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2-[(3,5-Di-*tert*-butyl-4-hydroxybenzyl)-sulfanyl]-*N*′-isopropylideneaceto-hydrazide

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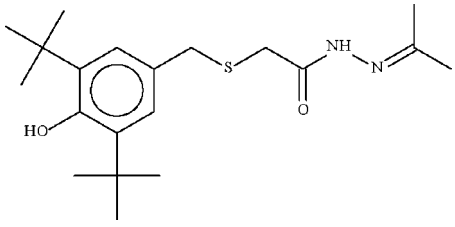
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.135; data-to-parameter ratio = 20.6.

The title compound, $C_{20}H_{32}N_2O_2S$, the condensation product of a thioacetohydrazine and acetone, has a two-coordinate S atom and the angle at this atom is 100.7 (1)°. The $(CH_3)C{=}N{-}NH{-}C(O)$– substituent engages in N—H···O hydrogen-bonding interactions with the substituent of an adjacent molecule across a center of inversion, generating a dimeric structure.

Related literature

There are several structural studies of $(CH_3)C{=}N{-}NH{-}C(O)$–*X* compounds; for *N*-acetyl-*N*′-isopropylidenehydrazine, see: Khusainova *et al.* (2004). For the synthesis of the thioacetohydrazine reactant, see: MacLeay & Meyers (1989); Myers & MacLeay (1989).



Experimental

Crystal data

$C_{20}H_{32}N_2O_2S$
M_r = 364.54
Monoclinic, $C2/c$
a = 30.8643 (10) Å
b = 10.0128 (3) Å
c = 13.9596 (5) Å
β = 96.227 (2)°
V = 4288.6 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.17 mm^{-1}
T = 100 K
0.25 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.960, T_{max} = 0.984
14652 measured reflections
4886 independent reflections
3240 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.135
S = 1.05
4886 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.88	2.10	2.940 (2)	159

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2379).

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supporting information

Acta Cryst. (2009). E**65**, o730 [doi:10.1107/S1600536809007843]

2-[(3,5-Di-*tert*-butyl-4-hydroxybenzyl)sulfanyl]-*N*′-isopropylideneacetohydrazide

Wagee A. Yehye, Azhar Ariffin and Seik Weng Ng

S1. Experimental

2-(3,5-Di-*tert*-butyl-4-hydroxybenzylthio)acetohydrazine (0.5 g, 1.54 mmol) and acetone (10 ml) were heated for 6 h; several drops of acetic acid were added to the reaction. The solvent was removed and the product recrystallized from hexane.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.95–0.99 Å) and were included in the refinement in the riding model approximation with *U*(H) set to 1.2–1.5*U*(C). The oxygen- and nitrogen-bound H-atoms were similarly treated (O–H 0.84 and N–H 0.88 Å).

The hydroxy H-atom does not form a hydrogen bond; it is probably disordered over several positions. In one position, it is less than 2 Å from a hydrogen atom of the C14 methyl group. The two *tert*-butyl groups are probably also disordered, but the disorder could not be resolved into multiple positions.

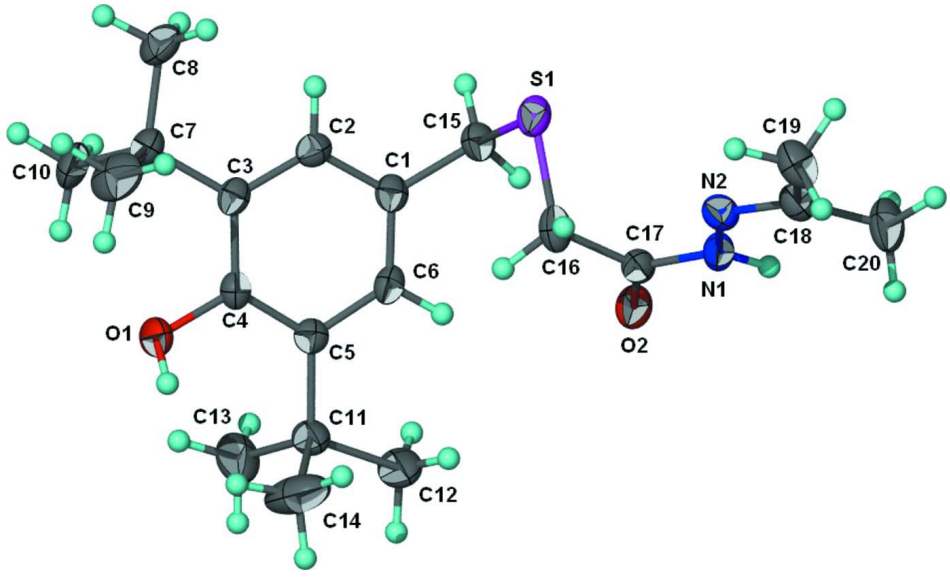

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{20}H_{32}N_2O_2S$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radii.

2-[(3,5-Di-*tert*-butyl-4-hydroxybenzyl)sulfanyl]-*N*′- isopropylideneacetohydrazide

Crystal data

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$c = 13.9596\ (5)$ Å
$\beta = 96.227\ (2)°$
$V = 4288.6\ (2)$ Å^3
$Z = 8$
$F(000) = 1584$
$D_x = 1.129$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1963 reflections
$\theta = 2.6–26.3°$
$\mu = 0.17$ mm^{-1}
$T = 100$ K
Block, colorless
0.25 × 0.15 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.960$, $T_{max} = 0.984$
14652 measured reflections
4886 independent reflections
3240 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.3°$
$h = -39\rightarrow40$
$k = -13\rightarrow13$
$l = -18\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.135$
$S = 1.05$
4886 reflections
237 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2 + 0.8959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.394869 (18)	0.18708 (5)	0.52819 (4)	0.02810 (16)
O1	0.32415 (5)	0.04431 (14)	0.08151 (10)	0.0287 (4)
H1O	0.3479	0.0304	0.0591	0.066 (10)*
O2	0.45752 (5)	0.41852 (14)	0.43360 (10)	0.0284 (4)
N1	0.49822 (5)	0.32105 (16)	0.55791 (12)	0.0228 (4)
H1	0.5109	0.3970	0.5762	0.033 (6)*
N2	0.51037 (5)	0.20268 (16)	0.60634 (12)	0.0233 (4)
C1	0.34695 (6)	0.22530 (19)	0.35011 (14)	0.0212 (4)
C2	0.31851 (6)	0.11801 (18)	0.33503 (15)	0.0208 (4)
H2	0.3045	0.0851	0.3876	0.025*
C3	0.30992 (6)	0.05737 (18)	0.24556 (15)	0.0205 (4)
C4	0.33196 (6)	0.10754 (18)	0.17019 (14)	0.0207 (4)
C5	0.35919 (6)	0.21967 (19)	0.18074 (14)	0.0206 (4)

C6	0.36638 (6)	0.27540 (19)	0.27299 (14)	0.0213 (4)
H6	0.3853	0.3502	0.2830	0.026*
C7	0.27708 (7)	−0.0579 (2)	0.22932 (15)	0.0257 (5)
C8	0.25474 (7)	−0.0877 (2)	0.31963 (16)	0.0326 (5)
H8A	0.2394	−0.0077	0.3383	0.049*
H8B	0.2767	−0.1138	0.3723	0.049*
H8C	0.2338	−0.1606	0.3061	0.049*
C9	0.30019 (9)	−0.1865 (2)	0.2033 (2)	0.0425 (6)
H9A	0.2790	−0.2594	0.1946	0.064*
H9B	0.3229	−0.2093	0.2554	0.064*
H9C	0.3135	−0.1729	0.1435	0.064*
C10	0.24093 (7)	−0.0210 (2)	0.14912 (17)	0.0360 (6)
H10A	0.2260	0.0600	0.1674	0.054*
H10B	0.2199	−0.0945	0.1398	0.054*
H10C	0.2538	−0.0049	0.0890	0.054*
C11	0.38057 (7)	0.2813 (2)	0.09618 (16)	0.0284 (5)
C12	0.40558 (9)	0.4089 (2)	0.12623 (17)	0.0404 (6)
H12A	0.4293	0.3879	0.1764	0.061*
H12B	0.3858	0.4734	0.1513	0.061*
H12C	0.4177	0.4472	0.0703	0.061*
C13	0.34528 (10)	0.3206 (3)	0.01478 (18)	0.0480 (7)
H13A	0.3255	0.3861	0.0390	0.072*
H13B	0.3287	0.2410	−0.0078	0.072*
H13C	0.3591	0.3596	−0.0387	0.072*
C14	0.41348 (10)	0.1852 (2)	0.0594 (2)	0.0544 (8)
H14A	0.4342	0.1558	0.1135	0.082*
H14B	0.4292	0.2307	0.0116	0.082*
H14C	0.3981	0.1075	0.0297	0.082*
C15	0.35698 (7)	0.2863 (2)	0.44856 (14)	0.0242 (5)
H15A	0.3295	0.2968	0.4783	0.029*
H15B	0.3695	0.3764	0.4417	0.029*
C16	0.44235 (7)	0.18946 (19)	0.46239 (16)	0.0258 (5)
H16A	0.4615	0.1128	0.4823	0.031*
H16B	0.4332	0.1816	0.3924	0.031*
C17	0.46677 (6)	0.3174 (2)	0.48258 (14)	0.0221 (4)
C18	0.54119 (7)	0.2084 (2)	0.67522 (15)	0.0252 (5)
C19	0.55491 (8)	0.0790 (2)	0.72261 (16)	0.0328 (5)
H19A	0.5361	0.0070	0.6946	0.049*
H19B	0.5524	0.0852	0.7919	0.049*
H19C	0.5852	0.0600	0.7124	0.049*
C20	0.56467 (8)	0.3315 (2)	0.71190 (17)	0.0376 (6)
H20A	0.5435	0.4023	0.7203	0.056*
H20B	0.5844	0.3607	0.6655	0.056*
H20C	0.5816	0.3124	0.7739	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0292 (3)	0.0299 (3)	0.0231 (3)	−0.0090 (2)	−0.0063 (2)	0.0075 (2)
O1	0.0321 (9)	0.0280 (8)	0.0264 (9)	−0.0086 (7)	0.0057 (7)	−0.0100 (6)
O2	0.0270 (8)	0.0279 (8)	0.0286 (9)	−0.0029 (6)	−0.0044 (7)	0.0037 (6)
N1	0.0219 (9)	0.0237 (8)	0.0219 (9)	−0.0034 (7)	−0.0018 (7)	−0.0016 (7)
N2	0.0237 (9)	0.0257 (9)	0.0203 (9)	0.0017 (7)	0.0019 (7)	0.0006 (7)
C1	0.0188 (10)	0.0232 (9)	0.0207 (11)	0.0016 (8)	−0.0024 (8)	0.0014 (8)
C2	0.0174 (10)	0.0224 (9)	0.0223 (11)	0.0006 (8)	0.0009 (8)	0.0044 (8)
C3	0.0170 (10)	0.0193 (9)	0.0243 (11)	0.0003 (8)	−0.0015 (8)	0.0014 (8)
C4	0.0200 (10)	0.0199 (9)	0.0214 (11)	−0.0004 (8)	−0.0012 (8)	−0.0024 (8)
C5	0.0208 (10)	0.0201 (9)	0.0208 (11)	0.0006 (8)	0.0015 (8)	0.0007 (8)
C6	0.0190 (10)	0.0193 (9)	0.0244 (11)	−0.0026 (8)	−0.0031 (8)	0.0013 (8)
C7	0.0237 (11)	0.0250 (10)	0.0282 (12)	−0.0071 (9)	0.0023 (9)	−0.0018 (9)
C8	0.0303 (12)	0.0305 (11)	0.0373 (14)	−0.0113 (10)	0.0048 (10)	0.0031 (10)
C9	0.0496 (16)	0.0223 (11)	0.0580 (18)	−0.0087 (11)	0.0172 (13)	−0.0045 (11)
C10	0.0269 (12)	0.0433 (13)	0.0359 (14)	−0.0155 (11)	−0.0046 (10)	−0.0016 (11)
C11	0.0367 (13)	0.0260 (11)	0.0232 (12)	−0.0095 (9)	0.0066 (10)	0.0000 (9)
C12	0.0553 (16)	0.0360 (13)	0.0310 (14)	−0.0208 (12)	0.0096 (12)	0.0026 (10)
C13	0.0673 (19)	0.0478 (15)	0.0261 (14)	−0.0202 (14)	−0.0072 (13)	0.0108 (11)
C14	0.0605 (18)	0.0398 (14)	0.071 (2)	−0.0102 (13)	0.0424 (16)	−0.0037 (14)
C15	0.0231 (11)	0.0274 (10)	0.0214 (11)	−0.0001 (9)	−0.0013 (9)	0.0000 (8)
C16	0.0252 (11)	0.0233 (10)	0.0270 (12)	0.0028 (9)	−0.0065 (9)	−0.0036 (9)
C17	0.0181 (10)	0.0275 (10)	0.0207 (11)	−0.0004 (8)	0.0024 (8)	−0.0024 (9)
C18	0.0276 (11)	0.0279 (11)	0.0199 (11)	0.0001 (9)	0.0018 (9)	−0.0020 (8)
C19	0.0395 (13)	0.0323 (12)	0.0253 (13)	0.0048 (10)	−0.0031 (10)	−0.0029 (9)
C20	0.0441 (14)	0.0353 (12)	0.0294 (13)	−0.0072 (11)	−0.0145 (11)	0.0031 (10)

Geometric parameters (Å, º)

S1—C16	1.812 (2)	C10—H10A	0.9800
S1—C15	1.819 (2)	C10—H10B	0.9800
O1—C4	1.388 (2)	C10—H10C	0.9800
O1—H1O	0.8400	C11—C14	1.528 (3)
O2—C17	1.237 (2)	C11—C12	1.527 (3)
N1—C17	1.352 (2)	C11—C13	1.537 (3)
N1—N2	1.396 (2)	C12—H12A	0.9800
N1—H1	0.8800	C12—H12B	0.9800
N2—C18	1.278 (3)	C12—H12C	0.9800
C1—C6	1.382 (3)	C13—H13A	0.9800
C1—C2	1.389 (3)	C13—H13B	0.9800
C1—C15	1.505 (3)	C13—H13C	0.9800
C2—C3	1.388 (3)	C14—H14A	0.9800
C2—H2	0.9500	C14—H14B	0.9800
C3—C4	1.406 (3)	C14—H14C	0.9800
C3—C7	1.536 (3)	C15—H15A	0.9900
C4—C5	1.401 (3)	C15—H15B	0.9900

C5—C6	1.399 (3)	C16—C17	1.498 (3)
C5—C11	1.542 (3)	C16—H16A	0.9900
C6—H6	0.9500	C16—H16B	0.9900
C7—C8	1.531 (3)	C18—C20	1.493 (3)
C7—C9	1.535 (3)	C18—C19	1.495 (3)
C7—C10	1.537 (3)	C19—H19A	0.9800
C8—H8A	0.9800	C19—H19B	0.9800
C8—H8B	0.9800	C19—H19C	0.9800
C8—H8C	0.9800	C20—H20A	0.9800
C9—H9A	0.9800	C20—H20B	0.9800
C9—H9B	0.9800	C20—H20C	0.9800
C9—H9C	0.9800		

C16—S1—C15	100.65 (10)	C14—C11—C5	111.09 (18)
C4—O1—H1O	109.5	C12—C11—C5	111.70 (17)
C17—N1—N2	119.11 (16)	C13—C11—C5	109.91 (19)
C17—N1—H1	120.4	C11—C12—H12A	109.5
N2—N1—H1	120.4	C11—C12—H12B	109.5
C18—N2—N1	117.74 (17)	H12A—C12—H12B	109.5
C6—C1—C2	118.89 (18)	C11—C12—H12C	109.5
C6—C1—C15	120.02 (18)	H12A—C12—H12C	109.5
C2—C1—C15	121.09 (18)	H12B—C12—H12C	109.5
C3—C2—C1	122.04 (19)	C11—C13—H13A	109.5
C3—C2—H2	119.0	C11—C13—H13B	109.5
C1—C2—H2	119.0	H13A—C13—H13B	109.5
C2—C3—C4	117.15 (17)	C11—C13—H13C	109.5
C2—C3—C7	121.32 (18)	H13A—C13—H13C	109.5
C4—C3—C7	121.52 (17)	H13B—C13—H13C	109.5
O1—C4—C5	120.24 (17)	C11—C14—H14A	109.5
O1—C4—C3	116.94 (17)	C11—C14—H14B	109.5
C5—C4—C3	122.76 (18)	H14A—C14—H14B	109.5
C6—C5—C4	116.74 (18)	C11—C14—H14C	109.5
C6—C5—C11	120.44 (17)	H14A—C14—H14C	109.5
C4—C5—C11	122.82 (17)	H14B—C14—H14C	109.5
C1—C6—C5	122.24 (18)	C1—C15—S1	113.01 (14)
C1—C6—H6	118.9	C1—C15—H15A	109.0
C5—C6—H6	118.9	S1—C15—H15A	109.0
C8—C7—C9	107.06 (18)	C1—C15—H15B	109.0
C8—C7—C3	111.95 (17)	S1—C15—H15B	109.0
C9—C7—C3	110.44 (17)	H15A—C15—H15B	107.8
C8—C7—C10	106.88 (18)	C17—C16—S1	109.51 (14)
C9—C7—C10	110.41 (19)	C17—C16—H16A	109.8
C3—C7—C10	110.01 (17)	S1—C16—H16A	109.8
C7—C8—H8A	109.5	C17—C16—H16B	109.8
C7—C8—H8B	109.5	S1—C16—H16B	109.8
H8A—C8—H8B	109.5	H16A—C16—H16B	108.2
C7—C8—H8C	109.5	O2—C17—N1	120.74 (18)
H8A—C8—H8C	109.5	O2—C17—C16	120.97 (18)

H8B—C8—H8C	109.5	N1—C17—C16	118.26 (18)
C7—C9—H9A	109.5	N2—C18—C20	126.07 (19)
C7—C9—H9B	109.5	N2—C18—C19	116.51 (19)
H9A—C9—H9B	109.5	C20—C18—C19	117.41 (19)
C7—C9—H9C	109.5	C18—C19—H19A	109.5
H9A—C9—H9C	109.5	C18—C19—H19B	109.5
H9B—C9—H9C	109.5	H19A—C19—H19B	109.5
C7—C10—H10A	109.5	C18—C19—H19C	109.5
C7—C10—H10B	109.5	H19A—C19—H19C	109.5
H10A—C10—H10B	109.5	H19B—C19—H19C	109.5
C7—C10—H10C	109.5	C18—C20—H20A	109.5
H10A—C10—H10C	109.5	C18—C20—H20B	109.5
H10B—C10—H10C	109.5	H20A—C20—H20B	109.5
C14—C11—C12	106.5 (2)	C18—C20—H20C	109.5
C14—C11—C13	110.9 (2)	H20A—C20—H20C	109.5
C12—C11—C13	106.61 (18)	H20B—C20—H20C	109.5

C17—N1—N2—C18	177.73 (18)	C4—C3—C7—C9	65.2 (3)
C6—C1—C2—C3	1.8 (3)	C2—C3—C7—C10	122.0 (2)
C15—C1—C2—C3	−177.81 (18)	C4—C3—C7—C10	−56.9 (2)
C1—C2—C3—C4	1.2 (3)	C6—C5—C11—C14	114.5 (2)
C1—C2—C3—C7	−177.73 (18)	C4—C5—C11—C14	−65.8 (3)
C2—C3—C4—O1	178.34 (17)	C6—C5—C11—C12	−4.3 (3)
C7—C3—C4—O1	−2.8 (3)	C4—C5—C11—C12	175.47 (19)
C2—C3—C4—C5	−4.5 (3)	C6—C5—C11—C13	−122.4 (2)
C7—C3—C4—C5	174.38 (18)	C4—C5—C11—C13	57.3 (3)
O1—C4—C5—C6	−178.28 (17)	C6—C1—C15—S1	−102.63 (19)
C3—C4—C5—C6	4.7 (3)	C2—C1—C15—S1	77.0 (2)
O1—C4—C5—C11	1.9 (3)	C16—S1—C15—C1	61.45 (16)
C3—C4—C5—C11	−175.14 (19)	C15—S1—C16—C17	81.49 (15)
C2—C1—C6—C5	−1.6 (3)	N2—N1—C17—O2	−174.54 (18)
C15—C1—C6—C5	178.00 (18)	N2—N1—C17—C16	7.7 (3)
C4—C5—C6—C1	−1.5 (3)	S1—C16—C17—O2	−87.7 (2)
C11—C5—C6—C1	178.29 (18)	S1—C16—C17—N1	89.99 (19)
C2—C3—C7—C8	3.3 (3)	N1—N2—C18—C20	2.5 (3)
C4—C3—C7—C8	−175.55 (18)	N1—N2—C18—C19	−177.17 (17)
C2—C3—C7—C9	−115.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.88	2.10	2.940 (2)	159

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