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1-*tert*-Butyl-2-(4-ethylbenzoyl)-1-(2-mercapto-3,5-dimethylbenzoyl)hydrazine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.060
wR factor = 0.176
Data-to-parameter ratio = 17.5

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

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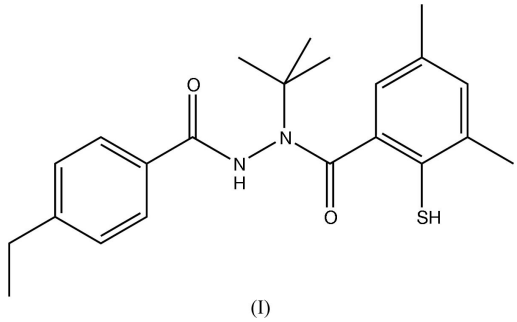
In the title compound, $C_{22}H_{28}N_2O_2S$, the carbonyl group closer to the thiol group is almost perpendicular to the attached benzene ring. The crystal packing is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds.

Comment

The 1-*tert*-butyl-1,2-diacylhydrazines, a new class of insect-growth regulators, have been found to mimic the action of 20-hydroxyecdysone to activate the ecdysone receptor, leading to lethal premature moulting (Wing, 1988, 1995; Wing *et al.*, 1988). Among nonsteroidal ecdysone agonists, 1-*tert*-butyl-2-(4-ethylbenzoyl)-1-(3,5-dimethylbenzoyl)hydrazine (tebufenozide, RH-5992) has been the first to be commercialized as a lepidopteran-specific insecticide, with a low toxicity profile towards mammals, birds and fishes, as well as towards non-target arthropods such as insect pollinators, predators, and parasitoids (Dhadialla & Jansson, 1999). At present, another three new structural analogues, namely methoxyfenozide (RH-2485), halofenozide (RH-0345) and chromafenozide (ANS-118), have been brought on the market (Carlson *et al.*, 2001; Yanagi *et al.*, 2000). Therefore, in a search for new insect-growth regulators with improved biological properties and a different activity spectrum, we synthesized the title compound, (I) (Fig. 1).



(I)

In (I), all bond lengths and angles (Table 1) are normal (Allen *et al.*, 1987). In the molecule, the carbonyl group closer to the thiol group is almost perpendicular to the attached benzene ring; the torsion angle O1—C1—C2—C3 is 73.8 (4)° (Table 1). This unusual conformation is mainly caused by the steric bulk of the *tert*-butyl group attached to atom N1. The crystal packing (Fig. 2) is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds (Table 2).

Experimental

To a stirred solution of sulfur dichloride (0.08 mol) and dichloromethane (15 ml) was added a solution of pyridine (0.008 mol) in

dichloromethane (5 ml) dropwise at 263 K. A solution of 1-*tert*-butyl-1-(3,5-dimethylbenzoyl)-2-(4-ethylbenzoyl)hydrazine (0.007 mol) in dichloromethane (5 ml) was then added at 263 K. The mixture was stirred at room temperature for 4 h and then poured onto ice. The solid was then filtered off and the filtrate was concentrated under vacuum. The residue was purified by column chromatography on silica gel using petroleum ether (60–90), dichloromethane and ethyl acetate (20:1:1 by volume) as the eluents..

Crystal data

$C_{22}H_{28}N_2O_2S$	D_x = 1.181 Mg m^{-3}
M_r = 384.52	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1021 reflections
a = 11.656 (3) Å	θ = 3.0–21.6°
b = 9.960 (3) Å	μ = 0.17 mm^{-1}
c = 18.796 (5) Å	T = 293 (2) K
β = 97.776 (5)°	Block, colourless
V = 2162.1 (10) Å^3	0.30 × 0.24 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4433 independent reflections
	2103 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.057
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	θ_{max} = 26.4°
	$h = -14 \rightarrow 14$
T_{min} = 0.938, T_{max} = 0.970	$k = -12 \rightarrow 4$
12 135 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.083P)^2 + 0.0808P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.060	
$wR(F^2)$ = 0.176	
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.001
4433 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
254 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.347 (3)	N2—C14	1.365 (3)
N1—N2	1.388 (3)		
C1—N1—N2—C14	−96.5 (3)	N1—N2—C14—O2	5.6 (4)
N2—N1—C1—O1	−177.1 (2)	O2—C14—C15—C16	176.2 (3)
O1—C1—C2—C3	73.7 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.87 (1)	2.053 (13)	2.893 (3)	162 (3)
C16—H16···O1^{i}	0.93	2.44	3.346 (3)	166
C20—H20···O2^{ii}	0.93	2.51	3.315 (4)	145

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

All C-bound H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). Atom H1, attached to S1, and H2, attached to N2, were located in a difference Fourier map. Atom H2 was refined freely, while for H1 the riding model was used with H1—S1 = 1.0 Å and U_{iso}(H1) = 1.5U_{eq}(S1).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

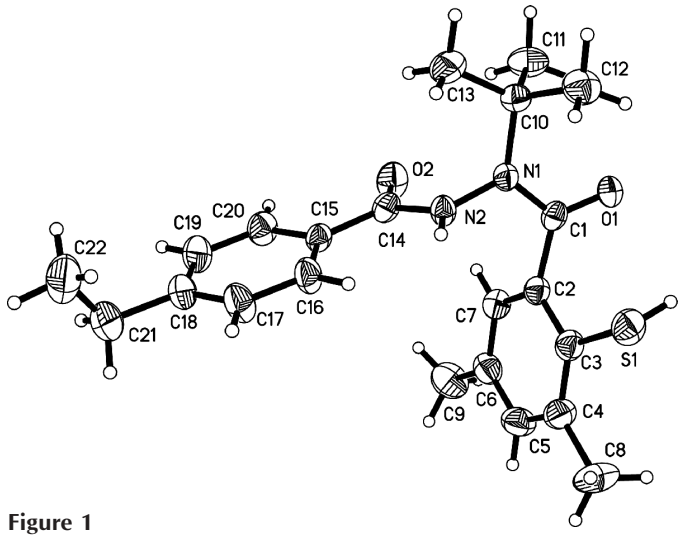


Figure 1
View of (I), with displacement ellipsoids drawn at the 30% probability level.

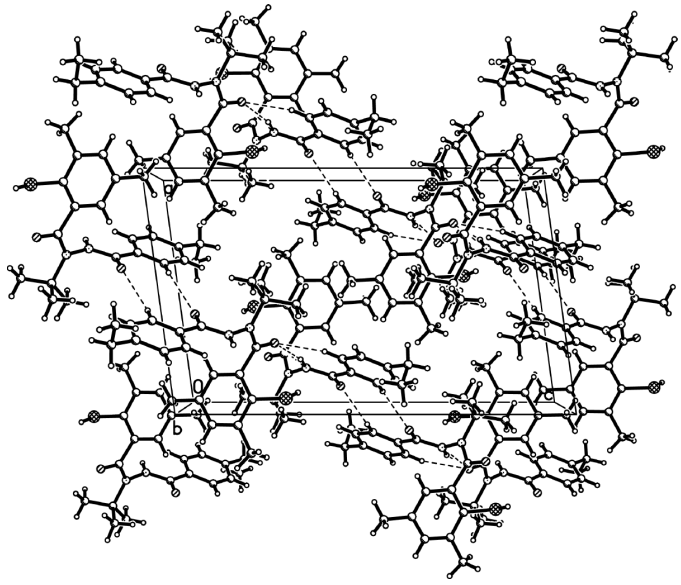


Figure 2
Packing diagram of (I), showing the intermolecular hydrogen bonds as dashed lines.

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