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Crystal structure of (*E*)-*N*-phenyl-*N'*-[1-(thiophen-2-yl)ethylidene]formohydrazide

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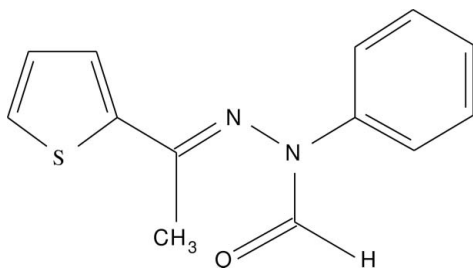
In the title compound, C₁₃H₁₂N₂OS, the planes of the thiophene and phenyl rings are nearly perpendicular to each other, making a dihedral angle of 86.42 (12)°. In the crystal, molecules are linked by C—H...O hydrogen bonds, forming a helical chain along the *b*-axis direction.

Keywords: crystal structure; thiophene derivative; hydrogen bonding.

CCDC reference: 1014287

1. Related literature

For the biological activity of thiophene derivatives, see: Bondock *et al.* (2010); Bellina *et al.* (2007); Konstantinova *et al.* (2009); Al-Said *et al.* (2011). For the crystal structure of a similar compound, *viz.* (*E*)-*N'*-[1-(thiophen-2-yl)ethylidene]-benzohydrazide, see: Shan *et al.* (2011). For a description of the Cambridge Structural Database, see: Allen (2002).



2. Experimental

2.1. Crystal data

C₁₃H₁₂N₂OS

M_r = 244.32

Orthorhombic, *P*2₁2₁2₁
a = 5.4960 (7) Å
b = 11.0177 (13) Å
c = 20.249 (2) Å
V = 1226.1 (2) Å³

Z = 4
 Cu *K*α radiation
 μ = 2.22 mm⁻¹
T = 296 K
 0.25 × 0.22 × 0.20 mm

2.2. Data collection

Bruker X8 Proteum diffractometer
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2013)
T_{min} = 0.604, *T_{max}* = 0.662

6298 measured reflections
 2010 independent reflections
 1904 reflections with *I* > 2σ(*I*)
R_{int} = 0.042

2.3. Refinement

R[*F*² > 2σ(*F*²)] = 0.038
wR(*F*²) = 0.102
S = 1.10
 2010 reflections
 165 parameters
 H-atom parameters constrained

Δρ_{max} = 0.21 e Å⁻³
 Δρ_{min} = -0.20 e Å⁻³
 Absolute structure: Flack (1983),
 805 Friedel pairs
 Absolute structure parameter:
 0.02 (2)

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

<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>
C1—H1...O1 ⁱ	0.93	2.39	3.202 (3)	145

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINTE* (Bruker, 2013); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: IS5369).

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supporting information

Acta Cryst. (2014). E70, o928–o929 [doi:10.1107/S1600536814016511]

Crystal structure of (*E*)-*N*-phenyl-*N'*-[1-(thiophen-2-yl)ethylidene]formohydrazide

C. S. Dileep, K. R. Raghavendra, N. K. Lokanath, K. Ajay Kumar and M. A. Sridhar

S1. Comment

In medicinal chemistry, thiophene derivatives have been very well known for their therapeutic applications. Many thiophene derivatives have been developed as chemotherapeutic agents and are extensively used. Thiophene nucleus is one of the most important heterocycles exhibiting remarkable pharmacological activities. The great interest in the synthesis of thiophene derivatives due to their diverse biological and chemical properties. Thiophene, as a prominent structural motif, is found in numerous active compounds, which contain 5-membered heterocyclic structure have attracted a lot of interests in many fields, and its rich biological activity in medicinal chemistry owing to their biological properties. Thiophene and thiazole derivatives are known to possess interesting biological properties like anticancer (Bondock *et al.*, 2010; Bellina *et al.*, 2007; Konstantinova *et al.*, 2009). Thiophene or benzothiophene moieties due to the well documented anti-cancer activity of these moieties to study their SAR and their anti-breast cancer activity (Al-Said *et al.*, 2011). In view of their importance as discussed above, thiophene derivatives were taken for their conformational studies to get better structural activity correlation.

In the title compound (Fig. 1), the bond lengths do not show much variation in the core structure of the derivatives, and are similar to the standard values (Allen *et al.*, 2002). The thiophene (S1/C1–C4) and phenyl (C8–C13) rings are nearly perpendicular with a dihedral angle of 86.42 (12)° between their mean planes. The bond lengths and bond angles do not show large deviations and are comparable with those reported for a similar structure (Shan *et al.*, 2011). The conformation of the attachment of the thiophene and phenyl rings can also be characterized by torsion angles of (C4—C5—N1—N2), (C5—N1—N2—C8), (O1—C7—N2—C8) and (S1—C4—C5—C6) being 178.38, 127.73, 171.34 and -170.31°, respectively. The crystal structure has an intermolecular C—H···O hydrogen bond. The molecular packing viewed down the *a* axis is shown in Fig. 2.

S2. Experimental

A mixture of (*E*)-1-phenyl-2-[(1-thiophen-2-yl)ethylidene]hydrazine (0.176 mmol) were added to the Vilsmeier-Haack reagent prepared by drop-wise addition of POCl₃ (1.2 ml) in ice cooled DMF (5 ml). The mixture was stirred at 60–65 °C for 6 h. The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was poured into ice cold water, neutralized with NaHCO₃, the solid separated was filtered, washed with water and recrystallized from ethanol to get the compound in 93% yield.

S3. Refinement

All H atoms were located from difference maps and were positioned geometrically and refined using a riding model with C—H = 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

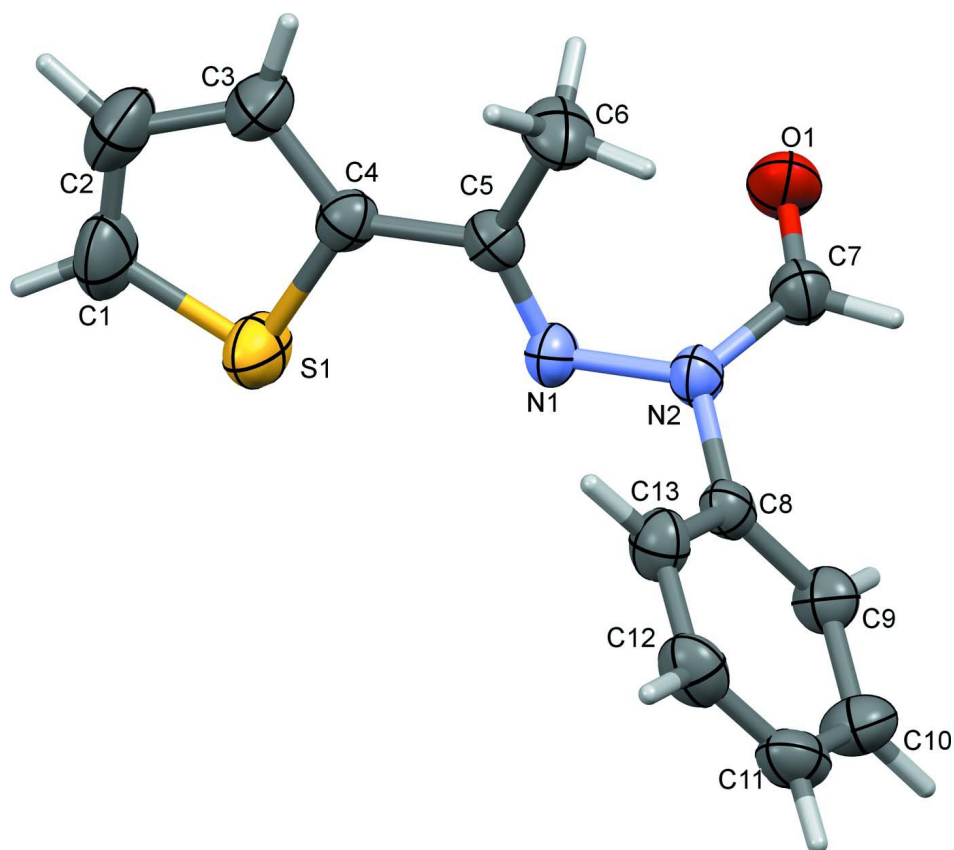
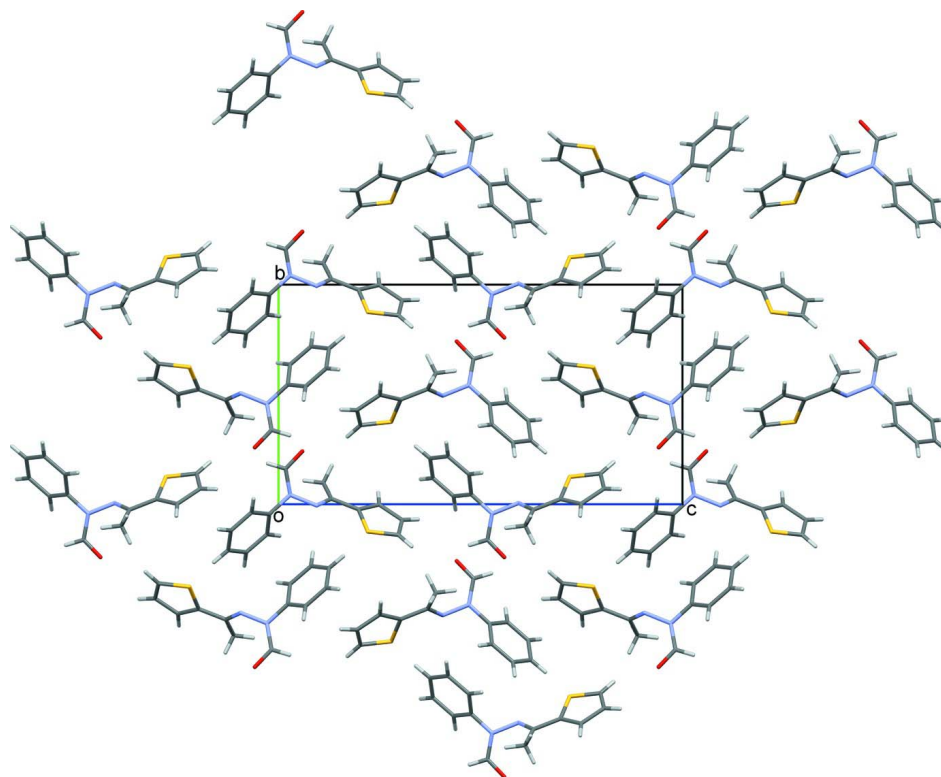


Figure 1

ORTEP view of the molecule with the atom-labeling scheme. The displacement ellipsoids are drawn at the 50% probability level.

**Figure 2**

A molecular packing view of the title compound down the *a*-axis.

(*E*)-*N*-Phenyl-*N'*-[1-(thiophen-2-yl)ethylidene]formohydrazide.

Crystal data

$C_{13}H_{12}N_2OS$

$M_r = 244.32$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 5.4960$ (7) Å

$b = 11.0177$ (13) Å

$c = 20.249$ (2) Å

$V = 1226.1$ (2) Å³

$Z = 4$

$F(000) = 512$

$D_x = 1.324$ Mg m⁻³

Cu *K*α radiation, $\lambda = 1.54178$ Å

Cell parameters from 6298 reflections

$\theta = 4.4$ – 64.6°

$\mu = 2.22$ mm⁻¹

$T = 296$ K

Block, pale yellow

$0.25 \times 0.22 \times 0.20$ mm

Data collection

Bruker X8 Proteum
diffractometer

Radiation source: Bruker MicroStar microfocus
rotating anode

Helios multilayer optics monochromator

Detector resolution: 10.7 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2013)

$T_{\min} = 0.604$, $T_{\max} = 0.662$

6298 measured reflections

2010 independent reflections

1904 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 64.6^\circ$, $\theta_{\min} = 4.4^\circ$

$h = -2 \rightarrow 6$

$k = -12 \rightarrow 12$

$l = -23 \rightarrow 22$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.102$ $S = 1.10$

2010 reflections

165 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.0861P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL*, $\text{FC}^* = \text{KFC}[1 + 0.001\text{XFC}^2\Lambda^3/\text{SIN}(2\Theta)]^{-1/4}$

Extinction coefficient: 0.0158 (16)

Absolute structure: Flack (1983), 805 Friedel
pairs

Absolute structure parameter: 0.02 (2)

*Special details***Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles**Refinement.** Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R -factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating $-R$ -factor-obs *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)*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.05502 (12)	0.12455 (6)	0.72104 (3)	0.0539 (2)
O1	0.0434 (4)	-0.24121 (15)	0.55887 (8)	0.0526 (6)
N1	0.0078 (3)	0.00557 (16)	0.59394 (8)	0.0348 (5)
N2	0.0086 (3)	-0.04249 (15)	0.52882 (8)	0.0344 (5)
C1	-0.0682 (6)	0.1399 (3)	0.79702 (12)	0.0623 (10)
C2	-0.2635 (6)	0.0712 (3)	0.80591 (13)	0.0648 (10)
C3	-0.3227 (5)	0.0016 (2)	0.74907 (11)	0.0513 (8)
C4	-0.1639 (4)	0.02325 (18)	0.69777 (10)	0.0358 (6)
C5	-0.1724 (4)	-0.02503 (18)	0.63061 (10)	0.0331 (6)
C6	-0.3905 (4)	-0.0972 (2)	0.61062 (13)	0.0509 (8)
C7	0.0373 (4)	-0.16179 (19)	0.51733 (10)	0.0410 (7)
C8	0.0373 (4)	0.04484 (17)	0.47710 (9)	0.0325 (6)
C9	0.2273 (4)	0.0366 (2)	0.43330 (11)	0.0397 (6)
C10	0.2429 (4)	0.1192 (2)	0.38203 (12)	0.0479 (7)
C11	0.0710 (5)	0.2091 (2)	0.37516 (11)	0.0469 (7)
C12	-0.1151 (5)	0.2185 (2)	0.41975 (12)	0.0463 (8)
C13	-0.1322 (4)	0.1365 (2)	0.47156 (11)	0.0413 (6)
H1	-0.00560	0.19130	0.82930	0.0750*
H2	-0.35200	0.06880	0.84500	0.0780*
H3	-0.45300	-0.05220	0.74680	0.0620*
H6A	-0.39060	-0.10770	0.56360	0.0760*
H6B	-0.53550	-0.05510	0.62380	0.0760*

H6C	-0.38540	-0.17530	0.63170	0.0760*
H7	0.05420	-0.18570	0.47350	0.0490*
H9	0.34410	-0.02390	0.43800	0.0480*
H10	0.37060	0.11400	0.35200	0.0570*
H11	0.08130	0.26340	0.34010	0.0560*
H12	-0.23010	0.27990	0.41530	0.0560*
H13	-0.25720	0.14340	0.50230	0.0500*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0546 (4)	0.0703 (5)	0.0368 (3)	-0.0147 (3)	-0.0029 (3)	-0.0071 (3)
O1	0.0729 (11)	0.0375 (9)	0.0474 (9)	0.0049 (8)	0.0049 (8)	0.0096 (7)
N1	0.0402 (10)	0.0379 (9)	0.0263 (8)	-0.0024 (8)	-0.0012 (7)	-0.0037 (6)
N2	0.0452 (10)	0.0313 (8)	0.0266 (8)	0.0006 (8)	-0.0005 (7)	-0.0022 (7)
C1	0.085 (2)	0.0689 (17)	0.0330 (12)	0.0037 (16)	-0.0059 (13)	-0.0095 (11)
C2	0.0757 (18)	0.0819 (19)	0.0368 (13)	0.0055 (16)	0.0152 (13)	0.0000 (13)
C3	0.0524 (14)	0.0590 (15)	0.0426 (13)	-0.0041 (12)	0.0127 (11)	-0.0022 (11)
C4	0.0373 (10)	0.0370 (11)	0.0332 (10)	0.0038 (9)	0.0009 (8)	0.0026 (8)
C5	0.0317 (10)	0.0314 (10)	0.0362 (11)	0.0037 (8)	-0.0030 (8)	0.0008 (8)
C6	0.0372 (12)	0.0564 (15)	0.0592 (14)	-0.0111 (10)	0.0008 (11)	-0.0119 (12)
C7	0.0511 (12)	0.0353 (11)	0.0366 (11)	0.0020 (9)	0.0024 (10)	-0.0018 (9)
C8	0.0388 (10)	0.0296 (10)	0.0291 (9)	-0.0017 (8)	-0.0051 (8)	-0.0012 (8)
C9	0.0355 (10)	0.0392 (11)	0.0444 (12)	0.0049 (9)	0.0011 (9)	0.0016 (10)
C10	0.0456 (12)	0.0511 (13)	0.0469 (13)	-0.0056 (11)	0.0089 (10)	0.0094 (11)
C11	0.0604 (14)	0.0365 (11)	0.0439 (12)	-0.0080 (11)	-0.0054 (11)	0.0088 (9)
C12	0.0536 (14)	0.0350 (12)	0.0503 (13)	0.0084 (11)	-0.0087 (11)	0.0024 (10)
C13	0.0429 (11)	0.0416 (11)	0.0395 (11)	0.0083 (10)	0.0012 (10)	-0.0035 (10)

Geometric parameters (Å, °)

S1—C1	1.690 (3)	C10—C11	1.376 (3)
S1—C4	1.707 (2)	C11—C12	1.368 (4)
O1—C7	1.214 (3)	C12—C13	1.388 (3)
N1—N2	1.421 (2)	C1—H1	0.9300
N1—C5	1.283 (3)	C2—H2	0.9300
N2—C7	1.344 (3)	C3—H3	0.9300
N2—C8	1.431 (2)	C6—H6A	0.9600
C1—C2	1.326 (5)	C6—H6B	0.9600
C2—C3	1.421 (4)	C6—H6C	0.9600
C3—C4	1.378 (3)	C7—H7	0.9300
C4—C5	1.461 (3)	C9—H9	0.9300
C5—C6	1.494 (3)	C10—H10	0.9300
C8—C9	1.373 (3)	C11—H11	0.9300
C8—C13	1.379 (3)	C12—H12	0.9300
C9—C10	1.383 (3)	C13—H13	0.9300
C1—S1—C4	91.96 (13)	C2—C1—H1	123.00

N2—N1—C5	116.20 (17)	C1—C2—H2	124.00
N1—N2—C7	121.70 (16)	C3—C2—H2	124.00
N1—N2—C8	115.40 (15)	C2—C3—H3	124.00
C7—N2—C8	121.19 (16)	C4—C3—H3	124.00
S1—C1—C2	113.0 (2)	C5—C6—H6A	109.00
C1—C2—C3	112.6 (2)	C5—C6—H6B	109.00
C2—C3—C4	111.9 (2)	C5—C6—H6C	109.00
S1—C4—C3	110.58 (16)	H6A—C6—H6B	109.00
S1—C4—C5	121.16 (16)	H6A—C6—H6C	109.00
C3—C4—C5	128.2 (2)	H6B—C6—H6C	109.00
N1—C5—C4	114.73 (19)	O1—C7—H7	117.00
N1—C5—C6	127.04 (19)	N2—C7—H7	117.00
C4—C5—C6	118.13 (19)	C8—C9—H9	120.00
O1—C7—N2	126.02 (19)	C10—C9—H9	120.00
N2—C8—C9	120.81 (18)	C9—C10—H10	120.00
N2—C8—C13	118.53 (18)	C11—C10—H10	120.00
C9—C8—C13	120.65 (19)	C10—C11—H11	120.00
C8—C9—C10	119.2 (2)	C12—C11—H11	120.00
C9—C10—C11	120.5 (2)	C11—C12—H12	120.00
C10—C11—C12	120.1 (2)	C13—C12—H12	120.00
C11—C12—C13	120.0 (2)	C8—C13—H13	120.00
C8—C13—C12	119.5 (2)	C12—C13—H13	120.00
S1—C1—H1	124.00		
C4—S1—C1—C2	0.9 (3)	C2—C3—C4—C5	-176.1 (2)
C1—S1—C4—C3	-1.3 (2)	C2—C3—C4—S1	1.4 (3)
C1—S1—C4—C5	176.4 (2)	S1—C4—C5—N1	6.3 (3)
C5—N1—N2—C7	-67.0 (2)	C3—C4—C5—N1	-176.4 (2)
C5—N1—N2—C8	127.7 (2)	C3—C4—C5—C6	7.0 (3)
N2—N1—C5—C4	178.38 (16)	S1—C4—C5—C6	-170.31 (16)
N2—N1—C5—C6	-5.3 (3)	N2—C8—C9—C10	176.94 (19)
C7—N2—C8—C13	135.3 (2)	C13—C8—C9—C10	-2.1 (3)
N1—N2—C7—O1	6.9 (3)	N2—C8—C13—C12	-176.7 (2)
C8—N2—C7—O1	171.3 (2)	C9—C8—C13—C12	2.3 (3)
N1—N2—C8—C9	121.6 (2)	C8—C9—C10—C11	0.4 (3)
C7—N2—C8—C9	-43.7 (3)	C9—C10—C11—C12	1.0 (4)
N1—N2—C8—C13	-59.4 (2)	C10—C11—C12—C13	-0.7 (4)
S1—C1—C2—C3	-0.3 (4)	C11—C12—C13—C8	-0.9 (3)
C1—C2—C3—C4	-0.7 (4)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1 \cdots O1 ⁱ	0.93	2.39	3.202 (3)	145

Symmetry code: (i) $-x, y+1/2, -z+3/2$.