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Methyl 2-(thiophene-2-carboxamido)-benzoate

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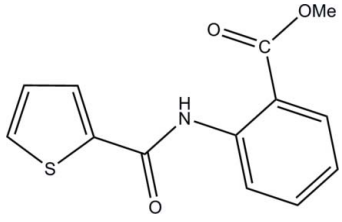
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.004 Å; R factor = 0.033; wR factor = 0.090; data-to-parameter ratio = 8.5.

The title compound, $C_{13}H_{11}NO_3S$, was synthesized from methyl anthranilate, triethylamine and 2-thiophenoyl chloride in benzene. The molecular conformation is stabilized by an intramolecular N—H···O hydrogen bond. The dihedral angle between the rings is 2.74 (12)°. In the crystal, C—H···O interactions link neighbouring molecules into a three-dimensional network.

Related literature

For the synthesis, see: Sladowska *et al.* (1980).



Experimental

Crystal data

$C_{13}H_{11}NO_3S$
M_r = 261.29
Orthorhombic, $Pca2_1$
a = 19.2845 (4) Å
b = 3.86753 (8) Å
c = 15.6430 (3) Å
V = 1166.71 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.48 mm^{-1}
T = 123 K
0.45 × 0.18 × 0.04 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Absorption correction: analytical (*CrysAlis PRO*; Agilent, 2012)
T_{min} = 0.573, T_{max} = 0.908
2379 measured reflections
1422 independent reflections
1381 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.090
S = 1.04
1422 reflections
168 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 206 Friedel pairs
Flack parameter: −0.02 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O2	0.85 (2)	1.99 (3)	2.665 (3)	135 (4)
C9—H9A···O2^{i}	0.95	2.43	3.380 (3)	174
C13—H13A···O1^{ii}	0.98	2.52	3.433 (4)	154

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

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

DPS and SP are grateful to Banaras Hindu University, Varanasi, for financial support. RJB acknowledges the NSF–MRI program (grant No. CHE0619278) for funds to purchase the X-ray diffractometer. SKG wishes to acknowledge the USIEF for the award of a Fulbright–Nehru Senior Research Fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5852).

References

Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sladowska, H., Sieklucka-Dziuba, M., Rajtar, G., Sodowski, M., Kleinrok, Z., Kirino, O., Yamamoto, S. & Kato, T. (1980). *Agric. Biol. Chem.* **44**, 2143–2147.

supplementary materials

Acta Cryst. (2012). E**68**, o1765 [doi:10.1107/S160053681202082X]

Methyl 2-(thiophene-2-carboxamido)benzoate

Durga Prasad Singh, Seema Pratap, Ray J. Butcher and Sushil K. Gupta

Comment

As part of our studies of substituent effects on the structures of amides, we report here the crystal structure of the amide, methyl 2-(thiophene-2-carboxamido)benzoate. The structure of the title compound is shown in Fig. 1. The conformation of the molecule with respect to the carbonyl and anthranilate part is nearly planar as reflected by torsion angles C4—C5—N1—C6, C6—N1—C5—O1 and C3—C4—C5—O1 of 179.6 (2), -0.8 (5) and 178.3 (3) Å respectively. The 2-thiophenoyl and anthranilate groups are *trans* to each other across the C5—N1 bond. Bonds C5—O1 and C12—O2 show typical double bond character with bond lengths of 1.226 (3) and 1.211 (4) respectively, while N1—C5, N1—C6 and C12—O3 show partial double bond character with bond lengths of 1.365 (4), 1.401 (4), and 1.329 (3) Å respectively. All bond length and bond angles confirm the sp^2 hybridization for all C and N atoms except C_{13}, indicating that the whole molecule is planar.

Experimental

The title compound was synthesized using the literature procedure (Sladowska *et al.*, 1980). To a solution of methyl anthranilate (10 mmol) and triethyl amine(10mmol) in benzene(30 ml) was added 2-thiophenoyl chloride (10 mmol) in benzene(10 ml) with stirring at room temperature. After stirring for three hour, the reaction mixture was washed successively with water, dilute HCl and aqueous Na_2CO_3 and the organic layer was dried over dry Na_2SO_4. After removal of the solvent, the residue was recrystallized from ethanol. Colorless, needles type crystal suitable for X-ray diffraction were obtained after few days. Yield 78%.

Refinement

H atoms were placed in calculated positions with C—H = 0.95–0.98 Å with isotropic displacement parameters fixed to U_{iso}(H) = 1.2 U_{eq}(C). The H attached to N was isotropically refined but with the N—H distance restrained to 0.88 Å.

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

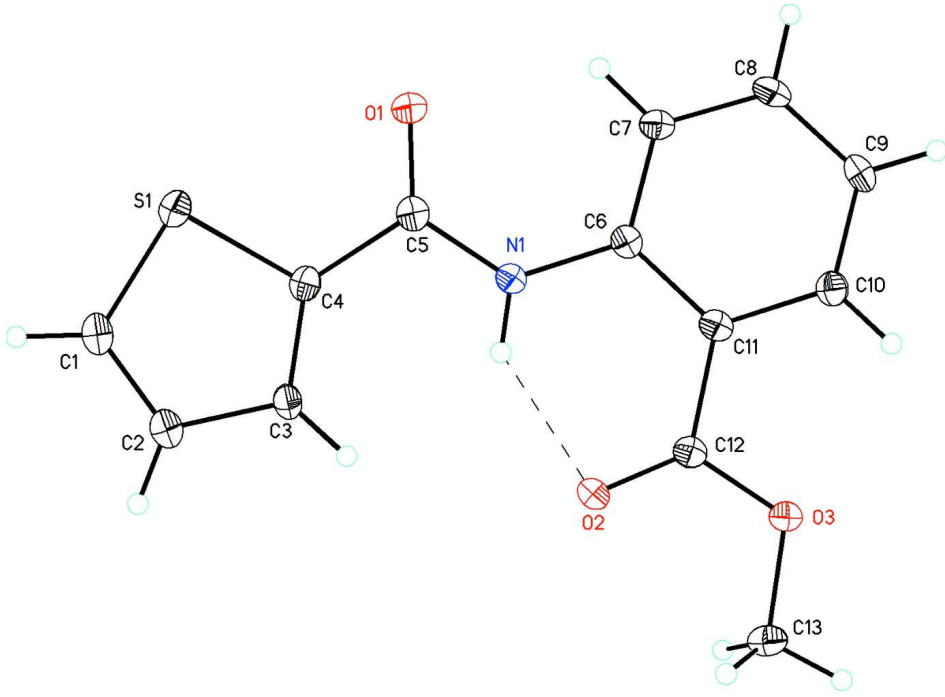


Figure 1

The molecular structure of compound(I) showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as dashed lines.

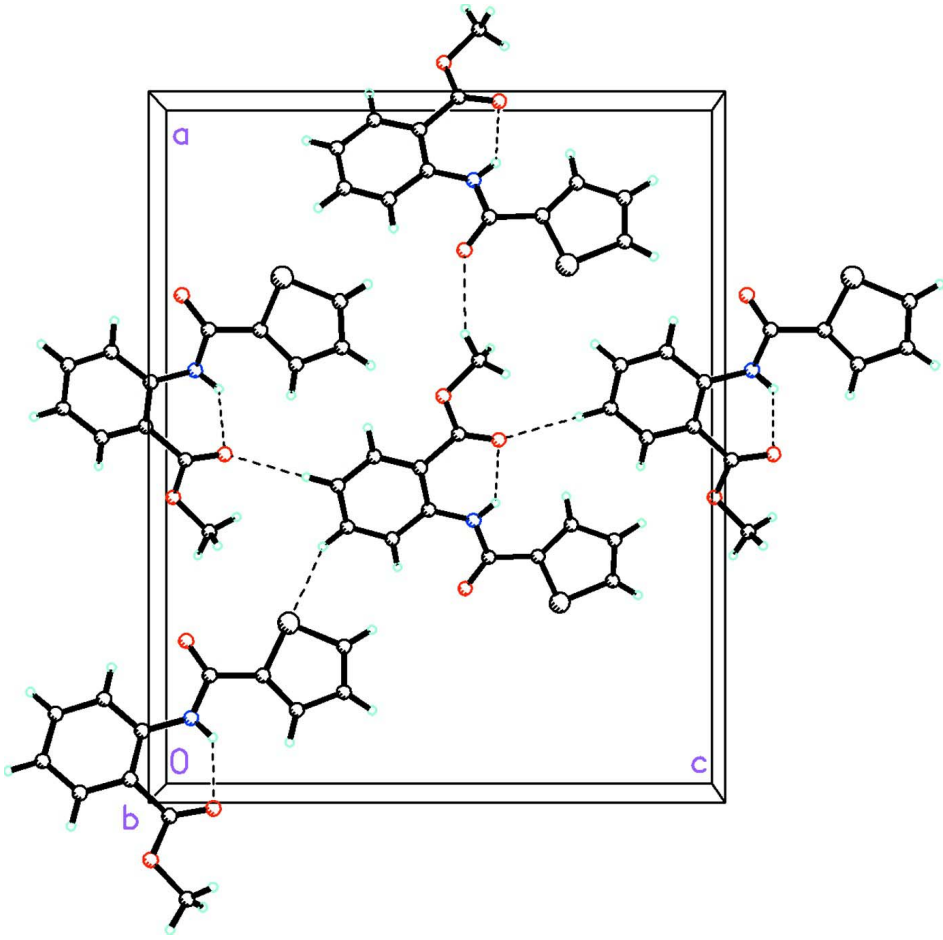


Figure 2

Part of the crystal structure of (I) showing intramolecular and intermolecular interactions as dashed lines.

Methyl 2-(thiophene-2-carboxamido)benzoate

Crystal data

$C_{13}H_{11}NO_3S$
$M_r = 261.29$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 19.2845$ (4) Å
$b = 3.86753$ (8) Å
$c = 15.6430$ (3) Å
$V = 1166.71$ (4) Å^3
$Z = 4$

$F(000) = 544$
$D_x = 1.488$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 1440 reflections
$\theta = 2.8$–$75.1°$
$\mu = 2.48$ mm^{-1}
$T = 123$ K
Needle, colorless
0.45 × 0.18 × 0.04 mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
ω scans

Absorption correction: analytical (*CrysAlis PRO*; Agilent, 2012)
$T_{min} = 0.573$, $T_{max} = 0.908$
2379 measured reflections
1422 independent reflections
1381 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$

θ_{max} = 75.2°, θ_{min} = 4.6°
h = −15→23
k = −4→4
l = −6→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.090
S = 1.04
1422 reflections
168 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2 + 0.0898P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 206 Friedel pairs
Flack parameter: −0.02 (2)

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	0.25886 (3)	0.20133 (16)	0.72770 (5)	0.03006 (19)
O1	0.28253 (10)	0.3657 (6)	0.54680 (14)	0.0304 (4)
O2	0.51080 (11)	0.8922 (6)	0.60945 (13)	0.0339 (5)
O3	0.57519 (10)	1.1406 (5)	0.50816 (14)	0.0308 (4)
N1	0.38872 (12)	0.6290 (6)	0.56315 (15)	0.0252 (5)
H1B	0.4151 (18)	0.698 (9)	0.603 (2)	0.037 (10)*
C1	0.29286 (16)	0.2186 (7)	0.8287 (2)	0.0307 (6)
H1A	0.2694	0.1372	0.8781	0.037*
C2	0.35685 (16)	0.3611 (8)	0.8301 (2)	0.0304 (6)
H2A	0.3831	0.3919	0.8810	0.037*
C3	0.38110 (14)	0.4607 (7)	0.74725 (17)	0.0249 (5)
H3A	0.4251	0.5621	0.7364	0.030*
C4	0.33202 (13)	0.3900 (7)	0.68499 (18)	0.0251 (5)
C5	0.33132 (14)	0.4577 (7)	0.59160 (18)	0.0247 (6)
C6	0.40520 (13)	0.7321 (7)	0.4797 (2)	0.0231 (5)
C7	0.36094 (14)	0.6720 (7)	0.41074 (19)	0.0262 (6)
H7A	0.3173	0.5642	0.4201	0.031*
C8	0.38002 (15)	0.7678 (7)	0.3290 (2)	0.0279 (6)
H8A	0.3498	0.7185	0.2826	0.034*
C9	0.44255 (15)	0.9352 (7)	0.31331 (18)	0.0277 (6)
H9A	0.4550	1.0010	0.2569	0.033*
C10	0.48604 (14)	1.0039 (7)	0.38091 (19)	0.0261 (6)

H10A	0.5284	1.1221	0.3708	0.031*
C11	0.46910 (13)	0.9032 (7)	0.46454 (18)	0.0241 (6)
C12	0.51909 (14)	0.9743 (7)	0.53544 (18)	0.0253 (5)
C13	0.62744 (16)	1.2115 (9)	0.5729 (2)	0.0359 (7)
H13A	0.6645	1.3525	0.5479	0.054*
H13B	0.6469	0.9929	0.5937	0.054*
H13C	0.6062	1.3367	0.6206	0.054*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0264 (3)	0.0293 (3)	0.0345 (4)	−0.0023 (2)	0.0046 (3)	0.0010 (4)
O1	0.0248 (9)	0.0361 (10)	0.0303 (10)	−0.0051 (8)	−0.0037 (8)	−0.0012 (9)
O2	0.0329 (10)	0.0454 (11)	0.0235 (10)	−0.0104 (9)	−0.0042 (9)	0.0033 (10)
O3	0.0254 (9)	0.0401 (11)	0.0268 (9)	−0.0077 (9)	−0.0033 (9)	0.0017 (9)
N1	0.0234 (10)	0.0295 (11)	0.0227 (11)	−0.0024 (9)	−0.0005 (9)	−0.0013 (9)
C1	0.0342 (14)	0.0284 (14)	0.0294 (15)	0.0026 (11)	0.0072 (12)	0.0021 (12)
C2	0.0341 (14)	0.0299 (13)	0.0272 (14)	0.0021 (12)	0.0037 (12)	0.0013 (12)
C3	0.0281 (12)	0.0242 (11)	0.0224 (12)	−0.0015 (10)	0.0044 (11)	0.0004 (10)
C4	0.0230 (12)	0.0226 (12)	0.0298 (14)	0.0020 (10)	0.0037 (11)	0.0009 (10)
C5	0.0245 (12)	0.0208 (12)	0.0288 (14)	0.0035 (10)	0.0019 (11)	0.0001 (10)
C6	0.0239 (13)	0.0211 (11)	0.0243 (12)	0.0017 (9)	0.0006 (11)	−0.0002 (11)
C7	0.0232 (12)	0.0263 (13)	0.0290 (15)	−0.0009 (10)	−0.0042 (12)	−0.0018 (11)
C8	0.0301 (13)	0.0282 (12)	0.0255 (14)	0.0035 (11)	−0.0094 (12)	−0.0020 (11)
C9	0.0333 (13)	0.0287 (14)	0.0210 (12)	0.0050 (11)	0.0007 (11)	0.0033 (11)
C10	0.0256 (12)	0.0251 (13)	0.0277 (14)	0.0007 (11)	0.0018 (11)	0.0005 (10)
C11	0.0239 (12)	0.0218 (11)	0.0267 (14)	0.0028 (10)	−0.0032 (11)	−0.0021 (11)
C12	0.0240 (12)	0.0260 (12)	0.0258 (13)	0.0006 (10)	−0.0017 (10)	−0.0011 (10)
C13	0.0292 (14)	0.0416 (17)	0.0370 (16)	−0.0082 (12)	−0.0090 (14)	0.0002 (15)

Geometric parameters (Å, º)

S1—C1	1.711 (3)	C4—C5	1.484 (4)
S1—C4	1.723 (3)	C6—C7	1.395 (4)
O1—C5	1.226 (3)	C6—C11	1.419 (3)
O2—C12	1.211 (4)	C7—C8	1.381 (4)
O3—C12	1.329 (3)	C7—H7A	0.9500
O3—C13	1.454 (4)	C8—C9	1.390 (4)
N1—C5	1.365 (4)	C8—H8A	0.9500
N1—C6	1.401 (4)	C9—C10	1.375 (4)
N1—H1B	0.852 (19)	C9—H9A	0.9500
C1—C2	1.352 (4)	C10—C11	1.404 (4)
C1—H1A	0.9500	C10—H10A	0.9500
C2—C3	1.431 (4)	C11—C12	1.495 (4)
C2—H2A	0.9500	C13—H13A	0.9800
C3—C4	1.385 (4)	C13—H13B	0.9800
C3—H3A	0.9500	C13—H13C	0.9800
C1—S1—C4	91.59 (15)	C8—C7—H7A	119.7
C12—O3—C13	115.6 (3)	C6—C7—H7A	119.7

C5—N1—C6	128.7 (2)	C7—C8—C9	121.3 (3)
C5—N1—H1B	113 (3)	C7—C8—H8A	119.4
C6—N1—H1B	117 (3)	C9—C8—H8A	119.4
C2—C1—S1	112.4 (2)	C10—C9—C8	118.9 (3)
C2—C1—H1A	123.8	C10—C9—H9A	120.6
S1—C1—H1A	123.8	C8—C9—H9A	120.6
C1—C2—C3	113.2 (3)	C9—C10—C11	121.4 (2)
C1—C2—H2A	123.4	C9—C10—H10A	119.3
C3—C2—H2A	123.4	C11—C10—H10A	119.3
C4—C3—C2	111.1 (2)	C10—C11—C6	119.2 (2)
C4—C3—H3A	124.4	C10—C11—C12	119.4 (2)
C2—C3—H3A	124.4	C6—C11—C12	121.4 (3)
C3—C4—C5	131.5 (2)	O2—C12—O3	122.8 (3)
C3—C4—S1	111.7 (2)	O2—C12—C11	125.2 (3)
C5—C4—S1	116.7 (2)	O3—C12—C11	112.1 (2)
O1—C5—N1	125.2 (3)	O3—C13—H13A	109.5
O1—C5—C4	121.2 (3)	O3—C13—H13B	109.5
N1—C5—C4	113.5 (2)	H13A—C13—H13B	109.5
C7—C6—N1	122.3 (2)	O3—C13—H13C	109.5
C7—C6—C11	118.6 (3)	H13A—C13—H13C	109.5
N1—C6—C11	119.1 (3)	H13B—C13—H13C	109.5
C8—C7—C6	120.6 (3)		

C4—S1—C1—C2	0.0 (2)	C11—C6—C7—C8	2.0 (4)
S1—C1—C2—C3	−0.5 (3)	C6—C7—C8—C9	−1.9 (4)
C1—C2—C3—C4	0.8 (4)	C7—C8—C9—C10	0.3 (4)
C2—C3—C4—C5	177.1 (3)	C8—C9—C10—C11	1.2 (4)
C2—C3—C4—S1	−0.8 (3)	C9—C10—C11—C6	−1.1 (4)
C1—S1—C4—C3	0.4 (2)	C9—C10—C11—C12	178.1 (2)
C1—S1—C4—C5	−177.8 (2)	C7—C6—C11—C10	−0.5 (4)
C6—N1—C5—O1	−0.8 (5)	N1—C6—C11—C10	179.4 (3)
C6—N1—C5—C4	179.6 (2)	C7—C6—C11—C12	−179.7 (2)
C3—C4—C5—O1	178.3 (3)	N1—C6—C11—C12	0.2 (4)
S1—C4—C5—O1	−3.9 (4)	C13—O3—C12—O2	1.5 (4)
C3—C4—C5—N1	−2.1 (4)	C13—O3—C12—C11	−178.4 (2)
S1—C4—C5—N1	175.66 (19)	C10—C11—C12—O2	−178.1 (3)
C5—N1—C6—C7	1.1 (4)	C6—C11—C12—O2	1.0 (4)
C5—N1—C6—C11	−178.8 (3)	C10—C11—C12—O3	1.7 (4)
N1—C6—C7—C8	−177.9 (3)	C6—C11—C12—O3	−179.1 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O2	0.85 (2)	1.99 (3)	2.665 (3)	135 (4)
C9—H9*A*···O2^{i}	0.95	2.43	3.380 (3)	174
C13—H13*A*···O1^{ii}	0.98	2.52	3.433 (4)	154

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