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(E)-3-Dimethylamino-1-(1,3-thiazol-2-yl)prop-2-en-1-oneXin-Yu You,^a Yao-Jie Shi^b and Luo-Ting Yu^{b*}^aDepartment of Pharmaceutical and Bioengineering, School of Chemical Engineering, Sichuan University, Chengdu 610065, People's Republic of China, and^bState Key Laboratory of Biotherapy and Cancer Center, West China Hospital, West China Medical School, Sichuan University, Chengdu 610041, People's Republic of China

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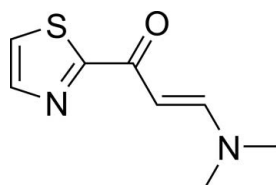
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Key indicators: single-crystal X-ray study; $T = 135$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.090; data-to-parameter ratio = 16.6.

In the title compound, $\text{C}_8\text{H}_{10}\text{N}_2\text{OS}$, the 3-(dimethylamino)-prop-2-en-1-one unit is approximately planar [give r.m.s. deviation] and the mean plane through the seven non-H atoms makes a dihedral angle of $8.88(3)^\circ$ with the thiazole ring. The carbonyl and ring $\text{C}=\text{N}$ double bonds adjacent to the carbonyl group are *trans* [$\text{N}-\text{C}-\text{C}-\text{O} = 172.31(15)^\circ$], while the conformation of the carbonyl and propene double bonds is *cis* [$\text{O}-\text{C}-\text{C}=\text{C} = 2.2(2)^\circ$]. In the crystal, short $\text{C}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\text{O}$ interactions together with $\text{C}-\text{H}\cdots\pi$ interactions generate a three-dimensional network.

Related literature

For the biological activity of enamino derivatives, see: Zeng (2010).



Experimental

Crystal data

 $\text{C}_8\text{H}_{10}\text{N}_2\text{OS}$ $M_r = 182.24$ Monoclinic, $P2_1/n$ $a = 5.6252(2)$ Å $b = 22.5957(8)$ Å $c = 7.5777(3)$ Å $\beta = 109.498(4)^\circ$ $V = 907.93(6)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.31$ mm⁻¹ $T = 135$ K $0.35 \times 0.30 \times 0.30$ mm

Data collection

Oxford Diffraction Xcalibur Eos diffractometer

Absorption correction: multi-scan

(CrysAlis PRO; Oxford

Diffraction, 2006)

 $T_{\min} = 0.974$, $T_{\max} = 1.000$

3683 measured reflections

1846 independent reflections

1565 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.018$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.035$ $wR(F^2) = 0.090$ $S = 1.06$

1846 reflections

111 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the S1/N1/C1–C3 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2}\cdots\text{N1}^{\text{i}}$	0.95	2.62	3.560 (2)	169
$\text{C6}-\text{H6}\cdots\text{O1}^{\text{ii}}$	0.95	2.60	3.462 (2)	151
$\text{C8}-\text{H8A}\cdots\text{O1}^{\text{ii}}$	0.98	2.31	3.269 (2)	167
$\text{C9}-\text{H9C}\cdots\text{O1}^{\text{iii}}$	0.98	2.51	3.433 (2)	157
$\text{C9}-\text{H9A}\cdots\text{Cg1}^{\text{iv}}$	0.98	2.93	3.549 (2)	122

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

Data collection: CrysAlis PRO (Oxford Diffraction, 2006); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: OLEX2 (Dolomanov *et al.*, 2009) and Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: OLEX2.

We thank the Analytical and Testing Center of Sichuan University for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VM2183).

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supporting information

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(E)-3-Dimethylamino-1-(1,3-thiazol-2-yl)prop-2-en-1-one

Xin-Yu You, Yao-Jie Shi and Luo-Ting Yu

S1. Comment

Enaminone derivatives are of great importance in organic synthesis (Zeng *et al.*, 2010). The title compound is one of the key intermediates in our synthetic investigations on compounds active against of anti-hepatocellular carcinoma and cancers. We report here its crystal structure.

In the title compound, C₈H₁₀N₂OS, (Fig.1), the dihedral angle between the thiazole ring and the mean plane through the remaining non-hydrogen atoms [O1/C4 - C9] is 8.88 (3) °. The two double bonds C1=N1 and C4=O1 are in *trans* (N1—C1—C4—O1: 172.31 (15) °), and the C4=O1 and C5=C6 double bonds are in *cis* (O1—C4—C5—C6: 2.2 (2) °).

In the crystal packing (Fig.2), the molecules are stabilized by short intermolecular C—H···N/O interactions and a C9—H9···Cg1 interaction (Cg1 is the centroid of the S1/N1/C1-C3 ring, Table 1).

S2. Experimental

A solution of 6.36 g (50.0 mmol) of 1-thiazol-2-yl-ethanone in 16.03 ml (17.87 g, 150.0 mmol) of DMF-DMA (dimethoxy-*N,N*-dimethylmethanamine) was stirred for 24 h at 114°C. The solvent was evaporated and the title compound was recrystallized from ethanol. Yield: 6.83 g (75%). Crystals suitable for X-ray analysis were obtained by slow evaporation from a solution of ethyl acetate.

S3. Refinement

All H atoms were positioned geometrically (for methyl, C—H = 0.98 Å; for the other H atoms, C—H = 0.95 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = kU_{\text{eq}}(\text{C})$, where $k = 1.5$ for methyl and 1.2 for all other H atoms.

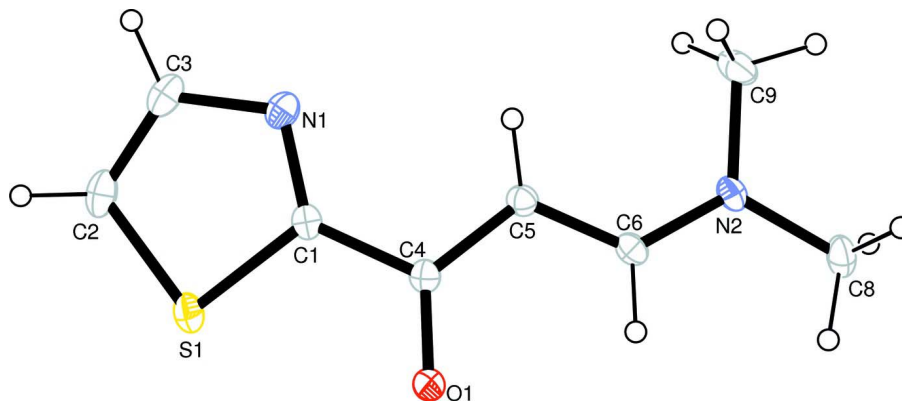


Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

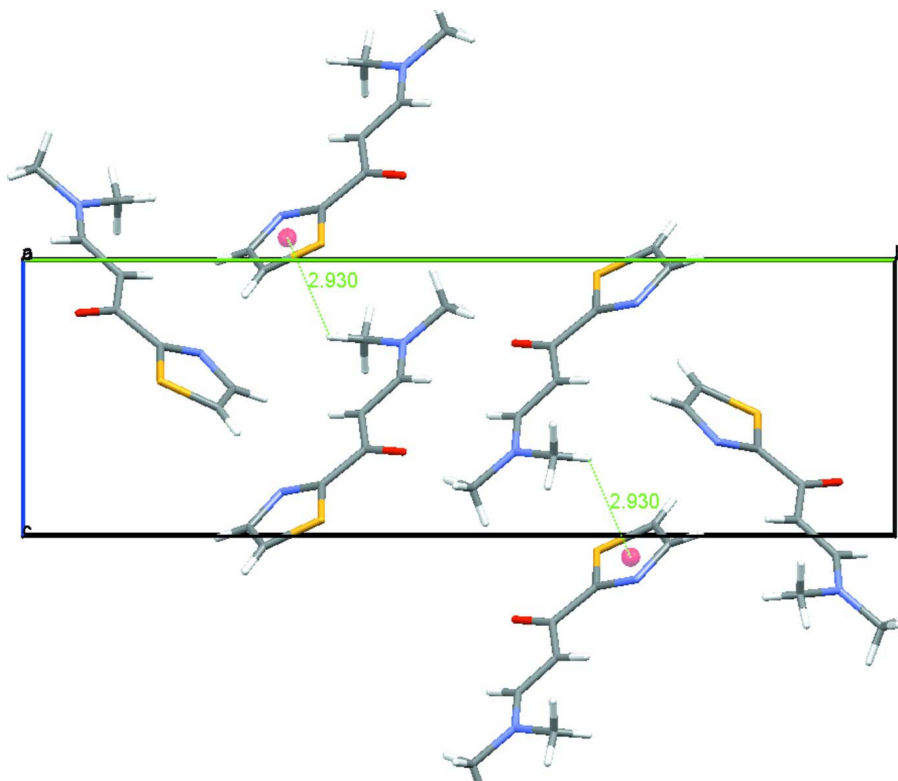


Figure 2

A crystal packing diagram of the title compound. The dashed lines represent C—H... π interactions.

(E)-3-Dimethylamino-1-(1,3-thiazol-2-yl)prop-2-en-1-one

Crystal data

$C_8H_{10}N_2OS$

$M_r = 182.24$

Monoclinic, $P2_1/n$

$a = 5.6252$ (2) Å

$b = 22.5957$ (8) Å

$c = 7.5777$ (3) Å

$\beta = 109.498$ (4)°

$V = 907.93$ (6) Å³

$Z = 4$

$F(000) = 384$

$D_x = 1.333$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å

Cell parameters from 1788 reflections

$\theta = 3.0$ – 28.8 °

$\mu = 0.31$ mm⁻¹

$T = 135$ K

Block, yellow

$0.35 \times 0.30 \times 0.30$ mm

Data collection

Oxford Diffraction Xcalibur Eos
diffractometer

Radiation source: Enhanced (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.0874 pixels mm⁻¹

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2006)

$T_{\min} = 0.974$, $T_{\max} = 1.000$

3683 measured reflections

1846 independent reflections

1565 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 3.0$ °

$h = -4 \rightarrow 7$

$k = -13 \rightarrow 28$

$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.090$
 $S = 1.06$
 1846 reflections
 111 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
 $w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.2132P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent Technologies, Version 1.171.35.19, empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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	0.48299 (8)	0.15794 (2)	0.44117 (6)	0.02965 (16)
O1	0.5387 (2)	0.06419 (5)	0.19443 (16)	0.0293 (3)
N1	0.8366 (2)	0.20354 (6)	0.34119 (19)	0.0269 (3)
N2	0.9036 (2)	0.06400 (6)	-0.19641 (18)	0.0243 (3)
C1	0.6877 (3)	0.15752 (7)	0.3145 (2)	0.0214 (4)
C2	0.6091 (3)	0.22388 (9)	0.5379 (2)	0.0347 (4)
H2	0.5586	0.2455	0.6269	0.042*
C3	0.7920 (3)	0.24077 (8)	0.4690 (2)	0.0336 (4)
H3	0.8841	0.2765	0.5073	0.040*
C4	0.6732 (3)	0.10684 (7)	0.1844 (2)	0.0215 (4)
C5	0.8103 (3)	0.11210 (7)	0.0581 (2)	0.0226 (4)
H5	0.9141	0.1456	0.0619	0.027*
C6	0.7890 (3)	0.06752 (7)	-0.0696 (2)	0.0221 (4)
H6	0.6800	0.0357	-0.0667	0.026*
C8	0.8565 (3)	0.01509 (8)	-0.3283 (2)	0.0308 (4)
H8A	0.7469	-0.0140	-0.2980	0.046*
H8B	1.0169	-0.0037	-0.3203	0.046*
H8C	0.7740	0.0300	-0.4556	0.046*
C9	1.0815 (3)	0.10914 (9)	-0.2102 (2)	0.0342 (4)
H9A	1.0028	0.1482	-0.2203	0.051*
H9B	1.1291	0.1017	-0.3214	0.051*
H9C	1.2324	0.1078	-0.0982	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0328 (3)	0.0320 (3)	0.0283 (3)	0.00526 (19)	0.0159 (2)	-0.00177 (19)
O1	0.0389 (7)	0.0259 (7)	0.0287 (6)	-0.0064 (6)	0.0186 (5)	-0.0032 (5)
N1	0.0274 (7)	0.0248 (8)	0.0253 (7)	0.0021 (6)	0.0046 (6)	-0.0015 (6)
N2	0.0267 (7)	0.0290 (8)	0.0204 (7)	0.0021 (6)	0.0121 (6)	0.0029 (6)
C1	0.0214 (8)	0.0238 (9)	0.0178 (8)	0.0062 (7)	0.0050 (6)	0.0029 (7)
C2	0.0428 (10)	0.0309 (10)	0.0292 (9)	0.0115 (9)	0.0102 (8)	-0.0059 (8)
C3	0.0378 (10)	0.0255 (10)	0.0318 (9)	0.0040 (8)	0.0040 (8)	-0.0064 (8)
C4	0.0232 (8)	0.0223 (8)	0.0179 (8)	0.0028 (7)	0.0052 (6)	0.0026 (7)
C5	0.0238 (8)	0.0235 (9)	0.0209 (8)	-0.0005 (7)	0.0079 (7)	0.0018 (7)
C6	0.0225 (8)	0.0253 (9)	0.0190 (8)	0.0021 (7)	0.0077 (6)	0.0055 (7)
C8	0.0439 (10)	0.0298 (10)	0.0247 (9)	0.0073 (8)	0.0193 (8)	0.0019 (8)
C9	0.0297 (9)	0.0466 (12)	0.0299 (9)	-0.0064 (9)	0.0148 (8)	0.0032 (9)

Geometric parameters (Å, °)

S1—C1	1.7282 (16)	C3—H3	0.9500
S1—C2	1.707 (2)	C4—C5	1.420 (2)
O1—C4	1.2432 (19)	C5—H5	0.9500
N1—C1	1.308 (2)	C5—C6	1.374 (2)
N1—C3	1.368 (2)	C6—H6	0.9500
N2—C6	1.3264 (19)	C8—H8A	0.9800
N2—C8	1.454 (2)	C8—H8B	0.9800
N2—C9	1.457 (2)	C8—H8C	0.9800
C1—C4	1.495 (2)	C9—H9A	0.9800
C2—H2	0.9500	C9—H9B	0.9800
C2—C3	1.355 (3)	C9—H9C	0.9800
C2—S1—C1	89.13 (9)	C6—C5—C4	118.28 (15)
C1—N1—C3	109.85 (14)	C6—C5—H5	120.9
C6—N2—C8	121.51 (14)	N2—C6—C5	127.23 (16)
C6—N2—C9	121.45 (15)	N2—C6—H6	116.4
C8—N2—C9	117.04 (13)	C5—C6—H6	116.4
N1—C1—S1	114.87 (12)	N2—C8—H8A	109.5
N1—C1—C4	127.05 (14)	N2—C8—H8B	109.5
C4—C1—S1	118.07 (12)	N2—C8—H8C	109.5
S1—C2—H2	125.0	H8A—C8—H8B	109.5
C3—C2—S1	109.92 (14)	H8A—C8—H8C	109.5
C3—C2—H2	125.0	H8B—C8—H8C	109.5
N1—C3—H3	121.9	N2—C9—H9A	109.5
C2—C3—N1	116.23 (17)	N2—C9—H9B	109.5
C2—C3—H3	121.9	N2—C9—H9C	109.5
O1—C4—C1	116.99 (14)	H9A—C9—H9B	109.5
O1—C4—C5	125.77 (15)	H9A—C9—H9C	109.5
C5—C4—C1	117.22 (14)	H9B—C9—H9C	109.5
C4—C5—H5	120.9		

S1—C1—C4—O1	-9.54 (19)	C1—C4—C5—C6	-176.53 (13)
S1—C1—C4—C5	169.33 (11)	C2—S1—C1—N1	-0.70 (13)
S1—C2—C3—N1	0.2 (2)	C2—S1—C1—C4	-179.08 (13)
O1—C4—C5—C6	2.2 (2)	C3—N1—C1—S1	0.92 (17)
N1—C1—C4—O1	172.31 (15)	C3—N1—C1—C4	179.13 (15)
N1—C1—C4—C5	-8.8 (2)	C4—C5—C6—N2	-179.08 (15)
C1—S1—C2—C3	0.25 (14)	C8—N2—C6—C5	-177.66 (15)
C1—N1—C3—C2	-0.7 (2)	C9—N2—C6—C5	2.1 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the S1/N1/C1—C3 ring.

<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>
C2—H2 \cdots N1 ⁱ	0.95	2.62	3.560 (2)	169
C6—H6 \cdots O1 ⁱⁱ	0.95	2.60	3.462 (2)	151
C8—H8 <i>A</i> \cdots O1 ⁱⁱ	0.98	2.31	3.269 (2)	167
C9—H9 <i>C</i> \cdots O1 ⁱⁱⁱ	0.98	2.51	3.433 (2)	157
C9—H9 <i>A</i> \cdots Cg1 ^{iv}	0.98	2.93	3.549 (2)	122

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