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Benzyl (*E*)-3-(4-methoxybenzylidene)-dithiocarbazate

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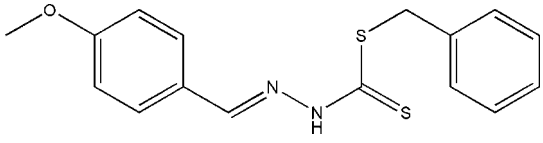
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.007 Å; R factor = 0.055; wR factor = 0.129; data-to-parameter ratio = 15.6.

The title compound, $C_{16}H_{16}N_2OS_2$, was obtained from a condensation reaction of benzyl dithiocarbazate and 4-methoxybenzaldehyde. In the molecule, the methoxyphenyl ring and dithiocarbazate fragment are located on opposite sides of the C=N double bond, showing an *E* configuration. The dithiocarbazate fragment is approximately planar (r.m.s. deviation = 0.0052 Å); its mean plane is oriented at dihedral angles of 8.19 (15) and 85.70 (13)°, respectively, to the methoxyphenyl and phenyl rings. Intermolecular N—H···S hydrogen bonds and weak C—H···π interactions are observed in the crystal structure.

Related literature

For applications of hydrazone and its derivatives in the biological field, see: Okabe *et al.* (1993); Hu *et al.* (2001). For related structures, see: Shan *et al.* (2008*a*,*b*). For the synthesis, see: Hu *et al.* (2001).



Experimental

Crystal data

$C_{16}H_{16}N_2OS_2$
M_r = 316.43
Monoclinic, $P2_1/c$
a = 10.267 (5) Å
b = 5.150 (2) Å
c = 31.686 (11) Å
β = 97.141 (5)°
V = 1662.4 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 294 K
0.32 × 0.25 × 0.23 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.84, T_{max} = 0.92
6025 measured reflections
2982 independent reflections
1869 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.129
S = 1.04
2982 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

Cg is the centroid of the C1–C6 benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···S1^{i}	0.86	2.59	3.397 (4)	158
C16—H16*C*···*Cg*^{ii}	0.96	2.83	3.671 (5)	147

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, y-1, z$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5352).

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supplementary materials

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Benzyl (*E*)-3-(4-methoxybenzylidene)dithiocarbazate

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Comment

Hydrazone and its derivatives have shown the potential application in the biological field (Okabe *et al.*, 1993; Hu *et al.*, 2001). As part of the ongoing investigation on anti-cancer compounds, the title compound has recently been prepared in our laboratory and its crystal structure is presented here.

In the molecules, the methoxylphenyl ring and dithiocarbazate fragment are located on the opposite sides of the C═N double bond, showing the E-configuration. The dithiocarbazate fragment is approximately planar [r.m.s deviation 0.0052 Å]; the mean plane of dithiocarbazate is oriented with respect to the methoxylphenyl and phenyl rings at 8.19 (15) and 85.70 (13)°, similar to those found in related structures (Shan *et al.* 2008*a*,b). Intermolecular N—H···S hydrogen bonding and weak C—H···π interaction are observed in the crystal structure (Table 1).

Experimental

Benzyl dithiocarbazate was synthesized as described previously (Hu *et al.*, 2001). Benzyl dithiocarbazate (0.40 g, 2 mmol) and 4-methoxybenzaldehyde (0.27 g, 2 mmol) were dissolved in ethanol (20 ml), then acetic acid (0.2 ml) was added to the ethanol solution with stirring. The mixture solution was refluxed for 6 h. After cooling to room temperature, microcrystals appeared. The microcrystals were separated from the solution and washed with cold water three times. Recrystallization was performed twice with absolute methanol to obtain colourless single crystals of the title compound.

Refinement

H atoms were placed in calculated positions with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,N) for the others.

Figures

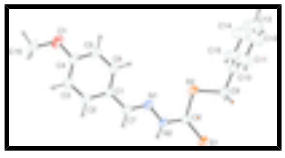

Fig. 1. The molecular structure of the title compound with 30% probability displacement (arbitrary spheres for H atoms).

Benzyl (*E*)-3-(4-methoxybenzylidene)dithiocarbazate

Crystal data

$C_{16}H_{16}N_2OS_2$	$F(000)$ = 664
M_r = 316.43	D_x = 1.264 Mg m^{-3}

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2982 reflections
$a = 10.267$ (5) Å	$\theta = 3.4–25.2°$
$b = 5.150$ (2) Å	$\mu = 0.32$ mm^{-1}
$c = 31.686$ (11) Å	$T = 294$ K
$\beta = 97.141$ (5)°	Block, colorless
$V = 1662.4$ (12) Å^3	0.32 × 0.25 × 0.23 mm
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	2982 independent reflections
Radiation source: fine-focus sealed tube	1869 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.035$
Detector resolution: 10.0 pixels mm^{-1}	$\theta_{max} = 25.2°$, $\theta_{min} = 3.5°$
ω scans	$h = -12\rightarrow10$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$k = -5\rightarrow6$
$T_{min} = 0.84$, $T_{max} = 0.92$	$l = -31\rightarrow37$
6025 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.055$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.129$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
2982 reflections	$(\Delta/\sigma)_{max} = 0.001$
191 parameters	$\Delta\rho_{max} = 0.32$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.28$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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	0.65124 (8)	0.64526 (18)	0.54959 (2)	0.0628 (3)
S2	0.57408 (9)	0.40404 (17)	0.63003 (2)	0.0631 (3)
O1	−0.0078 (2)	−0.7274 (5)	0.62062 (8)	0.0810 (7)
N1	0.4039 (2)	0.1129 (5)	0.57509 (7)	0.0545 (7)
N2	0.4768 (2)	0.2797 (5)	0.55381 (7)	0.0552 (7)
H2	0.4665	0.2815	0.5265	0.066*
C1	0.2443 (3)	−0.2240 (6)	0.56941 (8)	0.0491 (7)
C2	0.1678 (3)	−0.3916 (6)	0.54309 (9)	0.0582 (9)
H2A	0.1740	−0.3887	0.5141	0.070*
C3	0.0825 (3)	−0.5632 (6)	0.55848 (10)	0.0608 (8)
H3	0.0315	−0.6738	0.5400	0.073*
C4	0.0734 (3)	−0.5699 (6)	0.60140 (10)	0.0572 (8)
C5	0.1509 (3)	−0.4053 (7)	0.62824 (10)	0.0663 (9)
H5	0.1456	−0.4105	0.6573	0.080*
C6	0.2355 (3)	−0.2346 (6)	0.61278 (9)	0.0596 (9)
H6	0.2871	−0.1255	0.6313	0.072*
C7	0.3302 (3)	−0.0417 (6)	0.55184 (9)	0.0553 (8)
H7	0.3314	−0.0376	0.5226	0.066*
C8	0.5635 (3)	0.4391 (6)	0.57512 (8)	0.0498 (8)
C9	0.6968 (4)	0.6468 (7)	0.64781 (9)	0.0725 (10)
H9A	0.7778	0.6104	0.6361	0.087*
H9B	0.6662	0.8177	0.6383	0.087*
C10	0.7200 (5)	0.6388 (8)	0.69529 (11)	0.0815 (12)
C11	0.8198 (6)	0.4920 (11)	0.71552 (14)	0.1309 (19)
H11	0.8712	0.3939	0.6993	0.157*
C12	0.8464 (8)	0.4854 (16)	0.7592 (2)	0.189 (4)
H12	0.9153	0.3870	0.7727	0.226*
C13	0.7687 (13)	0.6270 (17)	0.7815 (2)	0.199 (5)
H13	0.7830	0.6173	0.8110	0.239*
C14	0.6712 (12)	0.7826 (15)	0.7635 (2)	0.212 (5)
H14	0.6231	0.8854	0.7801	0.255*
C15	0.6444 (7)	0.7838 (11)	0.71840 (14)	0.138 (2)
H15	0.5758	0.8830	0.7049	0.166*
C16	−0.0915 (4)	−0.8990 (7)	0.59433 (13)	0.0942 (13)
H16A	−0.1487	−0.7997	0.5741	0.141*
H16B	−0.1431	−0.9991	0.6117	0.141*
H16C	−0.0392	−1.0135	0.5795	0.141*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0604 (6)	0.0731 (6)	0.0556 (5)	−0.0213 (5)	0.0101 (4)	0.0138 (4)
S2	0.0699 (6)	0.0685 (6)	0.0511 (4)	−0.0216 (5)	0.0086 (4)	0.0112 (4)
O1	0.0753 (19)	0.0729 (16)	0.0980 (16)	−0.0258 (14)	0.0229 (14)	0.0156 (14)

N1	0.0472 (16)	0.0563 (16)	0.0604 (14)	−0.0115 (14)	0.0078 (12)	0.0121 (13)
N2	0.0510 (17)	0.0639 (17)	0.0514 (13)	−0.0149 (14)	0.0087 (12)	0.0088 (13)
C1	0.0451 (19)	0.0492 (18)	0.0530 (17)	−0.0071 (15)	0.0060 (14)	0.0066 (14)
C2	0.056 (2)	0.067 (2)	0.0508 (16)	−0.0112 (18)	0.0042 (14)	0.0004 (16)
C3	0.053 (2)	0.058 (2)	0.069 (2)	−0.0126 (17)	−0.0002 (15)	−0.0007 (17)
C4	0.050 (2)	0.0486 (19)	0.074 (2)	−0.0096 (16)	0.0093 (16)	0.0115 (17)
C5	0.074 (2)	0.073 (2)	0.0528 (17)	−0.016 (2)	0.0123 (16)	0.0100 (17)
C6	0.063 (2)	0.060 (2)	0.0542 (18)	−0.0183 (18)	0.0026 (15)	−0.0033 (16)
C7	0.0455 (19)	0.064 (2)	0.0565 (17)	−0.0062 (17)	0.0073 (14)	0.0112 (16)
C8	0.0430 (18)	0.0526 (19)	0.0544 (16)	−0.0034 (15)	0.0088 (14)	0.0089 (15)
C9	0.088 (3)	0.071 (2)	0.0579 (18)	−0.030 (2)	0.0062 (17)	0.0061 (17)
C10	0.120 (4)	0.066 (2)	0.057 (2)	−0.034 (2)	0.006 (2)	0.004 (2)
C11	0.157 (5)	0.132 (4)	0.094 (3)	−0.011 (4)	−0.023 (3)	0.025 (3)
C12	0.271 (10)	0.173 (7)	0.099 (4)	−0.046 (7)	−0.069 (5)	0.040 (5)
C13	0.408 (15)	0.124 (7)	0.064 (4)	−0.114 (8)	0.031 (6)	−0.006 (4)
C14	0.433 (16)	0.110 (6)	0.105 (5)	−0.051 (7)	0.080 (7)	−0.021 (4)
C15	0.228 (7)	0.119 (4)	0.073 (3)	−0.013 (4)	0.040 (4)	−0.005 (3)
C16	0.072 (3)	0.067 (3)	0.145 (3)	−0.028 (2)	0.019 (2)	0.016 (3)

Geometric parameters (Å, °)

S1—C8	1.665 (3)	C6—H6	0.9300
S2—C8	1.739 (3)	C7—H7	0.9300
S2—C9	1.814 (3)	C9—C10	1.494 (4)
O1—C4	1.360 (4)	C9—H9A	0.9700
O1—C16	1.427 (4)	C9—H9B	0.9700
N1—C7	1.269 (4)	C10—C15	1.356 (6)
N1—N2	1.371 (3)	C10—C11	1.367 (6)
N2—C8	1.331 (3)	C11—C12	1.376 (7)
N2—H2	0.8600	C11—H11	0.9300
C1—C2	1.376 (4)	C12—C13	1.345 (11)
C1—C6	1.390 (4)	C12—H12	0.9300
C1—C7	1.446 (4)	C13—C14	1.352 (13)
C2—C3	1.376 (4)	C13—H13	0.9300
C2—H2A	0.9300	C14—C15	1.423 (8)
C3—C4	1.375 (4)	C14—H14	0.9300
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.381 (4)	C16—H16A	0.9600
C5—C6	1.368 (4)	C16—H16B	0.9600
C5—H5	0.9300	C16—H16C	0.9600
C8—S2—C9	101.16 (14)	C10—C9—S2	108.1 (2)
C4—O1—C16	117.8 (3)	C10—C9—H9A	110.1
C7—N1—N2	115.5 (2)	S2—C9—H9A	110.1
C8—N2—N1	120.6 (2)	C10—C9—H9B	110.1
C8—N2—H2	119.7	S2—C9—H9B	110.1
N1—N2—H2	119.7	H9A—C9—H9B	108.4
C2—C1—C6	118.2 (3)	C15—C10—C11	119.8 (4)
C2—C1—C7	120.2 (3)	C15—C10—C9	119.9 (4)
C6—C1—C7	121.6 (3)	C11—C10—C9	120.2 (4)

C3—C2—C1	121.9 (3)	C10—C11—C12	121.9 (6)
C3—C2—H2A	119.0	C10—C11—H11	119.1
C1—C2—H2A	119.0	C12—C11—H11	119.1
C4—C3—C2	119.4 (3)	C13—C12—C11	117.3 (8)
C4—C3—H3	120.3	C13—C12—H12	121.3
C2—C3—H3	120.3	C11—C12—H12	121.3
O1—C4—C3	125.3 (3)	C12—C13—C14	123.8 (7)
O1—C4—C5	115.4 (3)	C12—C13—H13	118.1
C3—C4—C5	119.2 (3)	C14—C13—H13	118.1
C6—C5—C4	121.1 (3)	C13—C14—C15	117.8 (8)
C6—C5—H5	119.4	C13—C14—H14	121.1
C4—C5—H5	119.4	C15—C14—H14	121.1
C5—C6—C1	120.1 (3)	C10—C15—C14	119.2 (7)
C5—C6—H6	119.9	C10—C15—H15	120.4
C1—C6—H6	119.9	C14—C15—H15	120.4
N1—C7—C1	122.2 (3)	O1—C16—H16A	109.5
N1—C7—H7	118.9	O1—C16—H16B	109.5
C1—C7—H7	118.9	H16A—C16—H16B	109.5
N2—C8—S1	121.0 (2)	O1—C16—H16C	109.5
N2—C8—S2	113.5 (2)	H16A—C16—H16C	109.5
S1—C8—S2	125.59 (18)	H16B—C16—H16C	109.5

Hydrogen-bond geometry (Å, °)

Cg is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···S1^{i}	0.86	2.59	3.397 (4)	158
C16—H16C···Cg^{ii}	0.96	2.83	3.671 (5)	147

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

Fig. 1