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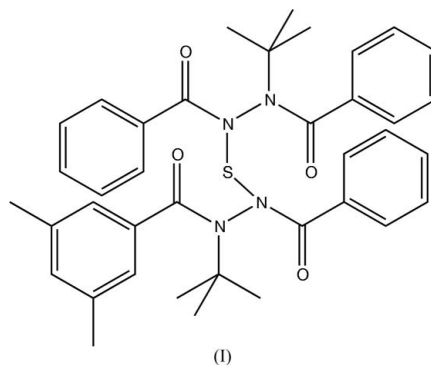
## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(C-C) = 0.004$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.137  
Data-to-parameter ratio = 16.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.2-Benzoyl-1-*tert*-butyl-2-(1,2-dibenzoyl-2-*tert*-butylhydrazinosulfanyl)-1-(3,5-dimethylbenzoyl)hydrazine

The crystal structure of the title compound,  $C_{38}H_{42}N_4O_4S$ , an insect-growth regulator, has been determined. The two *tert*-butyl groups are in different environments and this is reflected in the  $^1H$  NMR spectrum.

## Comment

The diacylhydrazines are a promising class of chemically and mechanistically novel insect control agents that were first discovered and characterized by the Rohm and Haas Company in the mid-1980s (Wing, 1995, 1988; Wing *et al.*, 1988). The first member of this class was RH-5849, which had interesting foliar- and root-systemic insecticidal activity against a range of larval lepidopteran, coleopteran, and dipteran pests, but was eventually abandoned in favor of other more commercially attractive analogs (Dhadialla & Jansson, 1999). At present, another three new structural analogs: methoxyfenozide (RH-2485), halofenozide (RH-0345) and chromafenozide (ANS-118) have already been brought to the market (Carlson *et al.*, 2001; Yanagi *et al.*, 2000). It has been reported that biscarbamoyl sulfide derivatives of methylcarbamate insecticides retain the insecticidal activity of the parent methyl carbamate but are substantially less toxic to the white mouse (Fahmy *et al.*, 1974, 1978). It also has been reported that bis(1-*tert*-butyl-1,2-dibenzoylhydrazino) sulfide retains the insecticidal activity (Drabek, 1990).



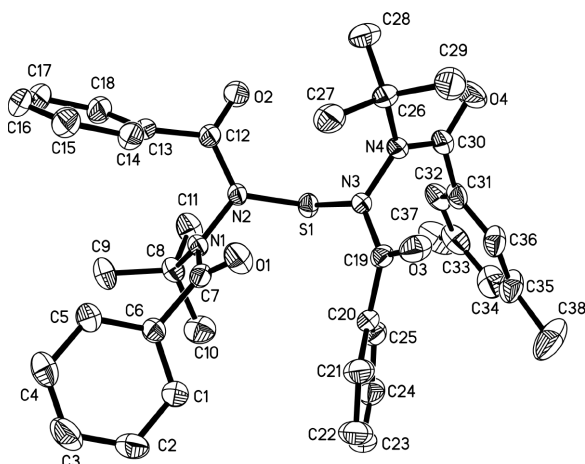
In a search for new insect-growth regulators with improved biological properties and a different activity spectrum, we began extensive synthesis-screening and studies of structure-activity relationships on bis(1-*tert*-butyl-1,2-diacylhydrazino) sulfide. The structure of the title compound, 2-benzoyl-1-*tert*-butyl-2-(2-*tert*-butyl-1,2-dibenzoylhydrazinosulfanyl)-1-(3,5-dimethylbenzoyl)hydrazine, (I), was therefore determined (Fig. 1).

In (I), the four carbonyl groups are not coplanar with their adjacent benzene rings. The  $C5-C6-C7-O1$ ,  $O2-C12-$

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**Figure 1**  
View of the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted.

C13—C14, O3—C19—C20—C21 and O4—C30—C31—C32 torsion angles are 97.6 (3),  $-113.5$  (3), 35.6 (3) and  $-75.5$  (3) $^\circ$ , respectively. The two *tert*-butyl groups are in different environments as the two nearest aromatic rings are not the same, one (nearest to C26) having two methyl substituents; this is reflected in its  $^1\text{H}$  NMR spectrum. While the four amide functions adopt the expected planar structure, their conformations are not identical. Of the two S-bound amide groups, one has a *cis* conformation [N4—N3—C19—O3 =  $-23.3$  (3) $^\circ$ ] and the other has a *trans* conformation [N1—N2—C12—O2 =  $-160.5$  (2) $^\circ$ ]. Similarly, the two amide groups bearing the *N*-*tert*-butyl groups have one with a *cis* conformation [N2—N1—C7—O1 =  $-2.6$  (3) $^\circ$ ] and the other with a *trans* conformation [N3—N4—C30—O4 =  $-170.3$  (2) $^\circ$ ], considerably different from the parent compound, 1,2-dibenzoyl-1-*tert*-butylhydrazine (Chan *et al.*, 1990).

## Experimental

To a stirred admixture of 1-*tert*-butyl-1-(3,5-dimethylbenzoyl)-2-benzoylhydrazine (0.006 mol) in anhydrous xylene (40 ml) was added sodium hydride (0.007 mol) over a period of 5 min. The mixture was stirred at the boiling temperature for 2 h and then cooled to 263 K. Then a chlorosulfonyl(1-*tert*-butyl-1,2-dibenzoylhydrazine) solution was added dropwise. After the addition was complete, the reaction mixture was stirred for 6 h at room temperature. 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 *v/v*) as the eluent. It was recrystallized from a solution of isopropyl alcohol by slow evaporation at room temperature.

### Crystal data

$\text{C}_{38}\text{H}_{42}\text{N}_4\text{O}_4\text{S}$   
 $M_r = 650.82$   
Monoclinic,  $P2_1/c$   
 $a = 15.641$  (2) Å  
 $b = 14.2886$  (18) Å  
 $c = 17.112$  (2) Å  
 $\beta = 112.678$  (2) $^\circ$   
 $V = 3528.5$  (8) Å $^3$   
 $Z = 4$

$D_x = 1.225$  Mg m $^{-3}$   
Mo  $K\alpha$  radiation  
Cell parameters from 3695 reflections  
 $\theta = 2.6$ – $21.2$  $^\circ$   
 $\mu = 0.14$  mm $^{-1}$   
 $T = 293$  (2) K  
Block, colorless  
 $0.24 \times 0.20 \times 0.16$  mm

### Data collection

Bruker SMART1000 CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.831$ ,  $T_{\max} = 0.980$   
20399 measured reflections

7258 independent reflections  
3786 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.049$   
 $\theta_{\max} = 26.4$  $^\circ$   
 $h = -19 \rightarrow 13$   
 $k = -15 \rightarrow 17$   
 $l = -20 \rightarrow 21$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.137$   
 $S = 1.04$   
7258 reflections  
432 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.18$  e Å $^{-3}$   
 $\Delta\rho_{\min} = -0.22$  e Å $^{-3}$

**Table 1**

Selected geometric parameters (Å,  $^\circ$ ).

S1—N3	1.6846 (19)	N1—N2	1.408 (2)
S1—N2	1.7029 (17)	N3—N4	1.411 (2)
N3—S1—N2	108.57 (9)	N4—N3—C19	115.68 (17)
C7—N1—N2	113.31 (17)	N4—N3—S1	118.39 (13)
C7—N1—C8	127.74 (18)	C19—N3—S1	125.36 (15)
N2—N1—C8	116.78 (16)	C30—N4—N3	115.31 (18)
C12—N2—N1	117.01 (16)	C30—N4—C26	122.21 (19)
C12—N2—S1	120.88 (14)	N3—N4—C26	122.48 (17)
N1—N2—S1	119.30 (13)		
N2—N1—C7—O1	$-2.6$ (3)	N4—N3—C19—O3	23.0 (3)
C5—C6—C7—O1	97.6 (3)	O3—C19—C20—C21	35.6 (3)
N1—N2—C12—O2	$-160.5$ (2)	N3—N4—C30—O4	170.3 (2)
O2—C12—C13—C14	$-113.5$ (3)	O4—C30—C31—C32	$-75.5$ (3)

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

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.

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