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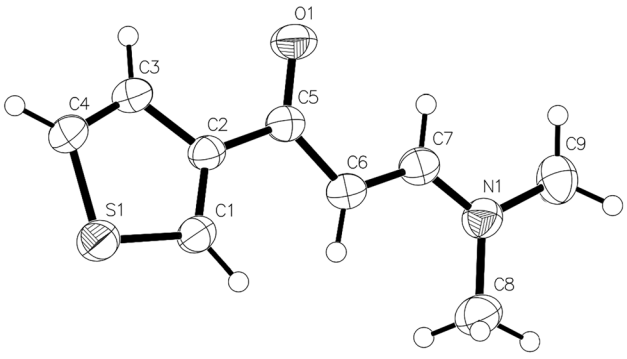
Crystal structure of (*E*)-3-(dimethylamino)-1-(thiophen-3-yl)prop-2-en-1-one, $C_9H_{11}NOS$



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Abstract

$C_9H_{11}NOS$, monoclinic, $P2_1/n$ (no. 14), a = 6.0194(4) Å, b = 20.9249(13) Å, c = 7.7103(5) Å, β = 107.783(2)°, V = 924.75(10) Å^3, Z = 4, $R_{gt}(F)$ = 0.0373, $wR_{ref}(F^2)$ = 0.1061, T = 223(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.31 × 0.17 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.30 mm^{-1}
Diffractometer, scan mode:	PHOTON II M14, φ and ω
θ_{max}, completeness:	28.6°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	29,440, 2329, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2031
$N(param)_{refined}$:	111
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
S1	0.83574 (7)	0.24100 (2)	1.03605 (6)	0.04767 (15)
C1	0.7464 (3)	0.16940 (7)	0.9327 (2)	0.0370 (3)
H1	0.8411	0.1422	0.8885	0.044*
C2	0.5199 (2)	0.15642 (6)	0.92212 (18)	0.0309 (3)
C3	0.4215 (2)	0.20653 (7)	1.0002 (2)	0.0370 (3)
H3	0.2669	0.2059	1.0042	0.044*
C4	0.5714 (3)	0.25511 (7)	1.0683 (2)	0.0392 (3)
H4	0.5351	0.2916	1.1255	0.047*
C5	0.3847 (2)	0.09877 (7)	0.83713 (19)	0.0332 (3)
C6	0.5110 (2)	0.04526 (7)	0.79932 (19)	0.0335 (3)
H6	0.6744	0.0466	0.8293	0.040*
C7	0.3911 (2)	−0.00762 (7)	0.71938 (19)	0.0346 (3)
H7	0.2279	−0.0053	0.6905	0.042*
O1	0.16866 (18)	0.10012 (6)	0.80304 (18)	0.0481 (3)
N1	0.4754 (2)	−0.06210 (6)	0.67699 (18)	0.0395 (3)
C8	0.7238 (3)	−0.07201 (8)	0.7171 (3)	0.0482 (4)
H8A	0.7873	−0.0400	0.6545	0.072*
H8B	0.7520	−0.1142	0.6763	0.072*
H8C	0.7986	−0.0685	0.8473	0.072*
C9	0.3200 (3)	−0.11355 (8)	0.5873 (2)	0.0472 (4)
H9A	0.3317	−0.1485	0.6723	0.071*
H9B	0.3639	−0.1285	0.4833	0.071*
H9C	0.1607	−0.0979	0.5466	0.071*

Source of material

The title compound of (*E*)-3-(dimethylamino)-1-(thiophen-3-yl)prop-2-en-1-one was prepared as previously described [5] via condensation of the 3-acetylthiophene and dimethylforamide dimethylacetal (DMF–DMA). 3-Acetylthiphene (10 mmol, 1.26 g) was dissolved in an excess amount of DMF–DMA (6 mL) and the reaction mixture was heated at 80 °C for 6 h. After the completion of the reaction (checked by TLC), the mixture was cooled to room temperature and was poured into distilled water (70 mL), which was extracted with methylene chloride (60 mL × 2). The combined organic layers were dried over $MgSO_4$. Filtration and evaporation of the organic solvent gave a crude solid, which was recrystallized in methanol to afford crystals used in this X-ray diffraction study.

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Experimental details

Data collections and reduction were carried out using the Bruker software APEX2 and SAINT including SADABS [1]. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

N,*N*-dimethylenamino (β-aminovinyl) ketones are versatile synthetic intermediates which provide access to various heterocyclic compounds with diverse biological properties in medicinal chemistry [6–8]. According to a recent review, the β-aminovinyl ketones possess both 'enamine' and 'enone' systems in the molecules [9]. Therefore they can react with nucleophiles as well as electrophiles to serve as scaffolds for fused heterocycles. As a continuation of our research program to expand the use of novel heterocyclic compounds [10, 11], we designed new β-aminovinyl ketone intermediate, which can be used to construct novel heterocyclic rings.

In the title compound, the whole molecule is slightly twisted and dihedral angles formed by thiophenyl ring system ([C1, C2, C3, C4, S1]; r.m.s. deviations 0.003 Å) with the central enone group ([C5, C6, C7, N1]; r.m.s. deviations 0.007 Å) is 15.33 (2)°. The double bond of C6=C7 bond in the central enone group adopts a *trans* configuration, which was defined by the dihedral angle of –179.09(1)° for C5–C6–C7–N1. In the crystal, pairs of weak C1–H1···O1 and C3–H3···S1 hydrogen bonds form a dimer.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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