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(2*E*)-2-(Thiophen-2-ylmethylidene)-1,2,3,4-tetrahydronaphthalen-1-one

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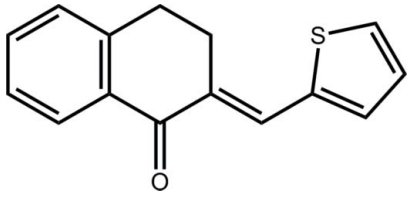
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 16.4.

In the title compound, $C_{15}H_{12}OS$, the cyclohexene ring has a twisted boat conformation with the C atom between the ketone and methylene atom and this methylene C atom lying 0.280 (3) and 0.760 (3) Å, respectively, from the plane through the remaining four atoms (r.m.s. deviation = 0.004 Å). The dihedral angle between the benzene and thiophene rings [21.64 (9)°] indicates an overall twist in the molecule. The thiophene S and ketone O atoms are *anti*, an orientation that allows the close approach of these atoms [3.3116 (17) Å] in the crystal structure and which leads to the formation of helical supramolecular chains along the *c* axis.

Related literature

For the activity of related species developed for the treatment of Chagas disease, see: Vera-DiVaio *et al.* (2009). For a related structure, see: Asiri *et al.* (2012).



Experimental

Crystal data

$C_{15}H_{12}OS$
M_r = 240.31
Orthorhombic, $Pna2_1$
a = 24.7989 (10) Å
b = 3.9976 (2) Å
c = 11.3163 (5) Å
V = 1121.85 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 100 K
0.35 × 0.30 × 0.25 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.812, T_{max} = 1.000
7054 measured reflections
2528 independent reflections
2383 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.03
2528 reflections
154 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1171 Friedel pairs
Flack parameter: 0.07 (10)

Data collection: *CrysAlis PRO* (Agilent, 2012); 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: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2087).

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supplementary materials

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(2*E*)-2-(Thiophen-2-ylmethylidene)-1,2,3,4-tetrahydronaphthalen-1-one

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Comment

In continuation of structural studies on tetrahydronaphthalen-1-one derivatives (Asiri *et al.*, 2012), the crystal and molecular structure of the title compound, 2-thiophen-2-ylmethylene-3,4-dihydro-2*H*-naphthalen-1-one (I), was investigated. Interest in this class of compound stems from their putative activity against Chagas disease (Vera-DiVaio *et al.*, 2009).

In (I), Fig. 1, the cyclohexene ring has a twisted boat conformation with the C6 and C15 atoms lying, respectively, 0.280 (3) and 0.760 (3) Å from the plane through the remaining four atoms which have a r.m.s. deviation = 0.004 Å. Overall, the molecule is twisted with the dihedral angle between the benzene and thiophen-2-yl rings being 21.64 (9)°. The conformation about the exocyclic methylidene C5═C6 [1.349 (3) Å] is *E*. The thiophen-2-yl-S and ketone-O atoms are *anti*.

In the crystal packing, weak *π*—*π* interactions are noted between translationally related benzene rings, *i.e*. inter-centroid distance = 3.9976 (11) Å (symmetry operation *x*, 1 + *y*, *z)* which lead to stacks along the *b* axis. Other than these, the most prominent interactions appear to be of the type S···O, *i.e*. S1···O1^{i} = 3.3116 (17) Å for *i*: 1 - *x*, 1 - *y*, 1/2 + *z*. The result is the formation of helical supramolecular chains along the *c* axis, Fig. 2.

Experimental

A solution of the 2-thiophen-2-carboxaldehyde (1.1 g, 0.01 *M*) in ethanol (20 ml) was added to a stirred solution of 1-tetralone (1.46 g,0.0 1*M*) in ethanolic KOH (20 ml, 20%). Stirring was maintained at room temperature for 6 h. The reaction mixture was then poured onto water (200 ml) and set aside overnight. The precipitated solid product was collected by filtration, washed with water, dried and recrystallized from its ethanol solution. *M*.pt: 351–352 K. Yield: 92%.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95–0.99 Å, U_{iso}(H) = 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation. Owing to poor agreement, one reflection, *i.e*. (6 3 - 3), was omitted from the final refinement.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO* (Agilent, 2012); data reduction: *CrysAlis PRO* (Agilent, 2012); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

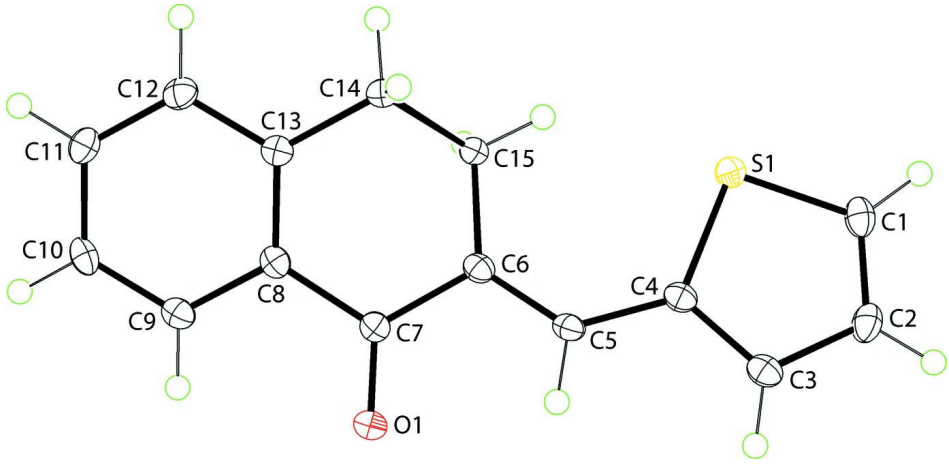

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

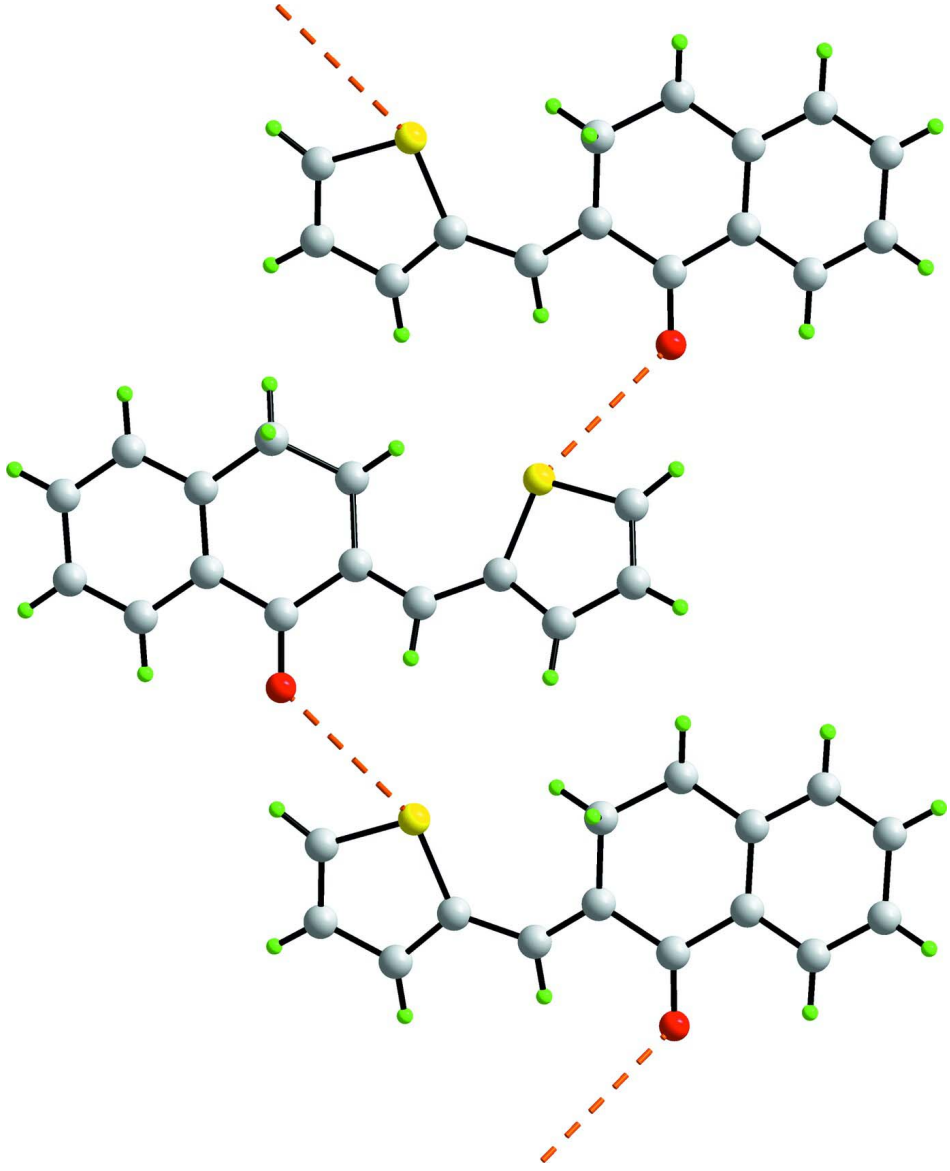

Figure 2

A view of the helical supramolecular chain along the *c* axis in (I) mediated by S···O interactions shown as orange dashed lines.

(2*E*)-2-(Thiophen-2-ylmethylidene)-1,2,3,4-tetrahydronaphthalen-1-one

Crystal data

$C_{15}H_{12}OS$
$M_r = 240.31$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 24.7989$ (10) Å
$b = 3.9976$ (2) Å
$c = 11.3163$ (5) Å
$V = 1121.85$ (9) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.423$ Mg m^{-3}
Melting point: 351 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3882 reflections
$\theta = 2.4$–27.5°
$\mu = 0.27$ mm^{-1}
$T = 100$ K
Prism, light-brown
0.35 × 0.30 × 0.25 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.812, T_{max} = 1.000
7054 measured reflections
2528 independent reflections
2383 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.6°, θ_{min} = 2.4°
h = −32→23
k = −5→4
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.03
2528 reflections
154 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack (1983), 1171 Friedel pairs
Flack parameter: 0.07 (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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.538955 (17)	0.26862 (11)	0.50161 (7)	0.01740 (13)
O1	0.36843 (6)	0.5071 (4)	0.19698 (15)	0.0251 (4)
C1	0.59800 (8)	0.0731 (5)	0.4638 (2)	0.0201 (4)
H1	0.6276	0.0445	0.5161	0.024*
C2	0.59738 (8)	−0.0357 (5)	0.3506 (2)	0.0201 (4)
H2	0.6268	−0.1480	0.3143	0.024*
C3	0.54817 (8)	0.0355 (5)	0.2914 (2)	0.0173 (4)
H3	0.5412	−0.0249	0.2117	0.021*
C4	0.51135 (8)	0.2025 (5)	0.36246 (19)	0.0153 (4)
C5	0.45890 (8)	0.3086 (5)	0.32082 (19)	0.0152 (4)
H5	0.4519	0.2571	0.2403	0.018*
C6	0.41829 (8)	0.4681 (4)	0.37662 (18)	0.0149 (4)
C7	0.36964 (8)	0.5503 (5)	0.30417 (19)	0.0157 (4)
C8	0.32189 (8)	0.6897 (4)	0.36676 (19)	0.0149 (4)
C9	0.27955 (8)	0.8271 (5)	0.3005 (2)	0.0170 (4)

H9	0.2824	0.8382	0.2168	0.020*
C10	0.23379 (8)	0.9464 (5)	0.35577 (19)	0.0177 (4)
H10	0.2053	1.0389	0.3103	0.021*
C11	0.22952 (8)	0.9309 (5)	0.47876 (19)	0.0179 (4)
H11	0.1979	1.0100	0.5171	0.022*
C12	0.27162 (9)	0.7996 (5)	0.5445 (2)	0.0176 (4)
H12	0.2688	0.7920	0.6282	0.021*
C13	0.31817 (7)	0.6783 (4)	0.4900 (2)	0.0140 (4)
C14	0.36339 (8)	0.5298 (5)	0.56230 (18)	0.0149 (4)
H14A	0.3570	0.2872	0.5728	0.018*
H14B	0.3637	0.6348	0.6415	0.018*
C15	0.41813 (7)	0.5824 (4)	0.5033 (2)	0.0150 (4)
H15A	0.4276	0.8228	0.5064	0.018*
H15B	0.4460	0.4573	0.5477	0.018*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0148 (2)	0.0213 (2)	0.0160 (2)	0.00044 (16)	−0.0005 (2)	−0.0013 (2)
O1	0.0217 (9)	0.0398 (9)	0.0137 (8)	0.0081 (6)	−0.0021 (6)	−0.0022 (7)
C1	0.0132 (9)	0.0204 (9)	0.0267 (12)	−0.0002 (7)	−0.0007 (8)	0.0033 (8)
C2	0.0145 (10)	0.0212 (10)	0.0247 (12)	0.0020 (7)	0.0048 (9)	0.0030 (9)
C3	0.0195 (10)	0.0168 (9)	0.0156 (10)	−0.0027 (7)	−0.0009 (8)	0.0041 (8)
C4	0.0174 (10)	0.0145 (8)	0.0139 (10)	−0.0021 (7)	−0.0012 (8)	0.0017 (8)
C5	0.0164 (10)	0.0178 (9)	0.0113 (10)	−0.0016 (7)	−0.0012 (7)	0.0011 (8)
C6	0.0162 (9)	0.0147 (9)	0.0137 (10)	−0.0011 (7)	−0.0007 (8)	0.0028 (8)
C7	0.0156 (10)	0.0168 (9)	0.0147 (10)	0.0009 (7)	−0.0004 (8)	0.0008 (8)
C8	0.0143 (9)	0.0138 (8)	0.0167 (10)	−0.0021 (7)	−0.0014 (8)	0.0012 (8)
C9	0.0182 (10)	0.0175 (9)	0.0153 (10)	−0.0021 (8)	−0.0019 (8)	0.0016 (8)
C10	0.0130 (9)	0.0177 (9)	0.0226 (12)	−0.0019 (7)	−0.0046 (8)	0.0028 (8)
C11	0.0150 (9)	0.0172 (9)	0.0216 (12)	−0.0008 (7)	0.0035 (8)	0.0002 (7)
C12	0.0173 (10)	0.0186 (10)	0.0169 (10)	−0.0033 (7)	0.0032 (8)	0.0012 (8)
C13	0.0155 (9)	0.0119 (8)	0.0147 (10)	−0.0029 (6)	−0.0001 (8)	0.0007 (8)
C14	0.0166 (9)	0.0152 (9)	0.0129 (10)	−0.0012 (7)	0.0010 (8)	0.0001 (8)
C15	0.0138 (8)	0.0187 (8)	0.0125 (9)	0.0005 (7)	−0.0006 (9)	−0.0001 (9)

Geometric parameters (Å, º)

S1—C1	1.714 (2)	C8—C9	1.403 (3)
S1—C4	1.737 (2)	C9—C10	1.381 (3)
O1—C7	1.226 (3)	C9—H9	0.9500
C1—C2	1.353 (3)	C10—C11	1.397 (3)
C1—H1	0.9500	C10—H10	0.9500
C2—C3	1.421 (3)	C11—C12	1.385 (3)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.388 (3)	C12—C13	1.396 (3)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.447 (3)	C13—C14	1.510 (3)
C5—C6	1.349 (3)	C14—C15	1.527 (3)
C5—H5	0.9500	C14—H14A	0.9900

C6—C7	1.495 (3)	C14—H14B	0.9900
C6—C15	1.504 (3)	C15—H15A	0.9900
C7—C8	1.488 (3)	C15—H15B	0.9900
C8—C13	1.399 (3)		

C1—S1—C4	92.35 (10)	C10—C9—H9	119.7
C2—C1—S1	111.91 (16)	C8—C9—H9	119.7
C2—C1—H1	124.0	C9—C10—C11	119.89 (19)
S1—C1—H1	124.0	C9—C10—H10	120.1
C1—C2—C3	113.1 (2)	C11—C10—H10	120.1
C1—C2—H2	123.5	C12—C11—C10	119.65 (19)
C3—C2—H2	123.5	C12—C11—H11	120.2
C4—C3—C2	112.9 (2)	C10—C11—H11	120.2
C4—C3—H3	123.6	C11—C12—C13	121.2 (2)
C2—C3—H3	123.6	C11—C12—H12	119.4
C3—C4—C5	122.9 (2)	C13—C12—H12	119.4
C3—C4—S1	109.82 (15)	C12—C13—C8	118.9 (2)
C5—C4—S1	127.21 (16)	C12—C13—C14	120.8 (2)
C6—C5—C4	131.1 (2)	C8—C13—C14	120.26 (17)
C6—C5—H5	114.5	C13—C14—C15	111.67 (17)
C4—C5—H5	114.5	C13—C14—H14A	109.3
C5—C6—C7	116.72 (18)	C15—C14—H14A	109.3
C5—C6—C15	126.27 (18)	C13—C14—H14B	109.3
C7—C6—C15	116.97 (16)	C15—C14—H14B	109.3
O1—C7—C8	120.28 (18)	H14A—C14—H14B	107.9
O1—C7—C6	122.11 (18)	C6—C15—C14	112.16 (16)
C8—C7—C6	117.60 (18)	C6—C15—H15A	109.2
C13—C8—C9	119.78 (19)	C14—C15—H15A	109.2
C13—C8—C7	121.00 (18)	C6—C15—H15B	109.2
C9—C8—C7	119.20 (19)	C14—C15—H15B	109.2
C10—C9—C8	120.5 (2)	H15A—C15—H15B	107.9

C4—S1—C1—C2	0.54 (16)	C6—C7—C8—C9	168.28 (17)
S1—C1—C2—C3	−0.5 (2)	C13—C8—C9—C10	−1.1 (3)
C1—C2—C3—C4	0.2 (3)	C7—C8—C9—C10	177.44 (17)
C2—C3—C4—C5	178.49 (17)	C8—C9—C10—C11	0.1 (3)
C2—C3—C4—S1	0.2 (2)	C9—C10—C11—C12	0.8 (3)
C1—S1—C4—C3	−0.40 (15)	C10—C11—C12—C13	−0.8 (3)
C1—S1—C4—C5	−178.62 (18)	C11—C12—C13—C8	−0.2 (3)
C3—C4—C5—C6	179.2 (2)	C11—C12—C13—C14	−178.85 (17)
S1—C4—C5—C6	−2.8 (3)	C9—C8—C13—C12	1.1 (3)
C4—C5—C6—C7	177.95 (19)	C7—C8—C13—C12	−177.36 (16)
C4—C5—C6—C15	0.4 (3)	C9—C8—C13—C14	179.76 (16)
C5—C6—C7—O1	−7.2 (3)	C7—C8—C13—C14	1.3 (3)
C15—C6—C7—O1	170.53 (18)	C12—C13—C14—C15	−150.02 (17)
C5—C6—C7—C8	172.98 (17)	C8—C13—C14—C15	31.4 (2)
C15—C6—C7—C8	−9.3 (2)	C5—C6—C15—C14	−140.98 (19)
O1—C7—C8—C13	166.95 (18)	C7—C6—C15—C14	41.5 (2)
C6—C7—C8—C13	−13.2 (3)	C13—C14—C15—C6	−51.7 (2)

O1—C7—C8—C9	−11.5 (3)