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Ethyl 2-acetamido-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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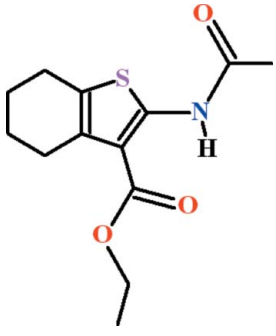
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.105; data-to-parameter ratio = 14.5.

In the title compound, $C_{13}H_{17}NO_3S$, the dihedral angles between the thiophene ring and the ethyl ester and acetamide groups are 5.21 (13) and 10.06 (16)°, respectively. The cyclohexene ring adopts a half-chair conformation. An S(6) ring is formed due to an intramolecular N—H···O hydrogen bond. In the crystal, molecules are linked by C—H···O interactions between the tetrahydro-1-benzothiophene unit and the ethyl ester group, forming C(7) chains propagating along the b-axis direction.

Related literature

For related structures, see: Mukhtar *et al.* (2010*a*,*b*).



Experimental

Crystal data

$C_{13}H_{17}NO_3S$ M_r = 267.34
Monoclinic, $P2_1/c$
a = 10.4267 (4) Å
b = 16.6554 (7) Å
c = 8.0961 (3) Å
β = 109.610 (1)°
V = 1324.43 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 296 K
0.28 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.953, T_{max} = 0.958
9994 measured reflections
2389 independent reflections
1831 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.05
2389 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86	2.03	2.674 (2)	131
C7—H7B···O3^{i}	0.97	2.50	3.392 (3)	153

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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 *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the provision of funds for the purchase of a diffractometer and encouragement by Dr Muhammad Akram Chaudhary, Vice Chancellor, University of Sargodha, Pakistan.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6836).

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supporting information

Acta Cryst. (2012). E**68**, o2042 [https://doi.org/10.1107/S160053681202524X]

Ethyl 2-acetamido-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

Asma Mukhtar, M. Nawaz Tahir, Misbahul Ain Khan, Abdul Qayyum Ather and Muhammad Naeem Khan

S1. Comment

We reported the crystal structures of ethyl 2-benzamido-4,5,6,7-tetrahydro-1- benzothiophene-3-carboxylate (Mukhtar *et al.*, 2010*a*) and diethyl 5-acetamido-3-methylthiophene-2,4-dicarboxylate (Mukhtar *et al.*, 2010*b*) which are related to the tile compound (I), (Fig. 1).

In (I), the thiophene ring A (S1/C8/C3/C2/C9), ethyl ester group B (O1/C1/O3/C10/C11) and acetamide moiety C (N1/C12/O2/C13) are planar with r. m. s. deviation of 0.0034, 0.0560 and 0.0029 Å, respectively. The dihedral angle between A/B, A/C and B/C is 5.21 (13), 5.17 (14) and 10.06 (16)°, respectively. In the title compound an S(6) ring motif is formed due to intramolecular H-bonding of N—H···O type (Table 1, Fig. 1). The molecules are linked in the form of C(7) chains extending along the [010] direction due to C—H···O type of H-bonding.

S2. Experimental

Ethyl 2-amino-4,5,6,7-tetrahydrobenzothiophene-3-carboxylate (0.3 g, 1 mmol) was dissolved in chloroform and in this solution 1 ml of acetyl chloride was added. The reaction mixture was refluxed for 8 h. The solvent was removed and the residue was recrystallized by ethanol to get colorless prisms of (I). m.p. 383 K, yield: 0.24 g, 85%.

S3. Refinement

The H-atoms were positioned geometrically (N—H = 0.86, C–H = 0.96–0.97 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C, N), where x = 1.5 for methyl and x = 1.2 for other H-atoms.

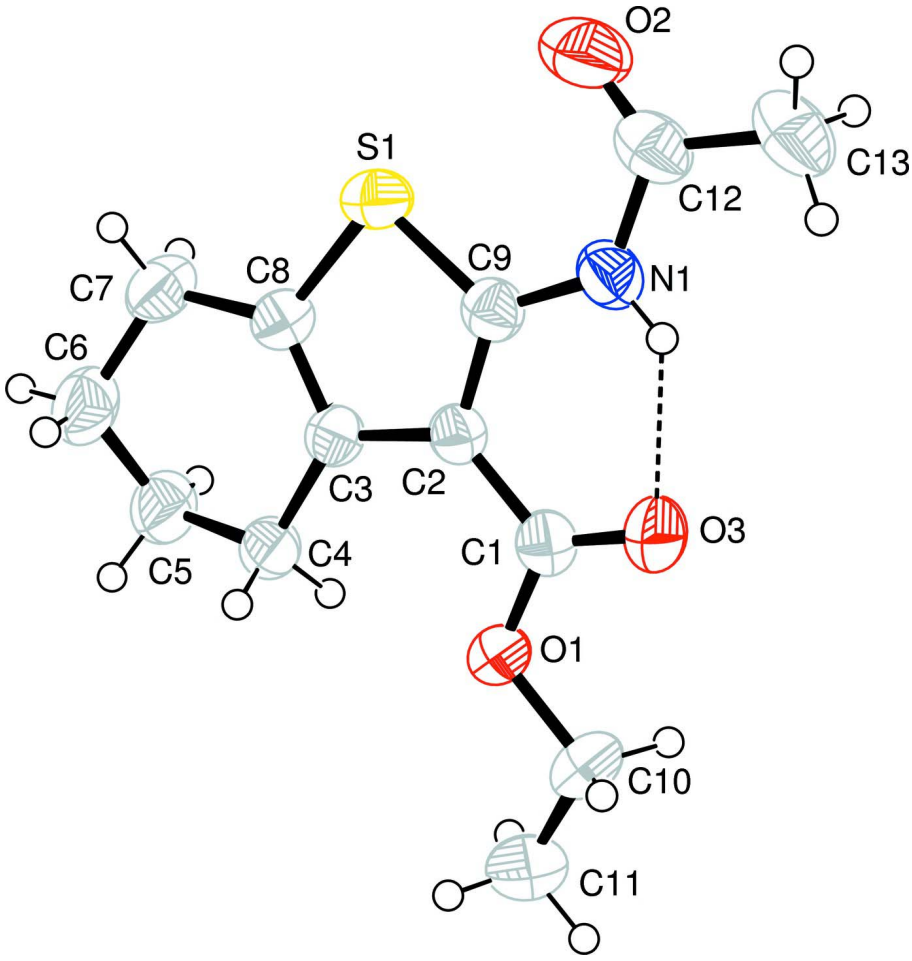

Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level. The dotted line show intramolecular H-bonding.

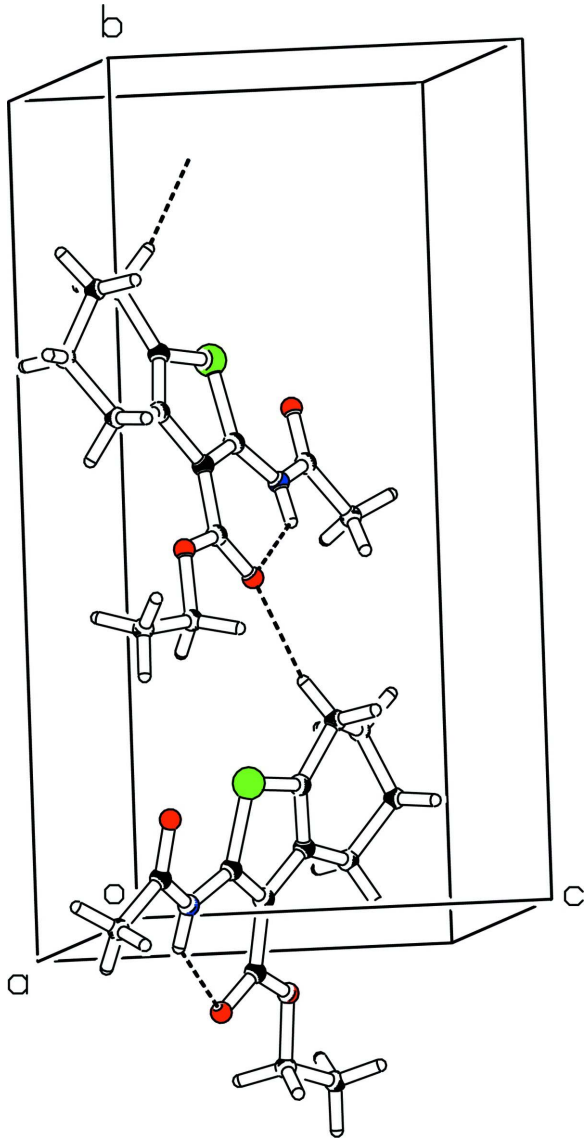

Figure 2

The partial packing, which shows that molecules form C(7) chains extending along the *b* axis.

Ethyl 2-acetamido-4,5,6,7-tetrahydro-1-benzothiophene-3-carboxylate

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$C_{13}H_{17}NO_3S$
$M_r = 267.34$
Monoclinic, $P2_1/c$
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$a = 10.4267\ (4)$ Å
$b = 16.6554\ (7)$ Å
$c = 8.0961\ (3)$ Å
$\beta = 109.610\ (1)°$
$V = 1324.43\ (9)$ Å^3
$Z = 4$

$F(000) = 568$
$D_x = 1.341$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1831 reflections
$\theta = 2.4–25.3°$
$\mu = 0.24$ mm^{-1}
$T = 296$ K
Prism, colorless
0.28 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.10 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.953, T_{max} = 0.958

9994 measured reflections
2389 independent reflections
1831 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.3°, θ_{min} = 2.4°
h = −12→12
k = −19→19
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.105
S = 1.05
2389 reflections
165 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
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.4757P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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 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	−0.07735 (6)	0.14705 (4)	0.26031 (7)	0.0539 (2)
O1	0.22774 (14)	−0.08502 (9)	0.4124 (2)	0.0570 (5)
O2	−0.31724 (17)	0.09817 (13)	0.0095 (2)	0.0848 (8)
O3	0.03140 (16)	−0.11247 (10)	0.2017 (2)	0.0646 (6)
N1	−0.15117 (16)	0.00537 (11)	0.0871 (2)	0.0514 (6)
C1	0.1049 (2)	−0.06486 (13)	0.3047 (3)	0.0471 (7)
C2	0.06878 (18)	0.01882 (12)	0.3219 (2)	0.0408 (6)
C3	0.14704 (19)	0.07906 (12)	0.4432 (2)	0.0414 (6)
C4	0.2851 (2)	0.06876 (13)	0.5811 (3)	0.0488 (7)
C5	0.3212 (3)	0.13991 (14)	0.7081 (3)	0.0653 (8)
C6	0.2832 (3)	0.21870 (14)	0.6192 (3)	0.0690 (9)
C7	0.1317 (2)	0.22493 (13)	0.5254 (3)	0.0591 (8)
C8	0.0810 (2)	0.14974 (12)	0.4223 (3)	0.0459 (7)
C9	−0.05484 (19)	0.04848 (13)	0.2159 (3)	0.0444 (7)
C10	0.2653 (3)	−0.16924 (14)	0.4095 (4)	0.0742 (10)
C11	0.3917 (3)	−0.18249 (18)	0.5583 (4)	0.0926 (13)
C12	−0.2783 (2)	0.03142 (17)	−0.0096 (3)	0.0592 (9)

C13	−0.3644 (2)	−0.02882 (17)	−0.1363 (3)	0.0729 (9)
H1	−0.12894	−0.04227	0.06581	0.0616*
H4A	0.35340	0.06365	0.52467	0.0585*
H4B	0.28594	0.01977	0.64624	0.0585*
H5A	0.27501	0.13359	0.79324	0.0783*
H5B	0.41841	0.13940	0.77120	0.0783*
H6A	0.33036	0.22575	0.53537	0.0828*
H6B	0.31193	0.26133	0.70545	0.0828*
H7A	0.08553	0.23261	0.61017	0.0709*
H7B	0.11222	0.27086	0.44724	0.0709*
H10A	0.27966	−0.18170	0.29995	0.0889*
H10B	0.19331	−0.20356	0.42015	0.0889*
H11A	0.37805	−0.16655	0.66514	0.1389*
H11B	0.46381	−0.15119	0.54198	0.1389*
H11C	0.41548	−0.23836	0.56466	0.1389*
H13A	−0.41082	−0.00295	−0.24610	0.1095*
H13B	−0.43007	−0.05116	−0.08977	0.1095*
H13C	−0.30755	−0.07092	−0.15408	0.1095*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0487 (3)	0.0506 (4)	0.0574 (4)	0.0098 (3)	0.0112 (3)	0.0046 (3)
O1	0.0475 (8)	0.0389 (9)	0.0741 (10)	0.0041 (7)	0.0066 (7)	−0.0051 (7)
O2	0.0593 (11)	0.0900 (15)	0.0849 (13)	0.0170 (10)	−0.0024 (9)	−0.0038 (11)
O3	0.0608 (10)	0.0489 (10)	0.0724 (10)	−0.0073 (8)	0.0068 (8)	−0.0142 (8)
N1	0.0423 (10)	0.0563 (12)	0.0496 (10)	−0.0041 (8)	0.0077 (8)	−0.0017 (9)
C1	0.0448 (12)	0.0451 (13)	0.0505 (12)	−0.0040 (10)	0.0148 (10)	−0.0003 (10)
C2	0.0381 (10)	0.0407 (12)	0.0439 (11)	−0.0022 (9)	0.0142 (8)	0.0012 (9)
C3	0.0441 (11)	0.0401 (12)	0.0407 (10)	−0.0008 (9)	0.0152 (9)	0.0028 (9)
C4	0.0471 (12)	0.0443 (12)	0.0481 (12)	−0.0007 (9)	0.0069 (9)	0.0002 (10)
C5	0.0655 (15)	0.0544 (15)	0.0590 (14)	−0.0056 (12)	−0.0015 (12)	−0.0052 (12)
C6	0.0776 (17)	0.0502 (15)	0.0660 (15)	−0.0082 (12)	0.0067 (13)	−0.0079 (12)
C7	0.0723 (16)	0.0418 (14)	0.0577 (13)	0.0061 (11)	0.0147 (12)	−0.0016 (10)
C8	0.0488 (12)	0.0424 (12)	0.0461 (11)	0.0033 (9)	0.0154 (9)	0.0028 (10)
C9	0.0428 (11)	0.0465 (12)	0.0448 (11)	−0.0016 (9)	0.0159 (9)	0.0035 (9)
C10	0.0717 (17)	0.0403 (14)	0.103 (2)	0.0095 (12)	0.0192 (15)	−0.0085 (13)
C11	0.0626 (17)	0.0620 (18)	0.139 (3)	0.0162 (14)	0.0149 (18)	0.0113 (18)
C12	0.0469 (13)	0.0751 (18)	0.0500 (13)	−0.0002 (12)	0.0087 (10)	0.0071 (12)
C13	0.0527 (14)	0.096 (2)	0.0562 (14)	−0.0106 (14)	0.0001 (11)	0.0007 (14)

Geometric parameters (Å, º)

S1—C8	1.731 (2)	C10—C11	1.474 (4)
S1—C9	1.714 (2)	C12—C13	1.499 (4)
O1—C1	1.328 (3)	C4—H4A	0.9700
O1—C10	1.459 (3)	C4—H4B	0.9700
O2—C12	1.211 (3)	C5—H5A	0.9700

O3—C1	1.218 (3)	C5—H5B	0.9700
N1—C9	1.382 (3)	C6—H6A	0.9700
N1—C12	1.364 (3)	C6—H6B	0.9700
N1—H1	0.8600	C7—H7A	0.9700
C1—C2	1.463 (3)	C7—H7B	0.9700
C2—C9	1.378 (3)	C10—H10A	0.9700
C2—C3	1.449 (3)	C10—H10B	0.9700
C3—C4	1.506 (3)	C11—H11A	0.9600
C3—C8	1.346 (3)	C11—H11B	0.9600
C4—C5	1.531 (3)	C11—H11C	0.9600
C5—C6	1.485 (3)	C13—H13A	0.9600
C6—C7	1.509 (4)	C13—H13B	0.9600
C7—C8	1.499 (3)	C13—H13C	0.9600
C8—S1—C9	91.19 (10)	C4—C5—H5A	109.00
C1—O1—C10	115.95 (19)	C4—C5—H5B	109.00
C9—N1—C12	126.0 (2)	C6—C5—H5A	109.00
C9—N1—H1	117.00	C6—C5—H5B	109.00
C12—N1—H1	117.00	H5A—C5—H5B	108.00
O1—C1—O3	122.1 (2)	C5—C6—H6A	109.00
O3—C1—C2	124.3 (2)	C5—C6—H6B	109.00
O1—C1—C2	113.63 (18)	C7—C6—H6A	109.00
C1—C2—C9	119.95 (18)	C7—C6—H6B	109.00
C1—C2—C3	128.32 (17)	H6A—C6—H6B	108.00
C3—C2—C9	111.72 (18)	C6—C7—H7A	110.00
C2—C3—C8	111.88 (17)	C6—C7—H7B	110.00
C2—C3—C4	127.17 (18)	C8—C7—H7A	110.00
C4—C3—C8	120.95 (18)	C8—C7—H7B	110.00
C3—C4—C5	111.59 (19)	H7A—C7—H7B	108.00
C4—C5—C6	113.14 (19)	O1—C10—H10A	110.00
C5—C6—C7	111.6 (2)	O1—C10—H10B	110.00
C6—C7—C8	109.67 (18)	C11—C10—H10A	110.00
C3—C8—C7	126.2 (2)	C11—C10—H10B	110.00
S1—C8—C3	112.99 (16)	H10A—C10—H10B	108.00
S1—C8—C7	120.80 (16)	C10—C11—H11A	109.00
N1—C9—C2	125.09 (19)	C10—C11—H11B	109.00
S1—C9—N1	122.70 (16)	C10—C11—H11C	109.00
S1—C9—C2	112.22 (16)	H11A—C11—H11B	109.00
O1—C10—C11	107.6 (2)	H11A—C11—H11C	109.00
N1—C12—C13	115.0 (2)	H11B—C11—H11C	109.00
O2—C12—N1	121.4 (2)	C12—C13—H13A	109.00
O2—C12—C13	123.5 (2)	C12—C13—H13B	109.00
C3—C4—H4A	109.00	C12—C13—H13C	109.00
C3—C4—H4B	109.00	H13A—C13—H13B	109.00
C5—C4—H4A	109.00	H13A—C13—H13C	110.00
C5—C4—H4B	109.00	H13B—C13—H13C	109.00
H4A—C4—H4B	108.00		

C9—S1—C8—C3	−0.74 (18)	C9—C2—C3—C4	178.71 (19)
C9—S1—C8—C7	−179.82 (19)	C9—C2—C3—C8	−0.6 (2)
C8—S1—C9—N1	−179.31 (19)	C1—C2—C9—S1	179.01 (15)
C8—S1—C9—C2	0.37 (17)	C1—C2—C9—N1	−1.3 (3)
C10—O1—C1—O3	−4.5 (3)	C3—C2—C9—S1	0.0 (2)
C10—O1—C1—C2	175.9 (2)	C3—C2—C9—N1	179.72 (19)
C1—O1—C10—C11	−169.7 (2)	C2—C3—C4—C5	−167.96 (19)
C12—N1—C9—S1	−5.9 (3)	C8—C3—C4—C5	11.3 (3)
C12—N1—C9—C2	174.4 (2)	C2—C3—C8—S1	0.9 (2)
C9—N1—C12—O2	1.2 (4)	C2—C3—C8—C7	179.9 (2)
C9—N1—C12—C13	−177.8 (2)	C4—C3—C8—S1	−178.48 (15)
O1—C1—C2—C3	−2.4 (3)	C4—C3—C8—C7	0.6 (3)
O1—C1—C2—C9	178.80 (19)	C3—C4—C5—C6	−41.8 (3)
O3—C1—C2—C3	178.0 (2)	C4—C5—C6—C7	61.4 (3)
O3—C1—C2—C9	−0.8 (3)	C5—C6—C7—C8	−46.2 (3)
C1—C2—C3—C4	−0.2 (3)	C6—C7—C8—S1	−164.26 (17)
C1—C2—C3—C8	−179.5 (2)	C6—C7—C8—C3	16.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	2.03	2.674 (2)	131
C7—H7*B*···O3^{i}	0.97	2.50	3.392 (3)	153

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1/2.