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

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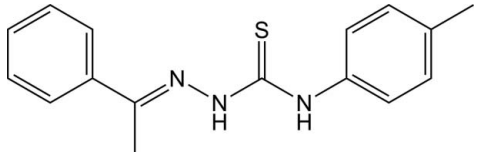
Key indicators: single-crystal X-ray study; $T$ = 291 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.126; data-to-parameter ratio = 15.4.

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
$\beta$ = 108.670 (2)°
$V$ = 1551.91 (8) Å$^3$
$Z$ = 4
Cu $K\alpha$ radiation
$\mu$ = 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—H16$B$···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).

## References

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Hu, W.-X., Zhou, W., Xia, C.-N. & Wen, X. (2006). *Bioorg. Med. Chem. Lett.* **16**, 2213–2218.
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# supporting information

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

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

**Yan-Ling Zhang, Chang-Zeng Wu and Fu-Juan Zhang**

### S1. 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).

### S2. 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.

### S3. 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) = 1.5$U_{eq}$(C) for methyl H atoms and 1.2$U_{eq}$(C) for the others.

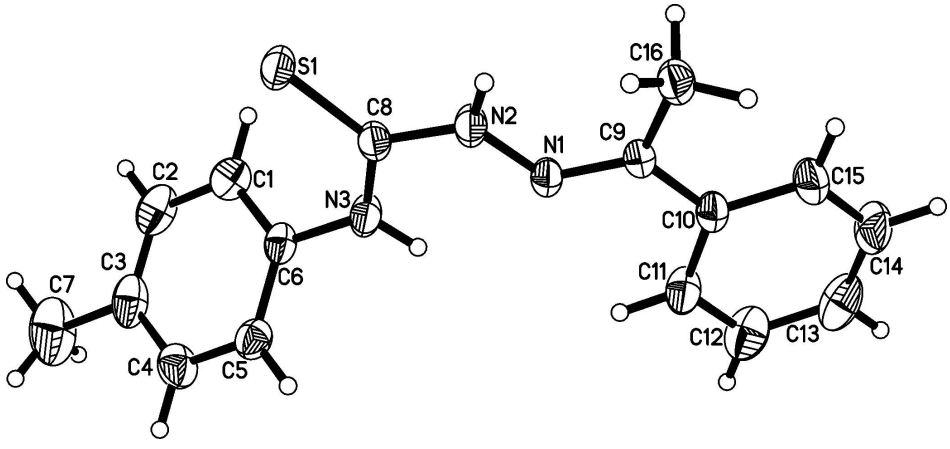


**Figure 1**

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*

$C_{16}H_{17}N_3S$
$M_r = 283.39$
Monoclinic, $P2_1/c$
$a = 10.5881$ (3) Å
$b = 5.7355$ (2) Å
$c = 26.9746$ (7) Å
$\beta = 108.670$ (2)°
$V = 1551.91$ (8) Å$^3$
$Z = 4$
$F(000) = 600$
$D_x = 1.213$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.5418$ Å
Cell parameters from 6724 reflections
$\theta = 3.5$–$70.9°$
$\mu = 1.79$ mm$^{-1}$
$T = 291$ K
Prismatic, colorless
0.25 × 0.18 × 0.18 mm

*Data collection*

Oxford Diffraction Xcalibur Eos Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.2312 pixels mm$^{-1}$
$\omega$ scans
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 $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 70.9°$, $\theta_{min} = 3.5°$
$h = -12 \rightarrow 12$
$k = -7 \rightarrow 6$
$l = -28 \rightarrow 31$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.126$
$S = 1.06$
2945 reflections
191 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 0.2489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *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—H16*B*···S1$^{i}$ | 0.96 | 2.74 | 3.471 (2) | 133 |

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