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4-Ethyl-1-(4-methylbenzylidene)thiosemicarbazide

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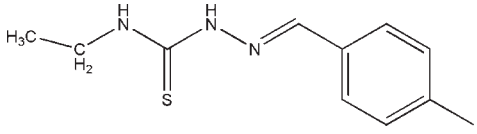
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.144; data-to-parameter ratio = 20.0.

In the title compound, $C_{11}H_{15}N_3S$, an intramolecular N—H···N hydrogen bond generates an S(5) ring. In the crystal, inversion dimers linked by pairs of N—H···S bonds occur, generating an $R_2^2(8)$ loop.

Related literature

For a related structure, see: Li & Jian (2010).



Experimental

Crystal data

$C_{11}H_{15}N_3S$
M_r = 221.32
Monoclinic, $P2_1/c$
a = 8.5777 (17) Å
b = 13.620 (3) Å
c = 10.364 (2) Å
β = 90.00 (3)°
V = 1210.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.23 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
11225 measured reflections
2724 independent reflections
1811 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.144
S = 1.10
2724 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···S1^{i}	0.86	2.69	3.505 (2)	158
N3—H3A···N1	0.86	2.21	2.605 (2)	108

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5448).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* E**66**, o1397.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2010). E**66**, o1399 [**doi:10.1107/S1600536810017988**]

4-Ethyl-1-(4-methylbenzylidene)thiosemicarbazide

Y.-F. Li and F.-F. Jian

Experimental

A mixture of 4-methylbenzaldehyde (0.10 mol), and 4-ethylthiosemicarbazide (0.10 mol) was stirred in refluxing ethanol (10 ml) for 4 h to afford the title compound (0.078 mol, yield 78%). Colourless blocks of (I) were obtained by recrystallization from ethanol at room temperature.

Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93-0.97 Å; N—H = 0.86Å and with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C_{methyl}).

Figures

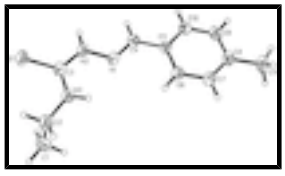


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids.

4-Ethyl-1-(4-methylbenzylidene)thiosemicarbazide

Crystal data

$C_{11}H_{15}N_3S$	$F(000)$ = 472
M_r = 221.32	D_x = 1.214 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1811 reflections
a = 8.5777 (17) Å	θ = 3.7–25.4°
b = 13.620 (3) Å	μ = 0.24 mm^{-1}
c = 10.364 (2) Å	*T* = 293 K
β = 90.00 (3)°	Block, colorless
V = 1210.8 (4) Å^3	0.23 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker SMART CCD diffractometer	1811 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.035
graphite	θ_{max} = 27.5°, θ_{min} = 3.4°

phi and ω scans	$h = -10 \rightarrow 11$
11225 measured reflections	$k = -17 \rightarrow 17$
2724 independent reflections	$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.045$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.144$	H-atom parameters constrained
$S = 1.10$	$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2 + 0.0957P]$ where $P = (F_o^2 + 2F_c^2)/3$
2724 reflections	$(\Delta/\sigma)_{max} = 0.001$
136 parameters	$\Delta\rho_{max} = 0.22$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	0.18659 (7)	0.61350 (4)	0.05705 (5)	0.0646 (2)
N2	0.02730 (19)	0.59199 (13)	−0.15745 (14)	0.0517 (4)
H2A	−0.0314	0.5527	−0.1144	0.062*
N1	−0.00273 (18)	0.61045 (12)	−0.28521 (14)	0.0471 (4)
C5	−0.1597 (2)	0.57835 (14)	−0.47004 (17)	0.0454 (4)
C6	−0.0800 (2)	0.64307 (15)	−0.55008 (18)	0.0514 (5)
H6A	0.0041	0.6785	−0.5179	0.062*
C8	−0.2494 (2)	0.60447 (16)	−0.72820 (18)	0.0535 (5)
C4	−0.1162 (2)	0.56386 (15)	−0.33565 (18)	0.0494 (5)
H4A	−0.1724	0.5196	−0.2855	0.059*
C3	0.1500 (2)	0.63616 (15)	−0.09996 (17)	0.0486 (5)
N3	0.2320 (2)	0.69703 (14)	−0.17353 (16)	0.0586 (5)
H3A	0.1966	0.7095	−0.2494	0.070*
C7	−0.1245 (2)	0.65502 (17)	−0.67644 (18)	0.0556 (5)
H7A	−0.0691	0.6983	−0.7285	0.067*

C10	−0.2838 (2)	0.52628 (17)	−0.52182 (19)	0.0582 (5)
H10A	−0.3384	0.4819	−0.4706	0.070*
C9	−0.3270 (2)	0.53994 (18)	−0.6490 (2)	0.0618 (6)
H9A	−0.4107	0.5045	−0.6819	0.074*
C11	−0.2989 (3)	0.6213 (2)	−0.8660 (2)	0.0747 (7)
H11A	−0.3868	0.5803	−0.8857	0.112*
H11B	−0.2142	0.6054	−0.9228	0.112*
H11C	−0.3272	0.6890	−0.8774	0.112*
C2	0.3779 (3)	0.7445 (2)	−0.1356 (2)	0.0759 (7)
H2B	0.3760	0.7574	−0.0436	0.091*
H2C	0.3869	0.8071	−0.1798	0.091*
C1	0.5171 (3)	0.6822 (2)	−0.1673 (3)	0.0875 (9)
H1B	0.6104	0.7152	−0.1403	0.131*
H1C	0.5210	0.6710	−0.2587	0.131*
H1D	0.5089	0.6204	−0.1232	0.131*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0854 (4)	0.0693 (4)	0.0392 (3)	−0.0124 (3)	−0.0082 (2)	0.0033 (2)
N2	0.0574 (10)	0.0600 (11)	0.0377 (8)	−0.0058 (8)	−0.0014 (7)	0.0049 (7)
N1	0.0559 (9)	0.0484 (9)	0.0372 (8)	0.0019 (7)	−0.0015 (7)	0.0003 (6)
C5	0.0449 (10)	0.0467 (11)	0.0445 (9)	0.0024 (8)	−0.0011 (7)	−0.0003 (7)
C6	0.0552 (11)	0.0497 (11)	0.0493 (10)	−0.0063 (9)	−0.0042 (8)	0.0026 (8)
C8	0.0596 (12)	0.0557 (12)	0.0451 (10)	0.0138 (10)	−0.0061 (9)	−0.0064 (8)
C4	0.0509 (11)	0.0510 (12)	0.0463 (10)	−0.0008 (9)	0.0016 (8)	0.0047 (8)
C3	0.0561 (11)	0.0490 (11)	0.0406 (9)	0.0019 (9)	−0.0004 (8)	−0.0009 (8)
N3	0.0645 (10)	0.0658 (11)	0.0455 (8)	−0.0144 (9)	−0.0061 (7)	0.0078 (7)
C7	0.0654 (12)	0.0525 (12)	0.0488 (10)	0.0008 (10)	0.0009 (9)	0.0059 (8)
C10	0.0551 (11)	0.0631 (14)	0.0563 (11)	−0.0121 (10)	−0.0008 (9)	0.0036 (9)
C9	0.0572 (12)	0.0670 (15)	0.0611 (12)	−0.0059 (10)	−0.0120 (10)	−0.0078 (10)
C11	0.0885 (16)	0.0861 (19)	0.0494 (12)	0.0190 (14)	−0.0140 (11)	−0.0061 (11)
C2	0.0977 (18)	0.0733 (17)	0.0569 (13)	−0.0359 (15)	−0.0120 (12)	0.0062 (11)
C1	0.0677 (16)	0.095 (2)	0.099 (2)	−0.0246 (15)	−0.0174 (14)	0.0220 (15)

Geometric parameters (Å, °)

S1—C3	1.6858 (18)	N3—C2	1.462 (3)
N2—C3	1.351 (2)	N3—H3A	0.8600
N2—N1	1.372 (2)	C7—H7A	0.9300
N2—H2A	0.8600	C10—C9	1.381 (3)
N1—C4	1.274 (2)	C10—H10A	0.9300
C5—C10	1.388 (3)	C9—H9A	0.9300
C5—C6	1.390 (3)	C11—H11A	0.9600
C5—C4	1.455 (2)	C11—H11B	0.9600
C6—C7	1.374 (3)	C11—H11C	0.9600
C6—H6A	0.9300	C2—C1	1.502 (4)
C8—C9	1.375 (3)	C2—H2B	0.9700
C8—C7	1.381 (3)	C2—H2C	0.9700

C8—C11	1.508 (3)	C1—H1B	0.9600
C4—H4A	0.9300	C1—H1C	0.9600
C3—N3	1.328 (3)	C1—H1D	0.9600
C3—N2—N1	119.36 (16)	C9—C10—C5	120.4 (2)
C3—N2—H2A	120.3	C9—C10—H10A	119.8
N1—N2—H2A	120.3	C5—C10—H10A	119.8
C4—N1—N2	116.63 (16)	C8—C9—C10	121.75 (19)
C10—C5—C6	118.08 (17)	C8—C9—H9A	119.1
C10—C5—C4	119.85 (18)	C10—C9—H9A	119.1
C6—C5—C4	122.08 (17)	C8—C11—H11A	109.5
C7—C6—C5	120.48 (18)	C8—C11—H11B	109.5
C7—C6—H6A	119.8	H11A—C11—H11B	109.5
C5—C6—H6A	119.8	C8—C11—H11C	109.5
C9—C8—C7	117.52 (18)	H11A—C11—H11C	109.5
C9—C8—C11	121.8 (2)	H11B—C11—H11C	109.5
C7—C8—C11	120.7 (2)	N3—C2—C1	111.8 (2)
N1—C4—C5	121.40 (18)	N3—C2—H2B	109.3
N1—C4—H4A	119.3	C1—C2—H2B	109.3
C5—C4—H4A	119.3	N3—C2—H2C	109.3
N3—C3—N2	115.95 (16)	C1—C2—H2C	109.3
N3—C3—S1	124.73 (15)	H2B—C2—H2C	107.9
N2—C3—S1	119.30 (15)	C2—C1—H1B	109.5
C3—N3—C2	125.12 (17)	C2—C1—H1C	109.5
C3—N3—H3A	117.4	H1B—C1—H1C	109.5
C2—N3—H3A	117.4	C2—C1—H1D	109.5
C6—C7—C8	121.8 (2)	H1B—C1—H1D	109.5
C6—C7—H7A	119.1	H1C—C1—H1D	109.5
C8—C7—H7A	119.1		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···S1^{i}	0.86	2.69	3.505 (2)	158
N3—H3A···N1	0.86	2.21	2.605 (2)	108

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

Fig. 1