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(3*E*)-3-(1,3-Dithiolan-2-ylidenehydrazono)butan-2-one

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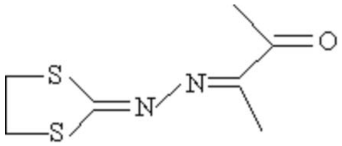
Key indicators: single-crystal X-ray study; *T* = 294 K; mean σ(C–C) = 0.005 Å; *R* factor = 0.043; *wR* factor = 0.141; data-to-parameter ratio = 18.2.

The title compound, $C_7H_{10}N_2OS_2$, was synthesized by the Lewis acid-catalysed reaction of butane-2,3-dione and 2-hydrazono-1,3-dithiolane. The molecule exists as the *E* isomer and there is an intramolecular C—H···N hydrogen bond forming a ring with graph set *S*(5). In the crystal structure, molecules are linked into W-form *C*(9) chains *via* a C—H···O intermolecular hydrogen bond and the two parallel chains pass through the unit cell in the *b*-axis direction.

Related literature

For related literature, see: Beghidja *et al.* (2005); Bernstein *et al.* (1995); Cremer & Pople (1975); Xu *et al.* (2005).



Experimental

Crystal data

$C_7H_{10}N_2OS_2$
M_r = 202.29
Monoclinic, $P2_1/n$
a = 10.878 (3) Å
b = 7.1855 (16) Å
c = 13.423 (3) Å
β = 112.095 (3)°
V = 972.2 (4) Å^3
Z = 4
Mo *K*α radiation
μ = 0.50 mm^{-1}
T = 294 (2) K
0.28 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997; Blessing, 1995)
$T_{\min}$ = 0.852, $T_{\max}$ = 0.964 (expected range = 0.799–0.904)
5366 measured reflections
2034 independent reflections
1524 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.141
S = 1.17
2034 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.40 e Å^{-3}
$\Delta\rho_{\min}$ = −0.23 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···N1	0.96	2.39	2.785 (3)	104
C1—H1*B*···O1$^{\rm i}$	0.97	2.57	3.387 (3)	142

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2064).

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supplementary materials

Acta Cryst. (2007). E**63**, o4019 [**doi:10.1107/S1600536807042420**]

(3*E*)-3-(1,3-Dithiolan-2-ylidenehydrazono)butan-2-one

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Comment

In solid state the conformation of the 1,3-dithiolane in (I) is in the envelope form (Cremer *et al.*, 1975) and atom C2 deviates by 0.613 (4) Å from the plane defined by C1, S1, C3 and S2. In an analogous compound, (II), it is in the half-chair form (Xu *et al.*, 2005; Beghidja *et al.*, 2005) (Fig.1, Table 1). Due to conjugation, the atoms N1, N2, C5, C4, C6, C7 form another plane and the dihedral angle between the two planes is 31.59 (15)°. In addition there are intramolecular C–H ··· N hydrogen-bonds forming a ring of graph set notation *S*(5) (Bernstein *et al.*, 1995) and directly influencing the coplanarity of the atoms involved (Fig. 1, Table 2). In the packing (Fig. 2) there are two *C*(9) *W*-shape chains (Bernstein *et al.*, 1995) *via* C—H···O intermolecular hydrogen-bonds involving molecules with C···O distance of 3.387 (13) Å and a C1–H1(B)···O1 angle of 142° running parallel to the (102) plane (Fig. 2, Table 2).

Experimental

The title compound was prepared from butanedione and 2-hydrazono-1, 3-dithiolane in equimolar ratio in 95% EtOH. The resulting yellow solid was recrystallized from CH_2Cl_2–EtOH to give crystals of suitable for single-crystal X-ray diffraction (yield 96%, m.p. 373 – 374 K). ^{1}H NMR (600 MHz, $CDCl_3$, δ, p.p.m.): 2.10 (3*H*, *s*, O═C—CH_3), 2.47 (3*H*, *s*, N═C—CH_3), 3.54 (*m*, 4H, CH_2CH_2). ^{13}C NMR (600 MHz, $CDCl_3$, δ, p.p.m.): 199.609 (C═O), 180.909 (N—N═ C), 160.820 (CS_2). 36.668, 35.703 (CH_2CH_2), 25.027 (CH_3—C═O), 11.708 (CH_3—C═N).

Refinement

After their location in a difference map, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms, with C — H distances of 0.96 (methyl) or 0.97 Å (CH_2), and with U_{iso}(H) values of 1.2Ueq(C), or 1.5Ueq(C) for the methyl groups.

Figures

Fig. 1. The molecular of (I) structure of the title compound, showing 50% probability ellipsoids. The C—H···N intramolecular hydrogen bond is shown dashed.

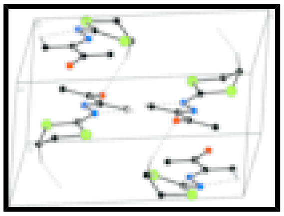


Fig. 2. Packing diagram of (I), showing the formation of the *S*(5) graph set *via* C–H···N intramolecular hydrogen-bonds and C(9) W-shape chains *via* C–H···O intermolecular hydrogen-bonds running parallel to the (104) plane (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

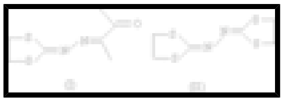


Fig. 3. The structures of (I) and (II)

(3E)-3-(1,3-Dithiolan-2-ylidenehydrazono)butan-2-one

Crystal data

$C_7H_{10}N_2OS_2$

$M_r = 202.29$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 10.878$ (3) Å

$b = 7.1855$ (16) Å

$c = 13.423$ (3) Å

$\beta = 112.095$ (3)°

$V = 972.2$ (4) Å^3

$Z = 4$

$F_{000} = 424$

$D_x = 1.382$ Mg m^{-3}

Melting point: 343 K

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

Cell parameters from 2185 reflections

$\theta = 3.0$–26.5°

$\mu = 0.50$ mm^{-1}

$T = 294$ (2) K

Block, light yellow

0.28 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1000 CCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 1997; Blessing, 1995)

$T_{\min} = 0.852$, $T_{\max} = 0.964$

5366 measured reflections

2034 independent reflections

1524 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 26.7$°

$\theta_{\min} = 2.1$°

$h = -13 \rightarrow 13$

$k = -9 \rightarrow 4$

$l = -15 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.141$

$S = 1.17$

2034 reflections

112 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.7077P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 0.40$ e Å^{-3}

$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.041 (4)

Secondary atom site location: difference Fourier map

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.09678 (8)	0.15655 (12)	0.39543 (6)	0.0507 (3)
S2	−0.15962 (8)	0.11731 (14)	0.21107 (7)	0.0597 (3)
O1	0.4927 (2)	0.1361 (3)	0.1918 (2)	0.0620 (7)
N1	0.0698 (3)	0.0643 (4)	0.1928 (2)	0.0522 (7)
N2	0.2093 (2)	0.0680 (4)	0.2460 (2)	0.0452 (6)
C1	−0.0507 (3)	0.2354 (5)	0.4160 (3)	0.0589 (9)
H1A	−0.0404	0.2155	0.4902	0.071*
H1B	−0.0641	0.3674	0.4005	0.071*
C2	−0.1678 (3)	0.1275 (5)	0.3424 (3)	0.0618 (9)
H2A	−0.2495	0.1877	0.3380	0.074*
H2B	−0.1667	0.0026	0.3701	0.074*
C3	0.0141 (3)	0.1045 (4)	0.2587 (2)	0.0428 (7)
C4	0.2748 (3)	0.0913 (4)	0.1848 (2)	0.0419 (7)
C5	0.2193 (4)	0.1190 (6)	0.0664 (3)	0.0671 (10)
H5A	0.1277	0.0824	0.0378	0.101*
H5B	0.2680	0.0447	0.0344	0.101*
H5C	0.2264	0.2479	0.0504	0.101*
C6	0.4230 (3)	0.0988 (4)	0.2422 (2)	0.0458 (7)
C7	0.4802 (3)	0.0648 (6)	0.3609 (3)	0.0631 (9)
H7A	0.5752	0.0594	0.3853	0.095*
H7B	0.4472	−0.0511	0.3764	0.095*
H7C	0.4549	0.1641	0.3970	0.095*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0442 (4)	0.0604 (5)	0.0499 (5)	−0.0007 (4)	0.0204 (3)	−0.0037 (4)
S2	0.0388 (4)	0.0768 (7)	0.0625 (6)	−0.0028 (4)	0.0181 (4)	0.0001 (4)
O1	0.0539 (13)	0.0731 (17)	0.0718 (16)	−0.0055 (11)	0.0381 (12)	−0.0039 (12)
N1	0.0472 (15)	0.0629 (17)	0.0473 (15)	−0.0029 (13)	0.0187 (12)	−0.0029 (12)
N2	0.0398 (13)	0.0519 (15)	0.0464 (14)	−0.0019 (11)	0.0193 (11)	−0.0030 (11)

C1	0.062 (2)	0.064 (2)	0.064 (2)	0.0043 (17)	0.0379 (17)	−0.0007 (17)
C2	0.0487 (18)	0.072 (2)	0.075 (2)	−0.0028 (17)	0.0349 (17)	0.0104 (18)
C3	0.0390 (15)	0.0415 (16)	0.0496 (17)	−0.0019 (12)	0.0188 (13)	0.0027 (13)
C4	0.0440 (16)	0.0415 (15)	0.0452 (16)	0.0044 (12)	0.0223 (13)	−0.0008 (12)
C5	0.058 (2)	0.096 (3)	0.0496 (19)	0.0141 (19)	0.0231 (16)	0.0145 (19)
C6	0.0485 (17)	0.0410 (16)	0.0533 (18)	0.0000 (13)	0.0253 (14)	−0.0045 (13)
C7	0.0473 (18)	0.086 (3)	0.053 (2)	−0.0066 (18)	0.0148 (15)	−0.0089 (18)

Geometric parameters (Å, °)

S1—C3	1.754 (3)	C2—H2A	0.9700
S1—C1	1.818 (3)	C2—H2B	0.9700
S2—C3	1.754 (3)	C4—C5	1.486 (4)
S2—C2	1.799 (4)	C4—C6	1.504 (4)
O1—C6	1.221 (4)	C5—H5A	0.9600
N1—C3	1.279 (4)	C5—H5B	0.9600
N1—N2	1.414 (3)	C5—H5C	0.9600
N2—C4	1.285 (4)	C6—C7	1.496 (5)
C1—C2	1.501 (5)	C7—H7A	0.9600
C1—H1A	0.9700	C7—H7B	0.9600
C1—H1B	0.9700	C7—H7C	0.9600
C3—S1—C1	95.21 (15)	N2—C4—C5	127.0 (3)
C3—S2—C2	95.09 (15)	N2—C4—C6	114.9 (3)
C3—N1—N2	110.1 (2)	C5—C4—C6	118.0 (3)
C4—N2—N1	115.3 (2)	C4—C5—H5A	109.5
C2—C1—S1	108.5 (2)	C4—C5—H5B	109.5
C2—C1—H1A	110.0	H5A—C5—H5B	109.5
S1—C1—H1A	110.0	C4—C5—H5C	109.5
C2—C1—H1B	110.0	H5A—C5—H5C	109.5
S1—C1—H1B	110.0	H5B—C5—H5C	109.5
H1A—C1—H1B	108.4	O1—C6—C7	122.0 (3)
C1—C2—S2	108.9 (2)	O1—C6—C4	119.5 (3)
C1—C2—H2A	109.9	C7—C6—C4	118.4 (3)
S2—C2—H2A	109.9	C6—C7—H7A	109.5
C1—C2—H2B	109.9	C6—C7—H7B	109.5
S2—C2—H2B	109.9	H7A—C7—H7B	109.5
H2A—C2—H2B	108.3	C6—C7—H7C	109.5
N1—C3—S1	125.6 (2)	H7A—C7—H7C	109.5
N1—C3—S2	119.1 (2)	H7B—C7—H7C	109.5
S1—C3—S2	115.35 (17)		
C3—N1—N2—C4	−157.9 (3)	C2—S2—C3—N1	168.8 (3)
C3—S1—C1—C2	31.8 (3)	C2—S2—C3—S1	−13.1 (2)
S1—C1—C2—S2	−44.9 (3)	N1—N2—C4—C5	1.7 (5)
C3—S2—C2—C1	35.0 (3)	N1—N2—C4—C6	178.9 (2)
N2—N1—C3—S1	−0.1 (4)	N2—C4—C6—O1	−173.9 (3)
N2—N1—C3—S2	177.8 (2)	C5—C4—C6—O1	3.6 (4)
C1—S1—C3—N1	169.9 (3)	N2—C4—C6—C7	4.6 (4)
C1—S1—C3—S2	−8.1 (2)	C5—C4—C6—C7	−178.0 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···N1	0.96	2.39	2.785 (3)	104
C1—H1B···O1^{i}	0.97	2.57	3.387 (3)	142

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

Fig. 1

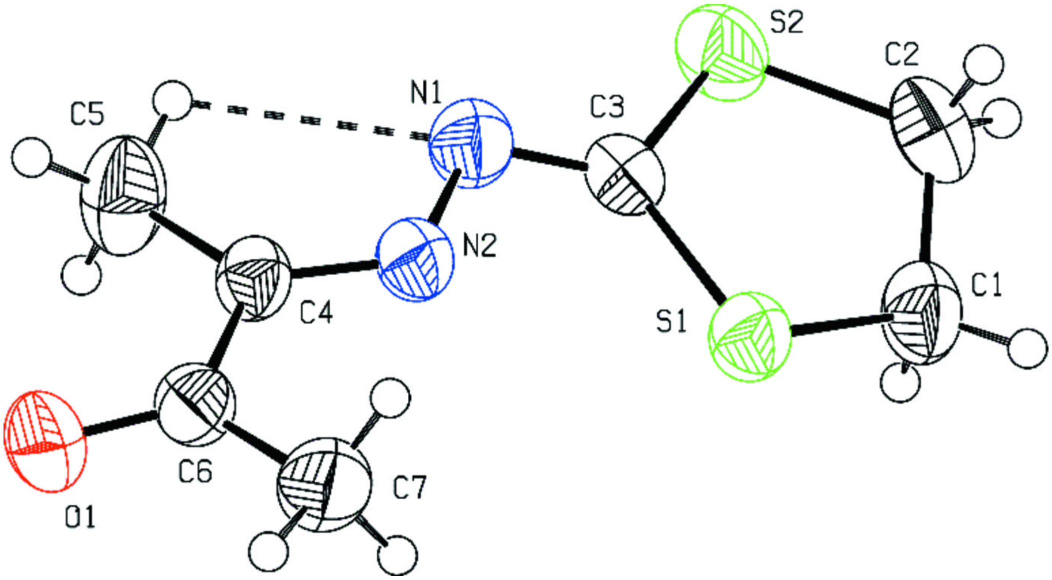


Fig. 2

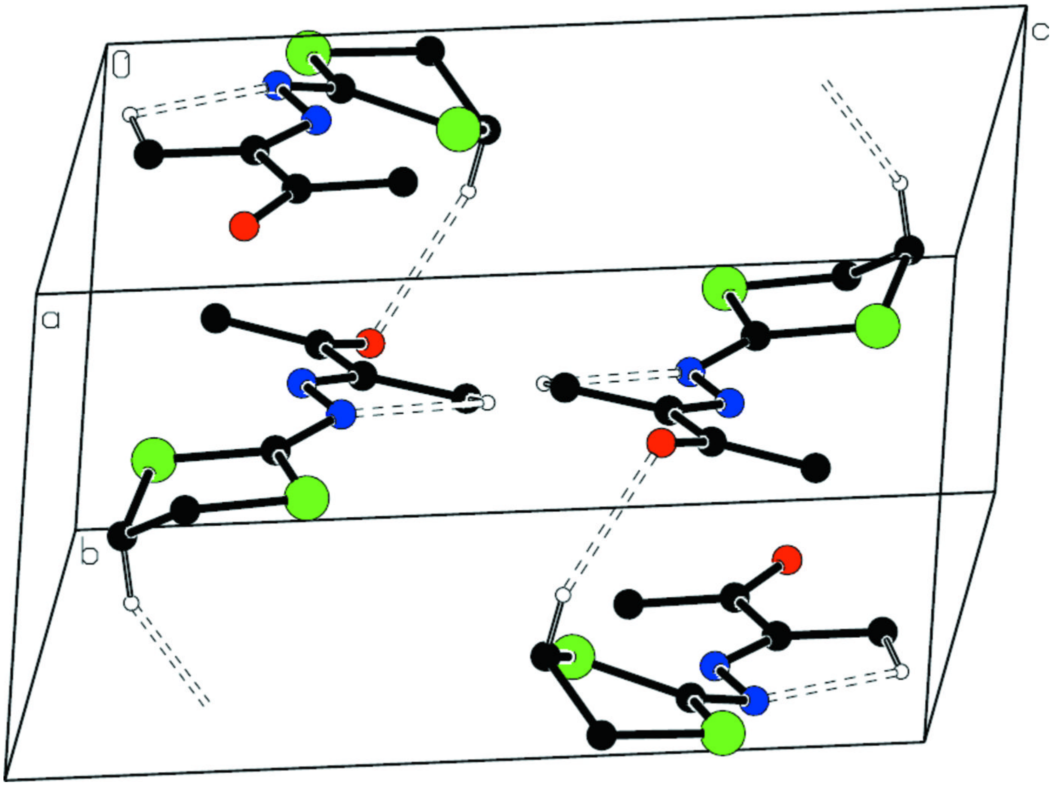
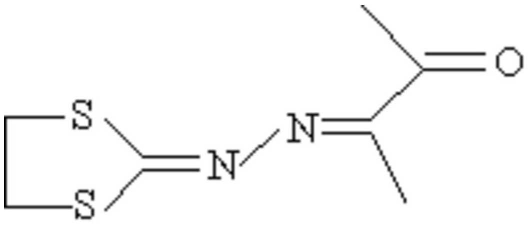
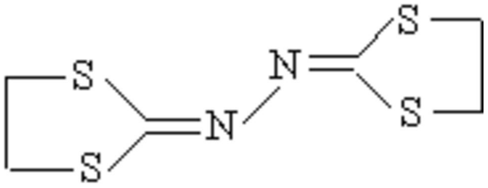


Fig. 3



(I)



(II)