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Methyl 3-[(*E*)-furfurylidene]dithiocarbamate

Shang Shan,* Shan-Heng Wang, Ying-Li Xu, Pei-Jin Xie and Yu-Liang Tian

College of Chemical Engineering and Materials Science, Zhejiang University of Technology, People's Republic of China

Correspondence e-mail: shangshan@mail.hz.zj.cn

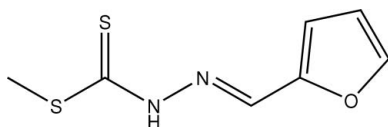
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.032; wR factor = 0.097; data-to-parameter ratio = 14.6.

The molecule of the title Schiff base compound, $\text{C}_7\text{H}_8\text{N}_2\text{OS}_2$, prepared by the reaction of methyl dithiocarbamate and furfural in an ethanol solution under reflux, adopts an *E* configuration; the dithiocarbamate and furan units are located on opposite sides of the $\text{C}=\text{N}$ double bond. The planar dithiocarbamate group is twisted slightly with respect to the furan ring, making a dihedral angle of $5.2(1)^\circ$. Adjacent molecules are linked by $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonding to form a supramolecular dimer across an inversion center.

Related literature

For general background, see: Okabe *et al.* (1993); Shan *et al.* (2002, 2003). For a related structure, see: Chen *et al.* (2007). For the synthesis, see: Hu *et al.* (2001).



Experimental

Crystal data

$\text{C}_7\text{H}_8\text{N}_2\text{OS}_2$
 $M_r = 200.27$
 Triclinic, $P\bar{1}$
 $a = 4.0866(8)$ Å
 $b = 8.8698(12)$ Å
 $c = 12.8453(15)$ Å
 $\alpha = 93.970(14)^\circ$
 $\beta = 91.856(12)^\circ$

$\gamma = 98.293(12)^\circ$
 $V = 459.21(12)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.53$ mm⁻¹
 $T = 294(2)$ K
 $0.34 \times 0.28 \times 0.20$ mm

Data collection

Rigaku R-Axis RAPID IP diffractometer
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.850$, $T_{\max} = 0.950$
 (expected range = 0.804–0.899)

4733 measured reflections
 1608 independent reflections
 1349 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.096$
 $S = 1.09$
 1608 reflections

110 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{S1}^i$	0.86	2.65	3.4892 (17)	165

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

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: XU2417).

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supporting information

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

Shang Shan, Shan-Heng Wang, Ying-Li Xu, Pei-Jin Xie and Yu-Liang Tian

S1. Comment

Since some hydrazone derivatives have shown the potential bioactivity as DNA-damaging or mutagenic agents (Okabe *et al.*, 1993), a lots of new hydrazone compounds has been synthesized in our laboratory (Shan *et al.*, 2002, 2003). As part of the ongoing investigation on hydrazone, we present here the crystal structure of the title compound.

The molecular structure is shown in Fig. 1. The N2=C3 bond distance of 1.284 (2) Å clearly indicates the double bond character for the Schiff base compound. The molecule adopts an *E* configuration, the carbazate and furan moieties located on the opposite positions of the N2=C3 bond; similar to that found in a related structure (Chen *et al.*, 2007). The dithiocarbazate moiety is well co-planar, the maximum atomic deviation being 0.037 (1) Å (S2), and the dithiocarbazate mean plane is slightly twisted with respect to the furan plane by a smaller dihedral angle of 5.2 (1)°. This shows the whole molecule is nearly co-planar.

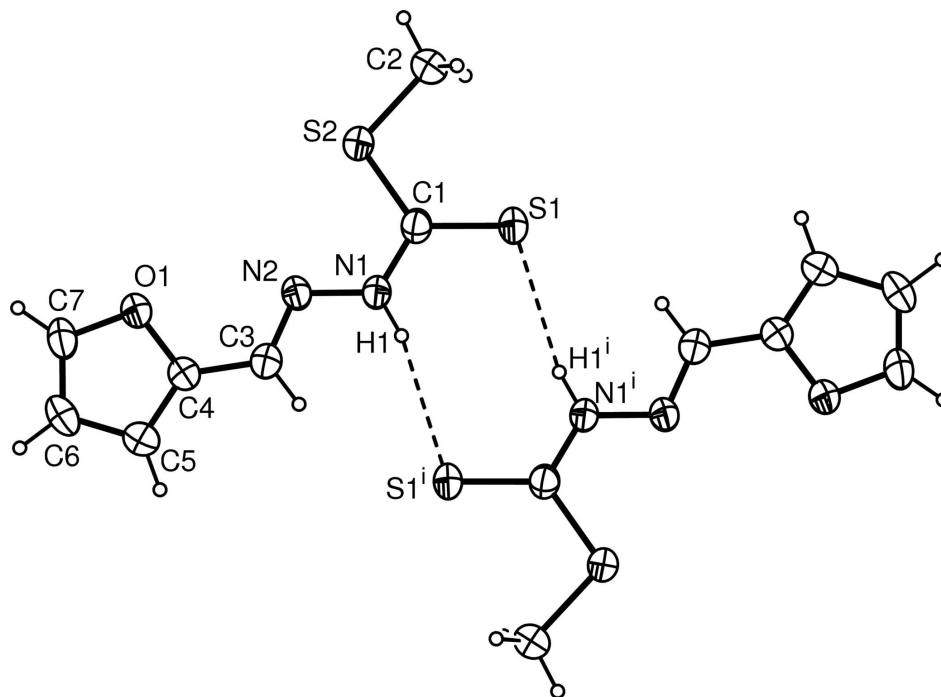
Inter-molecular N—H...S hydrogen bonding links adjacent molecules to form the centro-symmetric supra-molecular dimer (Fig. 1 and Table 1).

S2. Experimental

Methyl dithiocarbazate was synthesized in the manner reported previously (Hu *et al.*, 2001). Methyl dithiocarbazate (1.24 g, 10 mmol) and furfural (0.96 g, 10 mmol) were dissolved in ethanol (10 ml) and refluxed for 4 h. Yellow crystalline product appeared after cooling to room temperature. They were separated and washed with cold water. Single crystals of the title compound were obtained by recrystallization from an ethanol solution.

S3. Refinement

Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angle was refined to fit electron density, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. Other H atoms were placed in calculated positions with C—H = 0.93 and N—H = 0.86 Å, and refined in the riding mode, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

**Figure 1**

The molecular structure of the title compound with 40% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (i) $-x, 1 - y, 1 - z$].

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

Crystal data

$C_7H_8N_2OS_2$

$M_r = 200.27$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 4.0866$ (8) Å

$b = 8.8698$ (12) Å

$c = 12.8453$ (15) Å

$\alpha = 93.970$ (14)°

$\beta = 91.856$ (12)°

$\gamma = 98.293$ (12)°

$V = 459.21$ (12) Å³

$Z = 2$

$F(000) = 208$

$D_x = 1.448$ Mg m⁻³

Melting point = 414–416 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2276 reflections

$\theta = 2.0$ – 25.0 °

$\mu = 0.53$ mm⁻¹

$T = 294$ K

Prism, yellow

$0.34 \times 0.28 \times 0.20$ mm

Data collection

Rigaku R-AXIS RAPID IP
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.0 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.850$, $T_{\max} = 0.950$

4733 measured reflections

1608 independent reflections

1349 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 25.2$ °, $\theta_{\min} = 1.6$ °

$h = -4 \rightarrow 4$

$k = -10 \rightarrow 10$

$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0598P)^2]$
$S = 1.09$	where $P = (F_o^2 + 2F_c^2)/3$
1608 reflections	$(\Delta/\sigma)_{\max} = 0.001$
110 parameters	$\Delta\rho_{\max} = 0.19 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.20815 (13)	0.27619 (5)	0.50863 (4)	0.0516 (2)
S2	0.38692 (13)	0.20142 (5)	0.28541 (4)	0.0506 (2)
N1	0.1859 (4)	0.45265 (16)	0.35473 (12)	0.0464 (4)
H1	0.1098	0.5149	0.3988	0.056*
N2	0.2353 (4)	0.49265 (16)	0.25394 (11)	0.0449 (4)
O1	0.3206 (4)	0.60245 (14)	0.05809 (10)	0.0553 (4)
C1	0.2537 (4)	0.3197 (2)	0.38518 (14)	0.0407 (4)
C2	0.4665 (5)	0.0393 (2)	0.35393 (17)	0.0557 (5)
H2A	0.6311	0.0720	0.4090	0.084*
H2B	0.5448	-0.0343	0.3062	0.084*
H2C	0.2659	-0.0063	0.3831	0.084*
C3	0.1483 (5)	0.6216 (2)	0.23454 (16)	0.0502 (5)
H3	0.0577	0.6772	0.2875	0.060*
C4	0.1847 (5)	0.6835 (2)	0.13501 (16)	0.0481 (5)
C5	0.1076 (7)	0.8131 (3)	0.09881 (19)	0.0685 (6)
H5	0.0139	0.8886	0.1360	0.082*
C6	0.1961 (6)	0.8126 (3)	-0.00661 (18)	0.0673 (6)
H6	0.1705	0.8873	-0.0523	0.081*
C7	0.3227 (6)	0.6849 (3)	-0.02770 (17)	0.0608 (6)
H7	0.4018	0.6557	-0.0920	0.073*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0714 (4)	0.0502 (3)	0.0359 (3)	0.0125 (2)	0.0107 (2)	0.0105 (2)

S2	0.0694 (4)	0.0482 (3)	0.0378 (3)	0.0171 (2)	0.0114 (2)	0.0072 (2)
N1	0.0681 (10)	0.0401 (8)	0.0326 (9)	0.0104 (7)	0.0108 (7)	0.0056 (7)
N2	0.0614 (10)	0.0393 (8)	0.0352 (9)	0.0078 (7)	0.0060 (7)	0.0077 (7)
O1	0.0837 (10)	0.0443 (7)	0.0418 (8)	0.0168 (7)	0.0145 (7)	0.0112 (6)
C1	0.0456 (10)	0.0405 (9)	0.0345 (10)	0.0013 (8)	0.0024 (8)	0.0026 (8)
C2	0.0701 (14)	0.0467 (11)	0.0534 (13)	0.0161 (9)	0.0056 (10)	0.0087 (9)
C3	0.0655 (13)	0.0478 (11)	0.0390 (11)	0.0114 (9)	0.0094 (9)	0.0051 (9)
C4	0.0631 (12)	0.0419 (10)	0.0419 (11)	0.0129 (9)	0.0072 (9)	0.0076 (8)
C5	0.0988 (17)	0.0596 (13)	0.0577 (14)	0.0391 (12)	0.0168 (12)	0.0165 (11)
C6	0.0947 (17)	0.0596 (13)	0.0544 (15)	0.0224 (12)	0.0054 (12)	0.0278 (11)
C7	0.0875 (16)	0.0583 (12)	0.0393 (12)	0.0115 (11)	0.0109 (10)	0.0173 (9)

Geometric parameters (Å, °)

S1—C1	1.6675 (18)	C2—H2B	0.9600
S2—C1	1.7500 (19)	C2—H2C	0.9600
S2—C2	1.800 (2)	C3—C4	1.430 (3)
N1—N2	1.379 (2)	C3—H3	0.9300
N1—C1	1.331 (2)	C4—C5	1.344 (3)
N1—H1	0.8600	C5—C6	1.413 (3)
N2—C3	1.284 (2)	C5—H5	0.9300
O1—C4	1.361 (2)	C6—C7	1.327 (4)
O1—C7	1.364 (2)	C6—H6	0.9300
C2—H2A	0.9600	C7—H7	0.9300
C1—S2—C2	101.98 (9)	N2—C3—C4	122.79 (19)
C1—N1—N2	121.34 (16)	N2—C3—H3	118.6
C1—N1—H1	119.3	C4—C3—H3	118.6
N2—N1—H1	119.3	C5—C4—O1	109.57 (17)
C3—N2—N1	114.59 (16)	C5—C4—C3	132.1 (2)
C4—O1—C7	106.45 (15)	O1—C4—C3	118.37 (16)
N1—C1—S1	120.76 (14)	C4—C5—C6	106.8 (2)
N1—C1—S2	114.05 (13)	C4—C5—H5	126.6
S1—C1—S2	125.19 (11)	C6—C5—H5	126.6
S2—C2—H2A	109.5	C7—C6—C5	106.67 (19)
S2—C2—H2B	109.5	C7—C6—H6	126.7
H2A—C2—H2B	109.5	C5—C6—H6	126.7
S2—C2—H2C	109.5	C6—C7—O1	110.5 (2)
H2A—C2—H2C	109.5	C6—C7—H7	124.8
H2B—C2—H2C	109.5	O1—C7—H7	124.8
C1—N1—N2—C3	177.62 (17)	N2—C3—C4—C5	179.2 (2)
N2—N1—C1—S1	177.32 (12)	N2—C3—C4—O1	-0.7 (3)
N2—N1—C1—S2	-3.0 (2)	O1—C4—C5—C6	0.7 (3)
C2—S2—C1—N1	178.23 (14)	C3—C4—C5—C6	-179.2 (2)
C2—S2—C1—S1	-2.13 (15)	C4—C5—C6—C7	-0.5 (3)
N1—N2—C3—C4	179.09 (17)	C5—C6—C7—O1	0.1 (3)
C7—O1—C4—C5	-0.6 (2)	C4—O1—C7—C6	0.3 (3)

C7—O1—C4—C3 179.25 (18)

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N1—H1...S1 ⁱ	0.86	2.65	3.4892 (17)	165

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