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Dimethyl[(*E*)-(2-nitromethylidene-1,3-dithiolan-4-yl)methyl]amine

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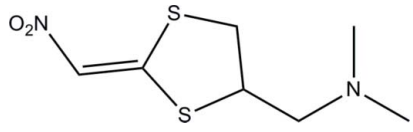
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.090; data-to-parameter ratio = 19.5.

In the title compound, $C_7H_{12}N_2O_2S_2$, the conformation of the dithiacyclopentane ring is a half-chair, with a total puckering amplitude Q_T = 0.473 (5) Å. Intermolecular C—H···N and C—H···O interactions help to establish the packing.

Related literature

For the crystal structures of related compounds, see: Xu *et al.* (2005); Ortega-Jimenez *et al.* (2000). For the biological activities of heterocyclic compounds, see: Xu *et al.* (2006); Yu *et al.* (2009). For puckering amplitude, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_7H_{12}N_2O_2S_2$
M_r = 220.31
Orthorhombic, $P2_12_12_1$
a = 5.927 (4) Å
b = 11.241 (8) Å
c = 14.90 (1) Å
V = 992.7 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.51 mm^{-1}
T = 113 K
0.30 × 0.18 × 0.08 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
T_{min} = 0.863, T_{max} = 0.961
10308 measured reflections
2346 independent reflections
2099 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.090
S = 1.02
2346 reflections
120 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983), 963 Friedel pairs
Flack parameter: −0.02 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N2^{i}	0.95	2.44	3.354 (4)	161
C4—H4···O1^{ii}	1.00	2.44	3.272 (4)	140

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (ii) $-x-\frac{1}{2}, -y+2, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors thank Dr Haibin Song, Nankai University, for the X-ray crystallographic determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5213).

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supplementary materials

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Dimethyl[(*E*)-(2-nitromethylidene-1,3-dithiolan-4-yl)methyl]amine

Shuang-Hua Yang and Zhi-Wei Zhai

Comment

Many heterocyclic compounds have been widely used as potent and broad-spectrum fungicides (Xu *et al.*, 2006; Yu *et al.*, 2009). In order to search for new heterocylic compounds with higher biological activities, we synthesized the (*E*)—*N*,*N*-dimethyl-1-(2-(nitromethylene)-1,3-dithiolan-4-yl)methanamine and describe its structure here.

In the title compound, all bond lengths in the molecular are normal and in good agreement with those reported previously (Xu *et al.*, 2005; Ortega-Jimenez *et al.*, 2000). The conformation of the dithiacyclopentane ring (C2—C4/S1/S2) is halfchair, with a total puckering amplitude (Cremer & Pople, 1975) Q_T = 0.456 (3) Å and a pseudo-twofold axis running along the direction through C2 and the mid-point of the C3—C4 bond. The intermolecular C—H···N and C—H···O hydrogen bonds stabilize the structure.

Experimental

A mixture of potassium 2-nitroethene-1,1-bis(thiolate) 10 mmol (2.13 g), 2,3-dichloro-*N*,*N*-dimethylpropan-1-amine (1.56 g, 10 mmol) is refluxed in absolute ethanol (25 ml) for 3 h. The mixture was filtered to provide crude product. The residue was purified by recrystallized from absolute EtOH, yield 1.89 g (86.0%). Single crystals suitable for X-ray measurements were obtained by recrystallization from acetonitrile at room temperature.

Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 or 1.00 Å and with U_{iso}(H) = 1.2 times U_{eq}(C) for methylene H atoms and 1.5U_{eq}(C) for the methyl H atoms.

Computing details

Data collection: *CrystalClear* (Rigaku, 2007); cell refinement: *CrystalClear* (Rigaku, 2007); data reduction: *CrystalClear* (Rigaku, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXL97* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

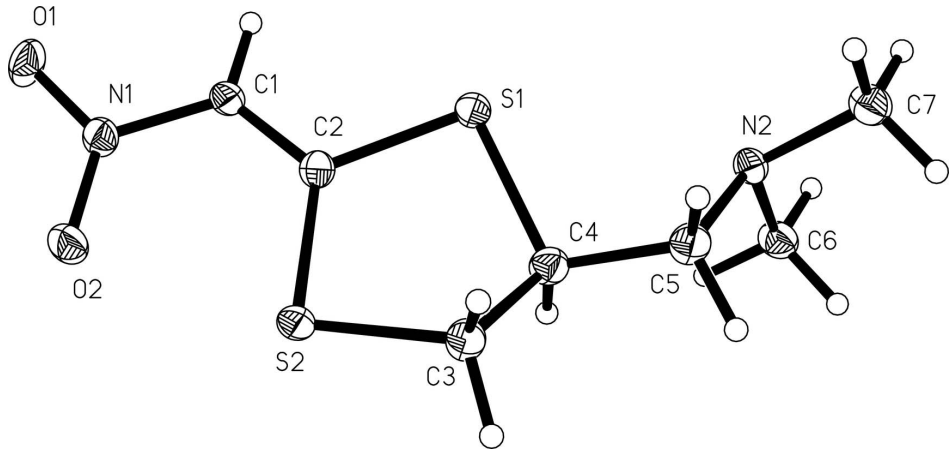

Figure 1

The molecular structure of (I), with atom labels and 40% probability displacement ellipsoids for non-H atoms.

Dimethyl[(*E*)-(2-nitromethylidene-1,3-dithiolan-4-yl)methyl]amine

Crystal data

$C_7H_{12}N_2O_2S_2$
$M_r = 220.31$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 5.927$ (4) Å
$b = 11.241$ (8) Å
$c = 14.90$ (1) Å
$V = 992.7$ (11) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.474$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3456 reflections
$\theta = 1.8$–$27.8°$
$\mu = 0.51$ mm^{-1}
$T = 113$ K
Prism, colorless
0.30 × 0.18 × 0.08 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.63 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2007)
$T_{min} = 0.863$, $T_{max} = 0.961$
10308 measured reflections
2346 independent reflections
2099 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.8°$, $\theta_{min} = 2.3°$
$h = -6 \rightarrow 7$
$k = -14 \rightarrow 14$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.090$
$S = 1.02$
2346 reflections
120 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Absolute structure: Flack (1983), 963 Friedel pairs
Flack parameter: −0.02 (10)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.16147 (11)	0.88829 (5)	0.02451 (4)	0.02114 (16)
S2	−0.05675 (11)	1.12418 (5)	0.02804 (4)	0.02176 (16)
O1	−0.4710 (3)	0.96345 (17)	−0.18337 (13)	0.0307 (5)
O2	−0.3988 (3)	1.10926 (16)	−0.09091 (12)	0.0276 (4)
N1	−0.3632 (4)	1.00756 (17)	−0.12006 (14)	0.0222 (5)
N2	0.4568 (4)	0.81812 (18)	0.18117 (14)	0.0196 (5)
C1	−0.1909 (5)	0.9375 (2)	−0.08113 (16)	0.0203 (5)
H1	−0.1734	0.8573	−0.0999	0.024*
C2	−0.0496 (4)	0.9819 (2)	−0.01767 (17)	0.0192 (5)
C3	0.1989 (5)	1.1078 (2)	0.09357 (18)	0.0256 (6)
H3A	0.3319	1.1288	0.0566	0.031*
H3B	0.1945	1.1617	0.1461	0.031*
C4	0.2158 (4)	0.9797 (2)	0.12456 (17)	0.0225 (6)
H4	0.0973	0.9636	0.1708	0.027*
C5	0.4449 (5)	0.9457 (2)	0.16155 (18)	0.0247 (6)
H5A	0.5630	0.9667	0.1173	0.030*
H5B	0.4743	0.9914	0.2172	0.030*
C6	0.3446 (5)	0.7885 (2)	0.26605 (17)	0.0255 (6)
H6A	0.3518	0.7023	0.2759	0.038*
H6B	0.1865	0.8137	0.2634	0.038*
H6C	0.4206	0.8295	0.3156	0.038*
C7	0.6931 (4)	0.7797 (3)	0.18472 (18)	0.0261 (6)
H7A	0.7689	0.8189	0.2352	0.039*
H7B	0.7688	0.8014	0.1286	0.039*
H7C	0.6995	0.6933	0.1928	0.039*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0270 (3)	0.0141 (3)	0.0223 (3)	0.0024 (3)	−0.0050 (3)	−0.0022 (3)
S2	0.0298 (4)	0.0134 (3)	0.0220 (3)	0.0023 (3)	−0.0032 (3)	−0.0015 (3)
O1	0.0375 (12)	0.0268 (10)	0.0279 (10)	−0.0037 (9)	−0.0164 (9)	−0.0017 (8)
O2	0.0313 (11)	0.0185 (9)	0.0330 (10)	0.0037 (8)	−0.0065 (8)	−0.0030 (8)
N1	0.0268 (12)	0.0167 (10)	0.0231 (11)	−0.0006 (9)	−0.0035 (10)	0.0016 (8)
N2	0.0235 (11)	0.0156 (10)	0.0198 (11)	0.0034 (9)	−0.0015 (9)	0.0023 (8)
C1	0.0270 (14)	0.0140 (11)	0.0198 (12)	0.0031 (11)	−0.0008 (11)	0.0006 (9)
C2	0.0219 (13)	0.0180 (11)	0.0177 (12)	−0.0001 (10)	0.0007 (11)	0.0028 (10)

C3	0.0344 (15)	0.0145 (11)	0.0279 (13)	−0.0005 (11)	−0.0100 (12)	−0.0005 (10)
C4	0.0263 (15)	0.0189 (12)	0.0221 (13)	−0.0007 (11)	−0.0016 (11)	−0.0007 (10)
C5	0.0296 (15)	0.0167 (12)	0.0277 (14)	−0.0022 (12)	−0.0055 (13)	−0.0007 (10)
C6	0.0291 (15)	0.0226 (13)	0.0248 (13)	0.0045 (12)	0.0021 (12)	0.0015 (11)
C7	0.0252 (15)	0.0266 (14)	0.0265 (14)	0.0027 (12)	0.0022 (12)	−0.0005 (11)

Geometric parameters (Å, º)

S1—C2	1.752 (2)	C3—H3A	0.9900
S1—C4	1.839 (3)	C3—H3B	0.9900
S2—C2	1.739 (3)	C4—C5	1.514 (4)
S2—C3	1.812 (3)	C4—H4	1.0000
O1—N1	1.243 (3)	C5—H5A	0.9900
O2—N1	1.241 (3)	C5—H5B	0.9900
N1—C1	1.414 (3)	C6—H6A	0.9800
N2—C5	1.466 (3)	C6—H6B	0.9800
N2—C7	1.466 (3)	C6—H6C	0.9800
N2—C6	1.467 (3)	C7—H7A	0.9800
C1—C2	1.358 (3)	C7—H7B	0.9800
C1—H1	0.9500	C7—H7C	0.9800
C3—C4	1.516 (4)		
C2—S1—C4	94.61 (12)	C3—C4—S1	105.80 (18)
C2—S2—C3	95.60 (12)	C5—C4—H4	109.5
O2—N1—O1	123.1 (2)	C3—C4—H4	109.5
O2—N1—C1	119.5 (2)	S1—C4—H4	109.5
O1—N1—C1	117.4 (2)	N2—C5—C4	111.3 (2)
C5—N2—C7	110.0 (2)	N2—C5—H5A	109.4
C5—N2—C6	111.9 (2)	C4—C5—H5A	109.4
C7—N2—C6	109.6 (2)	N2—C5—H5B	109.4
C2—C1—N1	121.8 (2)	C4—C5—H5B	109.4
C2—C1—H1	119.1	H5A—C5—H5B	108.0
N1—C1—H1	119.1	N2—C6—H6A	109.5
C1—C2—S2	126.56 (19)	N2—C6—H6B	109.5
C1—C2—S1	118.00 (19)	H6A—C6—H6B	109.5
S2—C2—S1	115.43 (14)	N2—C6—H6C	109.5
C4—C3—S2	108.41 (18)	H6A—C6—H6C	109.5
C4—C3—H3A	110.0	H6B—C6—H6C	109.5
S2—C3—H3A	110.0	N2—C7—H7A	109.5
C4—C3—H3B	110.0	N2—C7—H7B	109.5
S2—C3—H3B	110.0	H7A—C7—H7B	109.5
H3A—C3—H3B	108.4	N2—C7—H7C	109.5
C5—C4—C3	114.2 (2)	H7A—C7—H7C	109.5
C5—C4—S1	108.15 (18)	H7B—C7—H7C	109.5
O2—N1—C1—C2	−5.7 (4)	S2—C3—C4—C5	166.65 (18)
O1—N1—C1—C2	173.5 (2)	S2—C3—C4—S1	47.8 (2)
N1—C1—C2—S2	1.8 (4)	C2—S1—C4—C5	−161.36 (17)
N1—C1—C2—S1	−178.79 (18)	C2—S1—C4—C3	−38.61 (19)
C3—S2—C2—C1	−174.4 (2)	C7—N2—C5—C4	159.4 (2)

C3—S2—C2—S1	6.22 (17)	C6—N2—C5—C4	−78.6 (3)
C4—S1—C2—C1	−162.7 (2)	C3—C4—C5—N2	−173.9 (2)
C4—S1—C2—S2	16.82 (16)	S1—C4—C5—N2	−56.4 (2)
C2—S2—C3—C4	−33.5 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···N2^{i}	0.95	2.44	3.354 (4)	161
C4—H4···O1^{ii}	1.00	2.44	3.272 (4)	140

Symmetry codes: (i) *x*−1/2, −*y*+3/2, −*z*; (ii) −*x*−1/2, −*y*+2, *z*+1/2.