



Crystal structure of (2*E*)-3-[4-(dimethylamino)-phenyl]-1-(thiophen-2-yl)prop-2-en-1-one

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Keywords: crystal structure; chalcone thiophene derivative; Hirshfeld surface analysis; *in silico* evaluation.

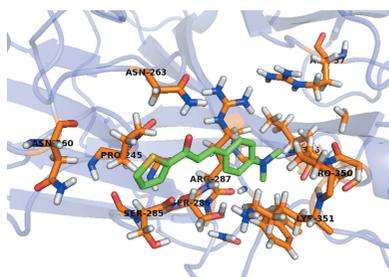
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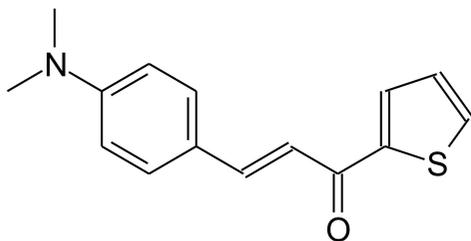
The equimolar reaction between 4-(dimethylamino)benzaldehyde and 2-acetylthiophene in basic ethanolic solution yields the title compound, C₁₅H₁₅NOS, whose molecular structure matches the asymmetric unit. The molecule is not planar, the dihedral angle between the aromatic and the thiophene rings being 11.4 (2)°. In the crystal, molecules are linked by C—H···O and weak C—H···S interactions along [100], forming R₂²(8) rings, and by weak C—H···O interactions along [010], forming chains with a C(6) graph-set motif. In addition, molecules are connected into centrosymmetric dimers by weak C—H···π interactions, as indicated by the Hirshfeld surface analysis. The most important contributions for the crystal structure are the H···H (46.50%) and H···C (23.40%) interactions. The crystal packing resembles a herringbone arrangement when viewed along [100]. A molecular docking calculation of the title compound with the neuraminidase enzyme was carried out. The enzyme shows (ASN263)N—H···O, (PRO245)C—H···Cg(thiophene ring) and (AGR287)C—H···N intermolecular interactions with the title compound. The crystal structure was refined as a two-component twin with a fractional contribution to the minor domain of 0.0181 (8).

1. Chemical context

Chalcone derivatives are compounds with an aromatic conjugated enone as the main fragment and are synthesized by hydroxide-catalysed aldol condensation between an aromatic aldehyde and a ketone. Some of the first preparative methods of the aldol condensation were reported in the second half of the 19th Century (Claisen & Claparède, 1881; Schmidt, 1881) and the experimental procedure remains the same to the present time. Chalcone compounds can be obtained from a great number of starting materials, resulting in a class of compounds with a wide range of properties and applications, specially in the medicinal chemistry. Several 4-dialkylamino-chalcones have shown antiproliferative activity on cancer cell lines and one method to monitor the chalcone–protein interaction, *e.g.* tubulin proteins, is the chalcone's fluorescence (Zhou *et al.*, 2016). Another example of the pharmacological background for the title compound and its derivatives is the anti-influenza viral activity through the neuraminidase enzymatic inhibition *in vitro* (Kinger *et al.*, 2012). Thus, the crystal structure determination of chalcone-based molecules is an intensive research area, in particular for its contributions in medicinal chemistry. As part of our studies in this field, we



describe herein the crystal structure, the Hirshfeld surface analysis and the molecular docking evaluation of the title compound.



2. Structural commentary

In the crystal structure of the title compound, a chalcone-thiophene derivative, the asymmetric unit contains one crystallographically independent molecule (Fig. 1). The molecule is not planar: the r.m.s deviations from the mean plane of the non-H atoms range from -0.158 (3) Å for C3 to 0.1318 (15) Å for S1 and the dihedral angle between the benzene and thiophene rings amounts to 11.4 (2)°. In addition, the plane through the amino group atoms (C7/C8/N1) is rotated by 9.7 (6)° with respect to the plane of the aromatic ring. Finally, the molecule shows the *E* configuration about the C9–C10 bond.

3. Supramolecular features

In the crystal, the molecules are connected by very weak C13–H13···O1ⁱ and C14–H14···S1ⁱ hydrogen-bonding interactions (see Table 1 for symmetry codes), forming rings with an $R_2^2(8)$ graph-set motif. The $R_2^2(8)$ rings are the subunits of the periodic arrangement along [100] and one very weak H7···H2ⁱ contact is also observed [H···H = 2.26 Å]. The molecular units are also linked by very weak C15–H15···O1ⁱⁱ links into chains along [010] with a $C(6)$ graph-set motif (Fig. 2; Table 1). Additionally, the molecules are connected into centrosymmetric dimers by very weak C–H··· π interactions involving the thiophene ring (Fig. 3; Table 1). The intermolecular contacts are slightly longer than the sum of the van der Waals radii for the respective atoms (Bondi, 1964; Rowland & Taylor, 1996) and suggest weak interactions only.

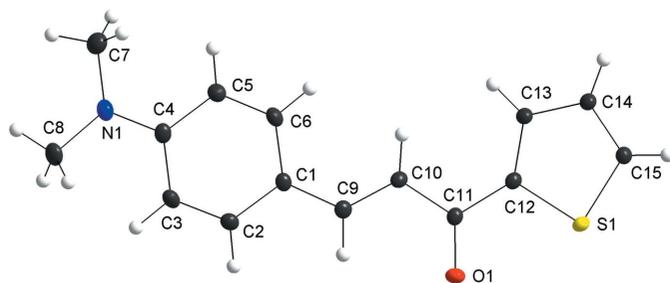


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 40% probability level.

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

C_g is the centroid of the S1/C12–C15 thiophene ring.

| <i>D</i> –H··· <i>A</i> | <i>D</i> –H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> –H··· <i>A</i> |
|---------------------------------------|-------------|---------------|-----------------------|-------------------------|
| C13–H13···O1 ⁱ | 0.95 | 2.65 | 3.451 (4) | 142 |
| C14–H14···S1 ⁱ | 0.95 | 3.00 | 3.779 (3) | 141 |
| C15–H15···O1 ⁱⁱ | 0.95 | 2.57 | 3.291 (4) | 133 |
| C8–H8···C _g ⁱⁱⁱ | 0.98 | 2.64 | 3.457 (4) | 141 |

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

4. Hirshfeld surface analysis

The Hirshfeld surface analysis (Hirshfeld, 1977) of the crystal structure suggests that the contribution of the H···H intermolecular interactions to the crystal packing amounts to 46.50% and the contribution of the H···C interactions

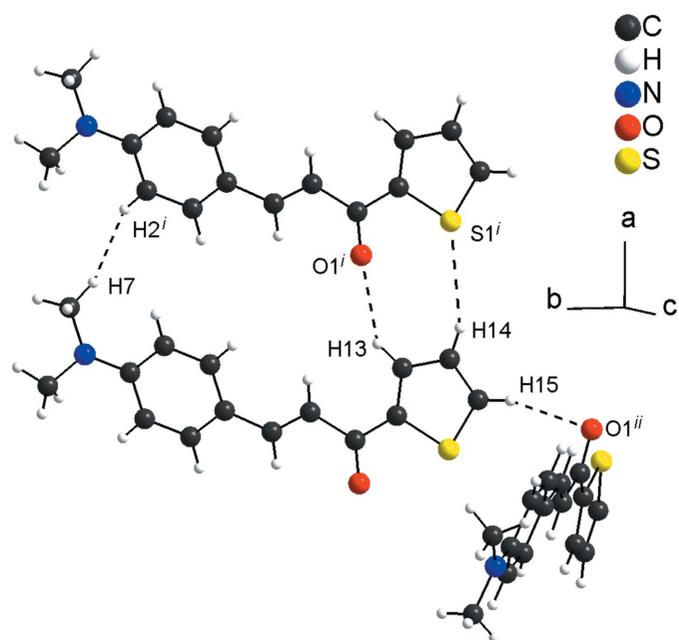


Figure 2
Graphical representation of the weak intermolecular C–H···O, C–H···S and H···H interactions (dashed lines) in the crystal structure of the title compound. [Symmetry codes: (i) $x + 1, y, z$; (ii) $-x - \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.]

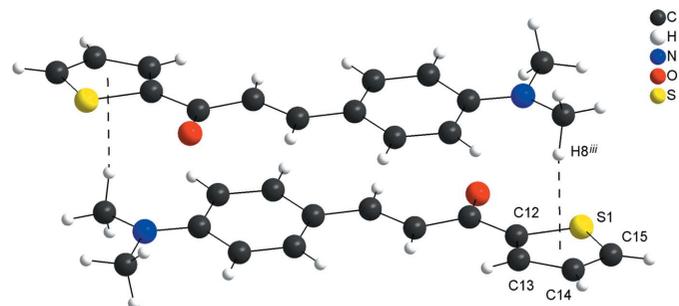
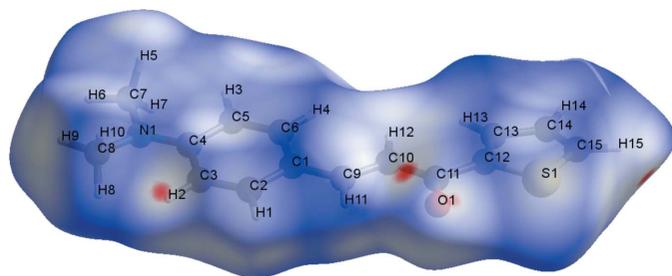


Figure 3
Graphical representation of the weak intermolecular C–H··· π interactions (dashed lines) in the crystal structure of the title compound, forming a centrosymmetric dimer. [Symmetry code: (iii) $-x, -y, -z + 1$.]

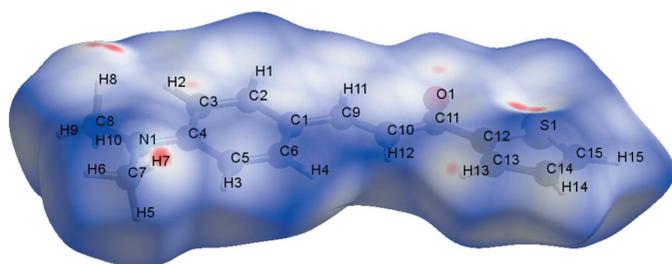

Figure 4

A graphical representation of the Hirshfeld surface (d_{norm}) for the title compound. The surface is drawn with transparency and all atoms are labelled. The surface regions with strongest intermolecular interactions for atoms H2, H15 and O1 are shown in magenta.

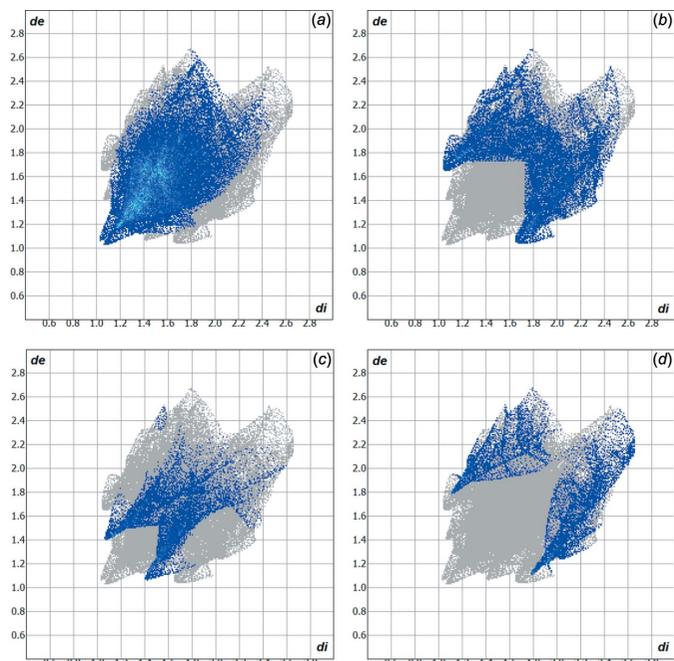
amounts to 23.40%. Other important intermolecular contacts for the cohesion of the structure are (values given in %): $\text{H}\cdots\text{O} = 10.80$ and $\text{H}\cdots\text{S} = 10.00$. Graphical representations of the Hirshfeld surface with transparency and labelled atoms (Figs. 4 and 5) indicate, in a magenta colour, the locations of the strongest intermolecular contacts, e.g. the H2, H7, H13, H15 and O1 atoms. The $\text{C}-\text{H}\cdots\pi$ interaction is also well represented in the Hirshfeld surface (for details, compare Figs. 3 and 5). The $\text{H}\cdots\text{H}$, $\text{H}\cdots\text{C}$, $\text{H}\cdots\text{O}$ and $\text{H}\cdots\text{S}$ contributions to the crystal packing are shown as a Hirshfeld surface two-dimensional fingerprint plot with cyan dots. The d_e (y axis) and d_i (x axis) values are the closest external and internal distances (values given in Å) from given points on the Hirshfeld surface contacts (Fig. 6; Wolff *et al.*, 2012).

5. Molecular docking evaluation

In addition, a lock-and-key supramolecular analysis between the neuraminidase enzyme, whose inhibition is believed to be a key point to block the influenza viral infection (Kinger *et al.*, 2012), and the title compound was performed. The semi-empirical equilibrium energy of the title compound was obtained using the PM6 Hamiltonian and the experimental bond lengths were conserved. The calculated parameters were: heat of formation = $139.28 \text{ kJ mol}^{-1}$, gradient normal = 0.62031, HOMO = -8.96 eV , LUMO = -0.866 eV and energy gap = 7.421 eV (Stewart, 2013). The rigid molecular docking was carried out with the *GOLD* software (Jones *et al.*, 1997)


Figure 5

A graphical representation of the Hirshfeld surface (d_{norm}) for the title compound. The surface is drawn with transparency and all atoms are labelled. The surface regions with strongest intermolecular interactions for atoms H7, H8, H13 and O1, and for the thiophene ring, are shown in magenta.

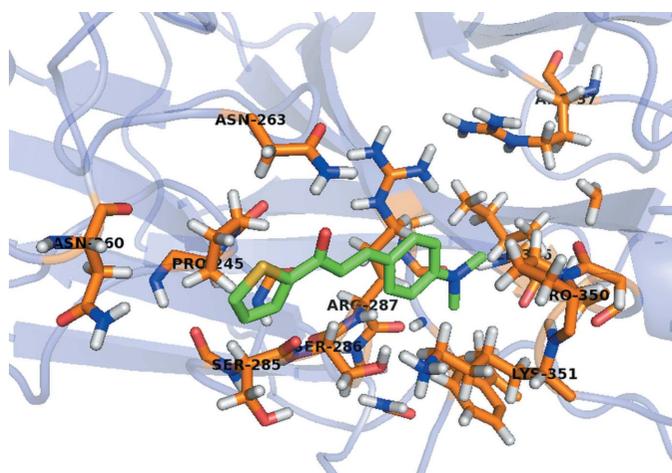

Figure 6

Hirshfeld surface two-dimensional fingerprint plots for the title compound showing the (a) $\text{H}\cdots\text{H}$, (b) $\text{H}\cdots\text{C}$, (c) $\text{O}\cdots\text{H}$ and (d) $\text{H}\cdots\text{S}$ contacts in detail (cyan dots). The contributions of the interactions to the crystal packing amount to 46.50, 23.40, 10.80 and 10.00%, respectively. The d_e (y axis) and d_i (x axis) values are the closest external and internal distances (values in Å) from given points on the Hirshfeld surface contacts.

using the *ChemPLP* score function (Chen, 2015). The chalcone thiophene derivative and the active site of the neuraminidase match (Fig. 7) and the structure–activity relationship can be assumed by the following observed intermolecular interactions ($\text{H}\cdots\text{A}$ distance values given in Å): (*ASN263*) $\text{N}-\text{H}\cdots\text{O1}$ ($d = 1.796$), (*PRO245*) $\text{C}-\text{H}\cdots\text{Cg}(\text{thiophene ring})$ ($d = 2.829$) and (*AGR287*) $\text{C}-\text{H}\cdots\text{N1}$ ($d = 2.620$) (Fig. 8). More details about the *in silico* evaluation, with additional references, can be found in the *Supporting Information*. For the intermolecular interactions, it is important to report that the $\text{H}\cdots\text{Cg}(\text{thiophene ring})$ contact is observed in the structure interpretation, by the centrosymmetric dimeric arrangement of the molecules (Figs. 3 and 9), in the Hirshfeld surface analysis (Fig. 5) and in the molecular docking evaluation (Fig. 8).

6. Database survey

Chalcone-thiophene derivatives have some molecular structural features in common, namely the nearly planar geometry, as a result of the sp^2 -hybridized C atoms of the main fragment, and the weak intermolecular interactions, e.g. $\text{H}\cdots\text{H}$, $\text{H}\cdots\text{C}$ or $\pi-\pi$ contacts. One example for comparison with the title compound is the crystal structure of the compound 3-(4-methylphenyl)-1-(3-thienyl)-2-propen-1-one (Li & Su, 1993). In both of the structures, the molecules are linked by weak interactions into centrosymmetric dimers and the crystal

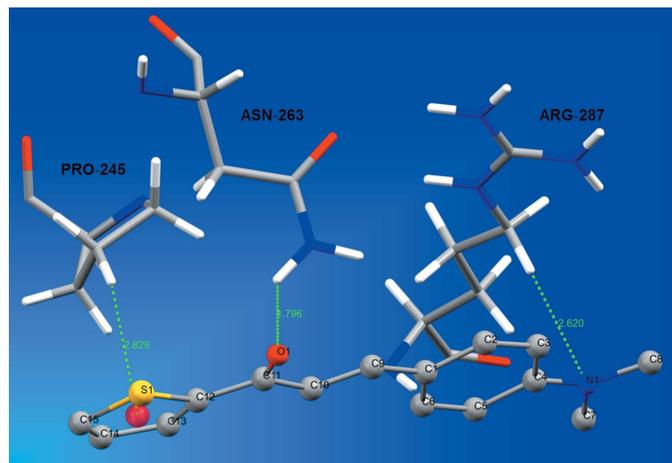

Figure 7

Graphical representation of the lock-and-key model for the title compound, with the molecular main fragment in green, and the neuraminidase structure, with selected amino acids residues, in stick model. The structure of the enzyme is simplified for clarity.

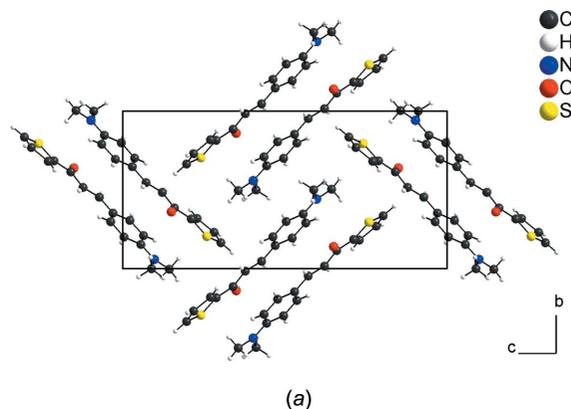
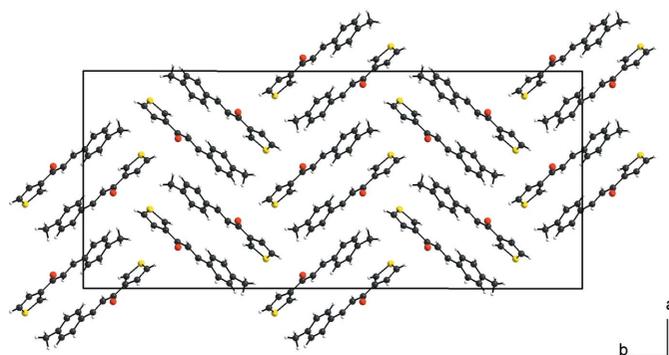
packing shows a herringbone motif: for the title compound this molecular arrangement is clear when looking along the [100] direction (Fig. 9a) and for the above-mentioned 3-thienyl derivative, along [001] (Fig. 9b).

7. Synthesis and crystallization

All starting materials are commercially available and were used without further purification. The synthesis of the title compound was adapted from a previously reported procedure (Claisen & Claparède, 1881; Schmidt, 1881; Zhou *et al.*, 2016). In a hydroxide-catalysed reaction, a mixture of 4-(dimethyl-amino)benzaldehyde (10 mmol) and 2-acetylthiophene (10 mmol) in ethanol (80 mL) was stirred under room temperature for 4 h. After cooling in an ice bath and filtering,


Figure 8

Intermolecular interactions between the title compound and the neuraminidase enzyme. The interactions are shown as dashed lines and the structure of the enzyme is simplified for clarity.


(a)

(b)
Figure 9

Section of the crystal structures of (a) the title compound viewed along [100], and (b) the 3-(4-methylphenyl)-1-(3-thienyl)-2-propen-1-one compound (Li & Su, 1993) viewed along [001], showing the herringbone motif.

an orange solid was obtained. Orange crystals were grown from the solution after 24 h.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were located in a difference-Fourier map but were positioned with idealized geometry and were refined with isotropic displacement parameters using a riding model (HFIX command) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. A rotating model was used for the methyl groups. The crystal was refined as a two-component twin {twin law: two-axis (001) [105], BASF = 0.0181 (8)}.

Acknowledgements

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Table 2
Experimental details.

| | |
|--|--|
| Crystal data | |
| Chemical formula | C ₁₅ H ₁₅ NOS |
| <i>M_r</i> | 257.34 |
| Crystal system, space group | Monoclinic, <i>P</i> 2 ₁ / <i>n</i> |
| Temperature (K) | 120 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 6.2405 (4), 9.9975 (6), 20.7815 (13) |
| β (°) | 93.097 (2) |
| <i>V</i> (Å ³) | 1294.65 (14) |
| <i>Z</i> | 4 |
| Radiation type | Mo <i>K</i> α |
| μ (mm ⁻¹) | 0.24 |
| Crystal size (mm) | 0.53 × 0.16 × 0.09 |
| Data collection | |
| Diffractometer | Bruker APEXII CCD area detector |
| Absorption correction | Multi-scan (<i>SADABS</i> ; Bruker, 2014) |
| <i>T</i> _{min} , <i>T</i> _{max} | 0.885, 0.979 |
| No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections | 50820, 3381, 3116 |
| <i>R</i> _{int} | 0.031 |
| (sin θ/λ) _{max} (Å ⁻¹) | 0.677 |
| Refinement | |
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.068, 0.182, 1.14 |
| No. of reflections | 3381 |
| No. of parameters | 166 |
| H-atom treatment | H-atom parameters constrained |
| Δρ _{max} , Δρ _{min} (e Å ⁻³) | 1.16, -0.81 |

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015), *WinGX* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006), *GOLD* (Chen *et al.*, 2015), *Crystal Explorer* (Wolff *et al.*, 2012), *pubCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

experimental facilities and Guilherme Alves de Moraes for the data collection (Federal University of Santa Maria, Brazil).

References

- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2014). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chen, Y.-C. (2015). *Trends Pharmacol. Sci.* **36**, 78–95.
- Claisen, L. & Claparède, A. (1881). *Chem. Ber.* **14**, 2460–2468.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Hirshfeld, H. L. (1977). *Theor. Chim. Acta*, **44**, 129–138.
- Jones, G., Willett, P., Glen, R. C., Leach, A. R. & Taylor, R. (1997). *J. Mol. Biol.* **267**, 727–748.
- Kinger, M., Park, Y. D., Park, J. H., Hur, M. G., Jeong, H. J., Park, S. J., Lee, W. S., Kim, S. W. & Yang, S. D. (2012). *Arch. Pharm. Res.* **35**, 633–638.
- Li, Z. & Su, G. (1993). *Acta Cryst.* **C49**, 1075–1077.
- Rowland, R. S. & Taylor, R. (1996). *J. Phys. Chem.* **100**, 7384–7391.
- Sheldrick, J. G. (1881). *Chem. Ber.* **14**, 1459–1461.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Sheldrick, G. M. (2015). *Acta Cryst.* **C71**, 3–8.
- Stewart, J. J. (2013). *J. Mol. Model.* **19**, 1–32.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Wolff, S. K., Grimwood, D. J., McKinnon, J. J., Turner, M. J., Jayatilaka, D. & Spackman, M. A. (2012). *Crystal Explorer*. University of Western Australia, Perth, Australia.
- Zhou, B., Jiang, P., Lu, J. & Xing, C. (2016). *Arch. Pharm. Chem. Life Sci.* **349**, 539–552.

supporting information

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2016* (Sheldrick, 2015) and *WinGX* (Farrugia, 2012); molecular graphics: *DIAMOND* (Brandenburg, 2006), *GOLD* (Chen *et al.*, 2015) and *Crystal Explorer* (Wolff *et al.*, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010) and *enCIFer* (Allen *et al.*, 2004).

(2*E*)-3-[4-(Dimethylamino)phenyl]-1-(thiophen-2-yl)prop-2-en-1-one

Crystal data

| | |
|----------------------------------|---|
| $C_{15}H_{15}NOS$ | $F(000) = 544$ |
| $M_r = 257.34$ | $D_x = 1.320 \text{ Mg m}^{-3}$ |
| Monoclinic, $P2_1/n$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| $a = 6.2405 (4) \text{ \AA}$ | Cell parameters from 9620 reflections |
| $b = 9.9975 (6) \text{ \AA}$ | $\theta = 2.3\text{--}28.7^\circ$ |
| $c = 20.7815 (13) \text{ \AA}$ | $\mu = 0.24 \text{ mm}^{-1}$ |
| $\beta = 93.097 (2)^\circ$ | $T = 120 \text{ K}$ |
| $V = 1294.65 (14) \text{ \AA}^3$ | Block, orange |
| $Z = 4$ | $0.53 \times 0.16 \times 0.09 \text{ mm}$ |

Data collection

| | |
|--|--|
| Bruker APEXII CCD area detector diffractometer | 50820 measured reflections |
| Radiation source: fine-focus sealed tube, Bruker APEXII CCD area detector | 3381 independent reflections |
| φ and ω scans | 3116 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2014) | $R_{\text{int}} = 0.031$ |
| $T_{\text{min}} = 0.885$, $T_{\text{max}} = 0.979$ | $\theta_{\text{max}} = 28.8^\circ$, $\theta_{\text{min}} = 2.3^\circ$ |
| | $h = -7 \rightarrow 8$ |
| | $k = -13 \rightarrow 13$ |
| | $l = -28 \rightarrow 28$ |

Refinement

| | |
|---------------------------------|---|
| Refinement on F^2 | 0 restraints |
| Least-squares matrix: full | Primary atom site location: structure-invariant direct methods |
| $R[F^2 > 2\sigma(F^2)] = 0.068$ | Secondary atom site location: difference Fourier map |
| $wR(F^2) = 0.182$ | Hydrogen site location: inferred from neighbouring sites |
| $S = 1.14$ | |
| 3381 reflections | |
| 166 parameters | |

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + 7.019P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.81 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refined as a 2-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|--------------|--------------|----------------------------------|
| C1 | -0.1108 (5) | 0.1473 (3) | 0.52979 (15) | 0.0159 (6) |
| C2 | -0.2422 (5) | 0.1940 (4) | 0.47768 (16) | 0.0189 (6) |
| H1 | -0.382513 | 0.157938 | 0.471036 | 0.023* |
| C3 | -0.1753 (5) | 0.2904 (4) | 0.43572 (15) | 0.0193 (6) |
| H2 | -0.269640 | 0.319201 | 0.401045 | 0.023* |
| C4 | 0.0319 (5) | 0.3467 (3) | 0.44378 (15) | 0.0178 (6) |
| C5 | 0.1659 (5) | 0.2992 (3) | 0.49602 (16) | 0.0188 (6) |
| H3 | 0.306383 | 0.334799 | 0.502939 | 0.023* |
| C6 | 0.0956 (5) | 0.2024 (3) | 0.53690 (15) | 0.0181 (6) |
| H4 | 0.190279 | 0.171734 | 0.571104 | 0.022* |
| C7 | 0.3103 (6) | 0.5007 (4) | 0.4112 (2) | 0.0295 (8) |
| H5 | 0.318786 | 0.553158 | 0.451058 | 0.044* |
| H6 | 0.336378 | 0.559200 | 0.374572 | 0.044* |
| H7 | 0.418747 | 0.429736 | 0.413870 | 0.044* |
| C8 | -0.0527 (6) | 0.5041 (4) | 0.35627 (17) | 0.0241 (7) |
| H8 | -0.117365 | 0.435643 | 0.327624 | 0.036* |
| H9 | 0.021999 | 0.570086 | 0.330728 | 0.036* |
| H10 | -0.165267 | 0.548805 | 0.379384 | 0.036* |
| C9 | -0.1918 (5) | 0.0467 (3) | 0.57189 (15) | 0.0168 (6) |
| H11 | -0.336808 | 0.020903 | 0.562907 | 0.020* |
| C10 | -0.0899 (5) | -0.0158 (3) | 0.62222 (15) | 0.0163 (6) |
| H12 | 0.056053 | 0.004730 | 0.633416 | 0.020* |
| C11 | -0.2021 (5) | -0.1147 (3) | 0.65969 (15) | 0.0164 (6) |
| C12 | -0.0788 (5) | -0.1847 (3) | 0.71167 (15) | 0.0155 (6) |
| C13 | 0.1366 (5) | -0.1816 (3) | 0.72937 (15) | 0.0174 (6) |
| H13 | 0.237533 | -0.126760 | 0.709136 | 0.021* |
| C14 | 0.1911 (5) | -0.2697 (3) | 0.78141 (16) | 0.0194 (6) |
| H14 | 0.333099 | -0.281313 | 0.799380 | 0.023* |
| C15 | 0.0180 (5) | -0.3357 (3) | 0.80267 (15) | 0.0180 (6) |
| H15 | 0.024715 | -0.397159 | 0.837576 | 0.022* |
| N1 | 0.0994 (5) | 0.4417 (3) | 0.40233 (14) | 0.0232 (6) |
| O1 | -0.3960 (4) | -0.1393 (3) | 0.65025 (12) | 0.0220 (5) |
| S1 | -0.21315 (13) | -0.29494 (9) | 0.75923 (4) | 0.0187 (2) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0188 (14) | 0.0149 (14) | 0.0140 (14) | 0.0025 (12) | 0.0012 (11) | -0.0014 (11) |
| C2 | 0.0160 (14) | 0.0211 (16) | 0.0195 (15) | 0.0028 (12) | 0.0003 (12) | -0.0010 (13) |
| C3 | 0.0187 (15) | 0.0221 (16) | 0.0167 (14) | 0.0051 (13) | -0.0014 (12) | 0.0004 (13) |
| C4 | 0.0226 (16) | 0.0152 (14) | 0.0159 (14) | 0.0039 (12) | 0.0030 (12) | -0.0011 (12) |
| C5 | 0.0200 (15) | 0.0170 (15) | 0.0191 (15) | -0.0006 (12) | -0.0011 (12) | -0.0016 (12) |
| C6 | 0.0211 (15) | 0.0179 (15) | 0.0148 (14) | 0.0020 (13) | -0.0029 (11) | -0.0011 (12) |
| C7 | 0.0287 (19) | 0.0276 (19) | 0.032 (2) | -0.0026 (16) | 0.0024 (15) | 0.0082 (16) |
| C8 | 0.0300 (18) | 0.0202 (16) | 0.0220 (16) | 0.0055 (14) | -0.0009 (14) | 0.0054 (13) |
| C9 | 0.0190 (15) | 0.0161 (14) | 0.0156 (14) | 0.0017 (12) | 0.0027 (11) | -0.0033 (12) |
| C10 | 0.0172 (14) | 0.0158 (14) | 0.0159 (14) | -0.0027 (12) | 0.0011 (11) | -0.0025 (12) |
| C11 | 0.0183 (14) | 0.0159 (14) | 0.0150 (14) | 0.0004 (12) | 0.0023 (11) | -0.0018 (11) |
| C12 | 0.0168 (14) | 0.0146 (14) | 0.0154 (14) | -0.0017 (11) | 0.0030 (11) | -0.0020 (11) |
| C13 | 0.0165 (14) | 0.0174 (15) | 0.0182 (15) | -0.0029 (12) | 0.0004 (11) | 0.0020 (12) |
| C14 | 0.0176 (14) | 0.0202 (16) | 0.0203 (15) | -0.0023 (12) | 0.0011 (12) | 0.0047 (13) |
| C15 | 0.0184 (15) | 0.0199 (15) | 0.0157 (14) | -0.0009 (12) | 0.0001 (11) | 0.0010 (12) |
| N1 | 0.0267 (15) | 0.0216 (14) | 0.0211 (14) | 0.0009 (12) | -0.0012 (12) | 0.0062 (12) |
| O1 | 0.0145 (11) | 0.0256 (13) | 0.0258 (12) | -0.0025 (10) | -0.0014 (9) | 0.0034 (10) |
| S1 | 0.0137 (4) | 0.0218 (4) | 0.0210 (4) | -0.0023 (3) | 0.0030 (3) | 0.0038 (3) |

Geometric parameters (Å, °)

| | | | |
|----------|-----------|------------|-----------|
| C1—C6 | 1.401 (5) | C8—H8 | 0.9800 |
| C1—C2 | 1.403 (4) | C8—H9 | 0.9800 |
| C1—C9 | 1.442 (4) | C8—H10 | 0.9800 |
| C2—C3 | 1.379 (5) | C9—C10 | 1.348 (5) |
| C2—H1 | 0.9500 | C9—H11 | 0.9500 |
| C3—C4 | 1.412 (5) | C10—C11 | 1.461 (4) |
| C3—H2 | 0.9500 | C10—H12 | 0.9500 |
| C4—N1 | 1.364 (4) | C11—O1 | 1.240 (4) |
| C4—C5 | 1.416 (5) | C11—C12 | 1.469 (4) |
| C5—C6 | 1.375 (5) | C12—C13 | 1.375 (4) |
| C5—H3 | 0.9500 | C12—S1 | 1.728 (3) |
| C6—H4 | 0.9500 | C13—C14 | 1.422 (4) |
| C7—N1 | 1.445 (5) | C13—H13 | 0.9500 |
| C7—H5 | 0.9800 | C14—C15 | 1.359 (4) |
| C7—H6 | 0.9800 | C14—H14 | 0.9500 |
| C7—H7 | 0.9800 | C15—S1 | 1.709 (3) |
| C8—N1 | 1.452 (4) | C15—H15 | 0.9500 |
| C6—C1—C2 | 116.5 (3) | H8—C8—H10 | 109.5 |
| C6—C1—C9 | 124.1 (3) | H9—C8—H10 | 109.5 |
| C2—C1—C9 | 119.4 (3) | C10—C9—C1 | 128.9 (3) |
| C3—C2—C1 | 122.4 (3) | C10—C9—H11 | 115.6 |
| C3—C2—H1 | 118.8 | C1—C9—H11 | 115.6 |
| C1—C2—H1 | 118.8 | C9—C10—C11 | 120.5 (3) |

| | | | |
|----------------|------------|-----------------|------------|
| C2—C3—C4 | 120.7 (3) | C9—C10—H12 | 119.7 |
| C2—C3—H2 | 119.7 | C11—C10—H12 | 119.7 |
| C4—C3—H2 | 119.7 | O1—C11—C10 | 122.8 (3) |
| N1—C4—C3 | 121.0 (3) | O1—C11—C12 | 119.3 (3) |
| N1—C4—C5 | 121.9 (3) | C10—C11—C12 | 117.9 (3) |
| C3—C4—C5 | 117.1 (3) | C13—C12—C11 | 130.7 (3) |
| C6—C5—C4 | 121.0 (3) | C13—C12—S1 | 111.0 (2) |
| C6—C5—H3 | 119.5 | C11—C12—S1 | 118.3 (2) |
| C4—C5—H3 | 119.5 | C12—C13—C14 | 112.3 (3) |
| C5—C6—C1 | 122.2 (3) | C12—C13—H13 | 123.8 |
| C5—C6—H4 | 118.9 | C14—C13—H13 | 123.8 |
| C1—C6—H4 | 118.9 | C15—C14—C13 | 112.7 (3) |
| N1—C7—H5 | 109.5 | C15—C14—H14 | 123.6 |
| N1—C7—H6 | 109.5 | C13—C14—H14 | 123.6 |
| H5—C7—H6 | 109.5 | C14—C15—S1 | 112.1 (3) |
| N1—C7—H7 | 109.5 | C14—C15—H15 | 124.0 |
| H5—C7—H7 | 109.5 | S1—C15—H15 | 124.0 |
| H6—C7—H7 | 109.5 | C4—N1—C7 | 120.9 (3) |
| N1—C8—H8 | 109.5 | C4—N1—C8 | 120.1 (3) |
| N1—C8—H9 | 109.5 | C7—N1—C8 | 117.8 (3) |
| H8—C8—H9 | 109.5 | C15—S1—C12 | 91.87 (16) |
| N1—C8—H10 | 109.5 | | |
| | | | |
| C6—C1—C2—C3 | -0.9 (5) | O1—C11—C12—C13 | 175.7 (3) |
| C9—C1—C2—C3 | 179.7 (3) | C10—C11—C12—C13 | -5.9 (5) |
| C1—C2—C3—C4 | 0.0 (5) | O1—C11—C12—S1 | -2.0 (4) |
| C2—C3—C4—N1 | 179.7 (3) | C10—C11—C12—S1 | 176.4 (2) |
| C2—C3—C4—C5 | 0.5 (5) | C11—C12—C13—C14 | -177.5 (3) |
| N1—C4—C5—C6 | -179.3 (3) | S1—C12—C13—C14 | 0.3 (4) |
| C3—C4—C5—C6 | 0.0 (5) | C12—C13—C14—C15 | -1.0 (4) |
| C4—C5—C6—C1 | -0.9 (5) | C13—C14—C15—S1 | 1.2 (4) |
| C2—C1—C6—C5 | 1.3 (5) | C3—C4—N1—C7 | 179.1 (3) |
| C9—C1—C6—C5 | -179.3 (3) | C5—C4—N1—C7 | -1.7 (5) |
| C6—C1—C9—C10 | -2.8 (5) | C3—C4—N1—C8 | 11.2 (5) |
| C2—C1—C9—C10 | 176.5 (3) | C5—C4—N1—C8 | -169.6 (3) |
| C1—C9—C10—C11 | 179.2 (3) | C14—C15—S1—C12 | -0.9 (3) |
| C9—C10—C11—O1 | -4.9 (5) | C13—C12—S1—C15 | 0.3 (3) |
| C9—C10—C11—C12 | 176.7 (3) | C11—C12—S1—C15 | 178.4 (3) |

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

Cg is the centroid of the S1/C12–C15 thiophene 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> |
|----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C13—H13 \cdots O1 ⁱ | 0.95 | 2.65 | 3.451 (4) | 142 |
| C14—H14 \cdots S1 ⁱ | 0.95 | 3.00 | 3.779 (3) | 141 |

| | | | | |
|-----------------------------------|------|------|-----------|-----|
| C15—H15 \cdots O1 ⁱⁱ | 0.95 | 2.57 | 3.291 (4) | 133 |
| C8—H8 \cdots Cg ⁱⁱⁱ | 0.98 | 2.64 | 3.457 (4) | 141 |

Symmetry codes: (i) $x+1, y, z$; (ii) $-x-1/2, y-1/2, -z+3/2$; (iii) $-x, -y, -z+1$.