

(E)-5-tert-Butyl-1-cinnamoylthiobiuret**Si-Jia Xue,* An Chai, Yan-Lin Guo and Qing-Dong Wang**

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Key indicatorsSingle-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.076
 wR factor = 0.172
Data-to-parameter ratio = 10.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

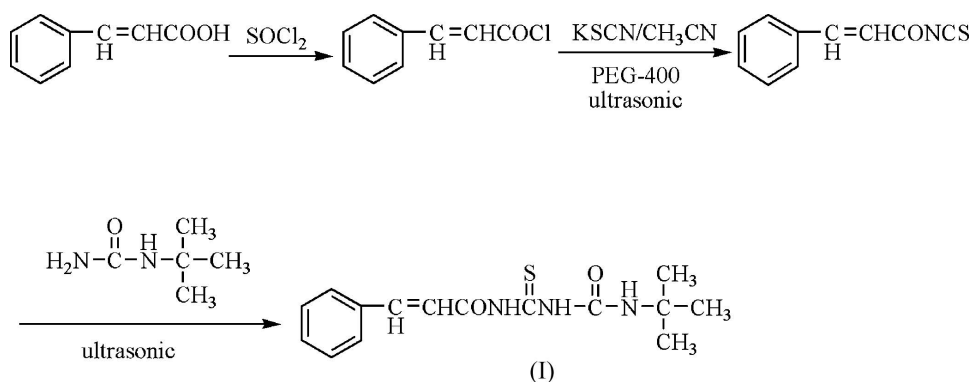
The title compound, $\text{C}_{15}\text{H}_{19}\text{N}_3\text{O}_2\text{S}$, is one of the thiourea herbicides in which 1-*tert*-butylurea replaces 4,6-disubstituted pyrimidine. The crystal structure reveals intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds that form six-membered rings, whereas intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds connect molecules along the b axis.

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Comment

Thiourea compounds display high biological activity as herbicides with low toxicity and low residue content. They are used extensively as pesticides, fungicides and regulating agents of plant growth in the agrochemical industry (Pu *et al.*, 1994; McCourt *et al.*, 2005). Thus, thiourea herbicides are a subject of intensive research and many novel structural thiourea herbicides have appeared in the literature (Ehrenfreund 1988; Takematsu *et al.*, 1988; Kehne *et al.*, 1991). We modified the synthesis of (I) according to Reeves *et al.* (1981), using 1-*tert*-butylurea instead of 4,6-disubstituted pyrimidine. The key feature of this thiourea herbicide is the dicarbonylthiourea, which might provide an opportunity for the study of the cooperative effect of combining these biologically active components in a single molecule. We report here the crystal structure of the title compound, (I) (Fig. 1).



There are two intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds, forming six-membered rings. Intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonding connects molecules along the b axis (Fig. 2 and Table 2).

Experimental

(*E*)-3-Phenylprop-2-enoyl isothiocyanate was synthesized according to the reported methods of Jiang *et al.* (2000) and Wang *et al.* (2001). The synthetic routes are indicated in the scheme (see *Comment*; PEG-400 is polyethylene glycol 400). Reaction of the isothiocyanate

derivative with 1-*tert*-butylurea was successfully carried out using acetonitrile as solvent. To a stirred solution of 3-phenylprop-2-enoyl isothiocyanate (1.89 g, 10 mmol) in acetonitrile (10 ml) was slowly added a solution of 1-*tert*-butylurea (1.16 g, 10 mmol) in dry acetonitrile (10 ml) over a period of 30 min at room temperature under nitrogen. The mixture was refluxed and stirred for 2 h. After being cooled to room temperature, a yellow solid precipitated from a red-orange solution immersed in the water bath of an ultrasonic cleaner at 333 K for 25–30 min. The residue was obtained after filtration and washing with water, and was crystallized from C₂H₅OH/H₂O (5:1 v/v), giving (I) as yellow crystals in 68% yield.

Crystal data

C₁₅H₁₉N₃O₂S
M_r = 305.39
 Orthorhombic, *Pbca*
a = 7.898 (2) Å
b = 11.910 (5) Å
c = 35.170 (16) Å
V = 3309 (2) Å³
Z = 8
D_x = 1.226 Mg m⁻³
 Mo *K*α radiation
 μ = 0.20 mm⁻¹
T = 298 (2) K
 Prism, yellow
 0.35 × 0.10 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.932, *T_{max}* = 0.990
 12822 measured reflections
 2908 independent reflections
 2209 reflections with *I* > 2σ(*I*)
R_{int} = 0.055
 θ_{max} = 25.0°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.076
wR (*F*²) = 0.172
S = 1.15
 2908 reflections
 267 parameters
 All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 4.2027P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.34 e Å⁻³
 Δρ_{min} = -0.21 e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0009 (7)

Table 1

Selected geometric parameters (Å, °).

S1—C10	1.659 (4)	N1—C10	1.392 (5)
O1—C9	1.222 (4)	N2—C10	1.341 (5)
O2—C11	1.222 (4)	N2—C11	1.429 (5)
N1—C9	1.382 (5)	N3—C11	1.319 (5)
O1—C9—N1	123.0 (4)	N2—C10—S1	128.0 (3)
N2—C10—N1	114.7 (3)	N3—C11—N2	116.9 (3)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N3—H3A...S1	0.97 (4)	2.21 (4)	3.058 (4)	146 (3)
N2—H2A...O1	0.90 (4)	1.92 (4)	2.634 (4)	135 (3)
N1—H1A...O2 ⁱ	0.97 (4)	1.86 (5)	2.820 (4)	176 (4)

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

H atoms were located in difference maps and refined freely [*C*—H = 0.83 (6)–1.09 (5) Å].

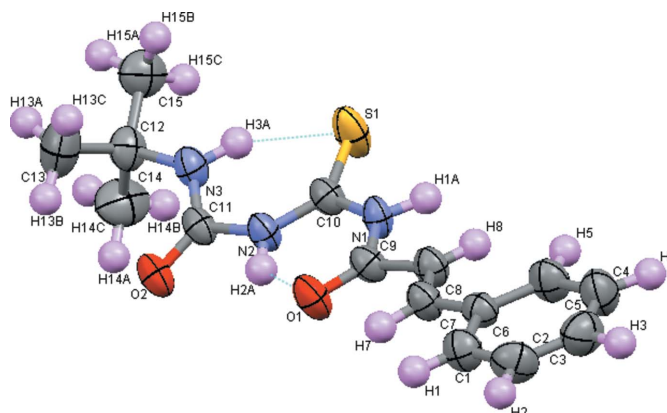


Figure 1

The structure of the title compound, showing the atom-numbering scheme. Intramolecular hydrogen bonds are shown as dashed lines. Displacement ellipsoids are drawn at the 50% probability level.

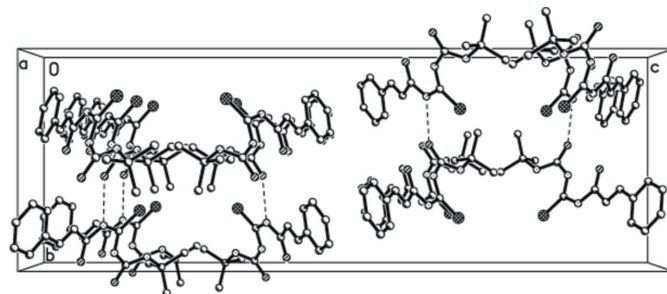


Figure 2

The crystal packing of (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT* and *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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