.424 (2)	C10—C11	1.391 (3)
N3—C8	1.340 (2)	C10—C15	1.395 (2)
N3—H3	0.82 (2)	C11—H11	0.9300
C1—H1	0.9300	C11—C12	1.375 (3)
C1—C2	1.386 (3)	C12—H12	0.9300
C1—C6	1.386 (3)	C12—C13	1.393 (4)
C2—H2A	0.9300	C13—H13	0.9300
C2—C3	1.373 (4)	C13—C14	1.361 (4)
C3—C4	1.378 (4)	C14—H14	0.9300
C3—C7	1.517 (3)	C14—C15	1.376 (3)
C4—H4	0.9300	C15—H15	0.9300
C4—C5	1.388 (3)	C16—H16A	0.9600
C5—H5	0.9300	C16—H16B	0.9600
C5—C6	1.376 (3)	C16—H16C	0.9600
C9—N1—N2	118.83 (15)	N2—C8—S1	119.57 (13)
N1—N2—H2	126.2 (14)	N3—C8—S1	125.62 (12)
C8—N2—N1	118.67 (15)	N3—C8—N2	114.78 (16)
C8—N2—H2	114.3 (14)	N1—C9—C10	115.87 (15)
C6—N3—H3	117.8 (16)	N1—C9—C16	123.94 (16)
C8—N3—C6	126.88 (15)	C10—C9—C16	120.17 (14)
C8—N3—H3	115.0 (16)	C11—C10—C9	121.03 (15)
C2—C1—H1	120.3	C11—C10—C15	118.04 (18)
C2—C1—C6	119.4 (2)	C15—C10—C9	120.93 (17)
C6—C1—H1	120.3	C10—C11—H11	119.7
C1—C2—H2A	119.3	C12—C11—C10	120.6 (2)
C3—C2—C1	121.4 (2)	C12—C11—H11	119.7
C3—C2—H2A	119.3	C11—C12—H12	119.9
C2—C3—C4	118.58 (18)	C11—C12—C13	120.2 (2)

C2—C3—C7	121.2 (3)	C13—C12—H12	119.9
C4—C3—C7	120.2 (3)	C12—C13—H13	120.2
C3—C4—H4	119.5	C14—C13—C12	119.7 (2)
C3—C4—C5	121.0 (2)	C14—C13—H13	120.2
C5—C4—H4	119.5	C13—C14—H14	119.8
C4—C5—H5	120.1	C13—C14—C15	120.5 (2)
C6—C5—C4	119.86 (19)	C15—C14—H14	119.8
C6—C5—H5	120.1	C10—C15—H15	119.5
C1—C6—N3	121.13 (18)	C14—C15—C10	121.0 (2)
C5—C6—N3	119.00 (17)	C14—C15—H15	119.5
C5—C6—C1	119.75 (16)	C9—C16—H16A	109.5
C3—C7—H7A	109.5	C9—C16—H16B	109.5
C3—C7—H7B	109.5	C9—C16—H16C	109.5
C3—C7—H7C	109.5	H16A—C16—H16B	109.5
H7A—C7—H7B	109.5	H16A—C16—H16C	109.5
H7A—C7—H7C	109.5	H16B—C16—H16C	109.5
H7B—C7—H7C	109.5		

N1—N2—C8—S1	−172.72 (13)	C6—C1—C2—C3	−0.6 (3)
N1—N2—C8—N3	9.3 (2)	C7—C3—C4—C5	−179.6 (2)
N1—C9—C10—C11	−15.8 (3)	C8—N3—C6—C1	56.4 (3)
N1—C9—C10—C15	164.35 (19)	C8—N3—C6—C5	−127.6 (2)
N2—N1—C9—C10	−177.12 (15)	C9—N1—N2—C8	175.59 (16)
N2—N1—C9—C16	1.2 (3)	C9—C10—C11—C12	178.7 (2)
C1—C2—C3—C4	0.4 (3)	C9—C10—C15—C14	−178.5 (2)
C1—C2—C3—C7	179.7 (2)	C10—C11—C12—C13	0.3 (4)
C2—C1—C6—N3	176.51 (16)	C11—C10—C15—C14	1.6 (3)
C2—C1—C6—C5	0.6 (3)	C11—C12—C13—C14	0.6 (4)
C2—C3—C4—C5	−0.3 (3)	C12—C13—C14—C15	−0.4 (4)
C3—C4—C5—C6	0.3 (3)	C13—C14—C15—C10	−0.8 (4)
C4—C5—C6—N3	−176.47 (16)	C15—C10—C11—C12	−1.4 (3)
C4—C5—C6—C1	−0.4 (3)	C16—C9—C10—C11	165.88 (19)
C6—N3—C8—S1	3.6 (3)	C16—C9—C10—C15	−14.0 (3)
C6—N3—C8—N2	−178.56 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···S1^{i}	0.90 (2)	2.86 (2)	3.7456 (16)	167.8 (19)
C16—H16B···S1^{i}	0.96	2.74	3.471 (2)	133

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Fig. 1