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Benzyl 3-[(*E*)-furfurylidene]dithiocarbazate

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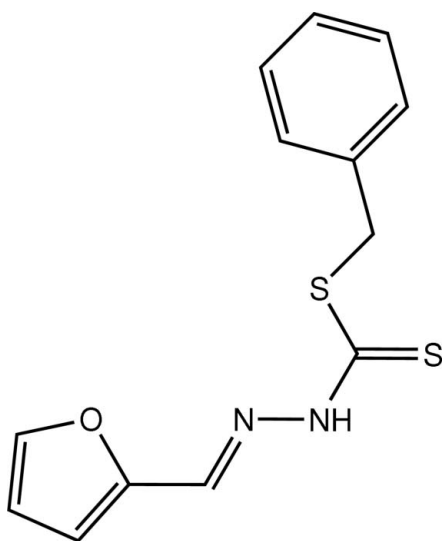
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.033; wR factor = 0.090; data-to-parameter ratio = 14.3.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{N}_2\text{OS}_2$, the molecule assumes an *E* configuration, with the furan ring and dithiocarbazate units located on opposite sides of the $\text{N}=\text{C}$ double bond. In the crystal structure, molecules are linked *via* two intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds to form centrosymmetric dimers.

Related literature

For general background, see: Okabe *et al.* (1993). For related structures, see: Shan *et al.* (2006, 2008). For the synthesis and background, see: Hu *et al.* (2001).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{12}\text{N}_2\text{OS}_2$
 $M_r = 276.37$
 Triclinic, $P\bar{1}$
 $a = 4.8331$ (11) Å
 $b = 12.040$ (3) Å
 $c = 12.549$ (3) Å
 $\alpha = 108.203$ (7)°
 $\beta = 99.704$ (9)°
 $\gamma = 97.910$ (8)°
 $V = 669.5$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.39$ mm⁻¹
 $T = 295$ (2) K
 $0.42 \times 0.36 \times 0.32$ mm

Data collection

Rigaku R-Axis RAPID IP diffractometer
 Absorption correction: none
 7084 measured reflections
 2324 independent reflections
 1799 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.090$
 $S = 1.07$
 2324 reflections
 163 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2N}\cdots\text{S1}^i$	0.86	2.56	3.3761 (19)	158

Symmetry code: (i) $-x + 2, -y, -z + 1$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 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: HB2727).

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supplementary materials

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Benzyl 3-[(*E*)-furfurylidene]dithiocarbazate

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Comment

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

The N1—C5 distance indicates a typical C=N double bond. The furan and dithiocarbazate moieties are located on the opposite positions of the C=N bond, thus the molecule assumes an *E*-configuration, which agrees with that found in methyl (β -*N*-phenylmethylene)dithiocarbazate (Shan *et al.*, 2006).

In the molecule of (I), the furan ring is slightly twisted with respect to the dithiocarbazate plane with a dihedral angle of 7.58 (14)°, whereas the phenyl ring of the thioester group is nearly perpendicular to the dithiocarbazate plane with a dihedral angle of 85.51 (5)°. This is similar to that found in a related structure, benzyl 3-[(*E*)-phenylmethylene]dithiocarbazate (Shan *et al.*, 2008).

In the crystal of (I), adjacent molecules are linked by intermolecular N—H⋯S hydrogen bonding into inversion dimers (Fig. 1 and Table 1).

Experimental

Benzyl dithiocarbazate was synthesized as described previously (Hu *et al.*, 2001). Benzyl dithiocarbazate (1.98 g, 10 mmol) and furfural (0.96 g, 10 mmol) were dissolved in ethanol (40 ml) and the solution was refluxed for 12 h. A yellow crystalline product appeared after cooling to room temperature; it was separated and washed with cold water three times. Yellow prisms of (I) were obtained by recrystallization from an ethanol solution.

Refinement

The H atoms were placed in calculated positions with C—H = 0.97 (methylene), 0.93 Å (aromatic) and N—H = 0.86 Å, and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$

Figures

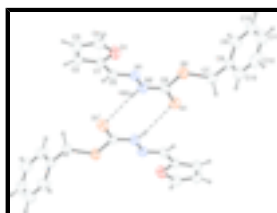


Fig. 1. The inversion dimer in the crystal of (I) drawn with 30% probability displacement ellipsoids (arbitrary spheres for H atoms). Dashed lines indicate hydrogen bonding [symmetry code: (i) 2 - x, -y, 1 - z].

Benzyl 3-[(*E*)-furfurylidene]dithiocarbazate

Crystal data

$C_{13}H_{12}N_2OS_2$	$Z = 2$
$M_r = 276.37$	$F_{000} = 288$
Triclinic, $P\bar{1}$	$D_x = 1.371 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 4.8331 (11) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 12.040 (3) \text{ \AA}$	Cell parameters from 3836 reflections
$c = 12.549 (3) \text{ \AA}$	$\theta = 1.8\text{--}25.0^\circ$
$\alpha = 108.203 (7)^\circ$	$\mu = 0.39 \text{ mm}^{-1}$
$\beta = 99.704 (9)^\circ$	$T = 295 (2) \text{ K}$
$\gamma = 97.910 (8)^\circ$	Prism, yellow
$V = 669.5 (3) \text{ \AA}^3$	$0.42 \times 0.36 \times 0.32 \text{ mm}$

Data collection

Rigaku R-Axis RAPID IP diffractometer	2324 independent reflections
Radiation source: fine-focus sealed tube	1799 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.024$
Detector resolution: $10.00 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 25.0^\circ$
$T = 295(2) \text{ K}$	$\theta_{\text{min}} = 1.8^\circ$
ω scans	$h = -5 \rightarrow 5$
Absorption correction: none	$k = -14 \rightarrow 13$
7084 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H-atom parameters constrained
$wR(F^2) = 0.090$	$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.0525P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
2324 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
163 parameters	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	1.03544 (13)	-0.02756 (5)	0.32307 (4)	0.0669 (2)
S2	0.84716 (12)	0.18647 (4)	0.27822 (4)	0.06143 (19)
O1	0.4143 (3)	0.41877 (12)	0.56065 (11)	0.0662 (4)
N1	0.6602 (3)	0.22213 (13)	0.48240 (13)	0.0533 (4)
N2	0.7837 (3)	0.12309 (13)	0.45371 (13)	0.0569 (4)
H2N	0.7943	0.0807	0.4976	0.068*
C1	0.4147 (4)	0.33502 (16)	0.61307 (15)	0.0527 (5)
C2	0.2854 (5)	0.36601 (19)	0.70185 (17)	0.0640 (5)
H2	0.2592	0.3242	0.7516	0.077*
C3	0.1967 (5)	0.47383 (19)	0.70534 (19)	0.0711 (6)
H3	0.1011	0.5168	0.7574	0.085*
C4	0.2776 (5)	0.50159 (19)	0.61914 (19)	0.0720 (6)
H4	0.2452	0.5687	0.6011	0.086*
C5	0.5457 (4)	0.23599 (17)	0.56929 (16)	0.0548 (5)
H5	0.5479	0.1793	0.6054	0.066*
C6	0.8870 (4)	0.09223 (16)	0.35868 (15)	0.0510 (5)
C7	1.0091 (5)	0.11971 (19)	0.15901 (17)	0.0647 (5)
H7A	1.2107	0.1229	0.1872	0.078*
H7B	0.9145	0.0368	0.1189	0.078*
C8	0.9740 (4)	0.19026 (17)	0.07913 (16)	0.0560 (5)
C9	0.7422 (5)	0.1560 (2)	-0.01297 (19)	0.0764 (6)
H9	0.6064	0.0877	-0.0258	0.092*
C10	0.7056 (7)	0.2192 (3)	-0.0861 (2)	0.0916 (8)
H10	0.5469	0.1939	-0.1476	0.110*
C11	0.9002 (8)	0.3183 (3)	-0.0689 (3)	0.0931 (9)
H11	0.8750	0.3616	-0.1182	0.112*
C12	1.1348 (7)	0.3554 (2)	0.0210 (3)	0.0979 (9)
H12	1.2695	0.4235	0.0325	0.117*
C13	1.1712 (5)	0.2906 (2)	0.0954 (2)	0.0795 (6)
H13	1.3306	0.3159	0.1565	0.095*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0906 (4)	0.0557 (3)	0.0675 (3)	0.0399 (3)	0.0222 (3)	0.0265 (2)
S2	0.0790 (4)	0.0596 (3)	0.0632 (3)	0.0369 (3)	0.0240 (3)	0.0313 (2)
O1	0.0945 (11)	0.0586 (8)	0.0640 (8)	0.0373 (7)	0.0296 (7)	0.0313 (7)
N1	0.0610 (10)	0.0490 (9)	0.0560 (9)	0.0254 (8)	0.0127 (8)	0.0206 (7)
N2	0.0722 (11)	0.0519 (9)	0.0595 (9)	0.0302 (8)	0.0188 (8)	0.0275 (7)
C1	0.0573 (12)	0.0517 (11)	0.0549 (10)	0.0174 (9)	0.0118 (9)	0.0244 (9)
C2	0.0741 (14)	0.0667 (13)	0.0656 (12)	0.0244 (11)	0.0285 (11)	0.0316 (10)
C3	0.0787 (15)	0.0672 (14)	0.0736 (14)	0.0314 (12)	0.0303 (12)	0.0187 (11)
C4	0.0920 (17)	0.0575 (13)	0.0773 (14)	0.0389 (12)	0.0243 (12)	0.0253 (11)
C5	0.0623 (12)	0.0526 (11)	0.0585 (11)	0.0213 (9)	0.0138 (9)	0.0275 (9)
C6	0.0525 (11)	0.0483 (11)	0.0547 (10)	0.0173 (9)	0.0079 (8)	0.0201 (8)
C7	0.0737 (14)	0.0654 (13)	0.0702 (12)	0.0341 (11)	0.0265 (11)	0.0303 (10)
C8	0.0629 (13)	0.0559 (12)	0.0600 (11)	0.0263 (10)	0.0251 (10)	0.0229 (9)
C9	0.0843 (17)	0.0717 (15)	0.0764 (14)	0.0172 (12)	0.0151 (13)	0.0309 (12)
C10	0.117 (2)	0.100 (2)	0.0697 (15)	0.0416 (18)	0.0182 (14)	0.0385 (15)
C11	0.132 (3)	0.103 (2)	0.0922 (19)	0.065 (2)	0.0647 (19)	0.0602 (17)
C12	0.103 (2)	0.0752 (17)	0.142 (3)	0.0230 (16)	0.062 (2)	0.0551 (18)
C13	0.0761 (16)	0.0753 (16)	0.0940 (16)	0.0195 (13)	0.0238 (13)	0.0346 (13)

Geometric parameters (\AA , $^\circ$)

S1—C6	1.6686 (18)	C5—H5	0.9300
S2—C6	1.7477 (19)	C7—C8	1.507 (3)
S2—C7	1.820 (2)	C7—H7A	0.9700
O1—C4	1.363 (2)	C7—H7B	0.9700
O1—C1	1.365 (2)	C8—C13	1.369 (3)
N1—C5	1.280 (2)	C8—C9	1.378 (3)
N1—N2	1.381 (2)	C9—C10	1.369 (3)
N2—C6	1.336 (2)	C9—H9	0.9300
N2—H2N	0.8600	C10—C11	1.348 (4)
C1—C2	1.345 (3)	C10—H10	0.9300
C1—C5	1.428 (3)	C11—C12	1.368 (4)
C2—C3	1.412 (3)	C11—H11	0.9300
C2—H2	0.9300	C12—C13	1.395 (4)
C3—C4	1.329 (3)	C12—H12	0.9300
C3—H3	0.9300	C13—H13	0.9300
C4—H4	0.9300		
C6—S2—C7	102.07 (9)	C8—C7—S2	107.15 (13)
C4—O1—C1	106.12 (15)	C8—C7—H7A	110.3
C5—N1—N2	114.92 (16)	S2—C7—H7A	110.3
C6—N2—N1	120.90 (15)	C8—C7—H7B	110.3
C6—N2—H2N	119.6	S2—C7—H7B	110.3
N1—N2—H2N	119.6	H7A—C7—H7B	108.5
C2—C1—O1	109.46 (17)	C13—C8—C9	117.6 (2)

C2—C1—C5	131.99 (18)	C13—C8—C7	121.2 (2)
O1—C1—C5	118.55 (16)	C9—C8—C7	121.1 (2)
C1—C2—C3	107.23 (19)	C10—C9—C8	122.0 (2)
C1—C2—H2	126.4	C10—C9—H9	119.0
C3—C2—H2	126.4	C8—C9—H9	119.0
C4—C3—C2	106.14 (18)	C11—C10—C9	119.8 (3)
C4—C3—H3	126.9	C11—C10—H10	120.1
C2—C3—H3	126.9	C9—C10—H10	120.1
C3—C4—O1	111.05 (19)	C10—C11—C12	120.3 (3)
C3—C4—H4	124.5	C10—C11—H11	119.9
O1—C4—H4	124.5	C12—C11—H11	119.9
N1—C5—C1	122.66 (18)	C11—C12—C13	119.7 (3)
N1—C5—H5	118.7	C11—C12—H12	120.1
C1—C5—H5	118.7	C13—C12—H12	120.1
N2—C6—S1	121.22 (14)	C8—C13—C12	120.5 (2)
N2—C6—S2	114.01 (13)	C8—C13—H13	119.7
S1—C6—S2	124.76 (11)	C12—C13—H13	119.7

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2N \cdots S1 ⁱ	0.86	2.56	3.3761 (19)	158

Symmetry codes: (i) $-x+2, -y, -z+1$.

Fig. 1

