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## Structure Reports

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A monoclinic polymorph of *N*-(3-chlorophenyl)benzamideAamer Saeed,<sup>a\*</sup> Muhammad Arshad<sup>b</sup> and Jim Simpson<sup>c</sup><sup>a</sup>Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan,<sup>b</sup>Chemistry Division, Directorate of Science, PINSTECH, Nilore, Islamabad, Pakistan,and <sup>c</sup>Department of Chemistry, University of Otago, PO Box 56, Dunedin, New

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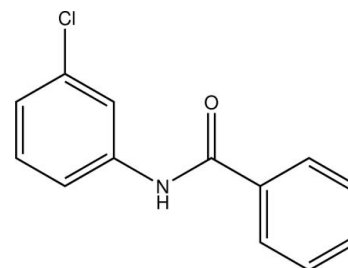
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Key indicators: single-crystal X-ray study; *T* = 90 K; mean  $\sigma(\text{C}-\text{C})$  = 0.003 Å; *R* factor = 0.041; *wR* factor = 0.104; data-to-parameter ratio = 13.8.

The title compound,  $\text{C}_{13}\text{H}_{10}\text{ClNO}$ , (I), is a polymorph of the structure, (II), first reported by Gowda *et al.* [*Acta Cryst.* (2008), E64, o462]. In the original report, the compound crystallized in the orthorhombic space group *Pbca* (*Z* = 8), whereas the structure reported here is monoclinic *P2<sub>1</sub>/c* (*Z* = 4). The principal difference between the two forms lies in the relative orientations of the phenyl and benzene rings [dihedral angle = 8.90 (13)° for (I) and 61.0 (1)° for (II)]. The inclination of the amide –CONH– units to the benzoyl ring is more similar [15.8 (7)° for (I) and 18.2 (2)° for (II)]. In both forms, the N–H bonds are *anti* to the 3-chloro substituents of the aniline rings. In the crystal, intermolecular N–H···O hydrogen bonds form *C*(4) chains along *c*. These chains are bolstered by weak C–H···O interactions that generate  $R_2^1(6)$  and  $R_2^1(7)$  ring motifs.

## Related literature

For background to the biological activity of *N*-substituted benzamides and their use in synthesis, see: Saeed *et al.* (2010). For the orthorhombic polymorph of (I), see: Gowda, Tokarčík *et al.* (2008). For the structures of related chlorophenylbenzamides, see: Gowda *et al.* (2007*a,b,c*); Gowda, Foro *et al.* (2008). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987).



## Experimental

## Crystal data

 $\text{C}_{13}\text{H}_{10}\text{ClNO}$  $M_r = 231.67$ Monoclinic, *P2<sub>1</sub>/c* $a = 12.5598$  (17) Å $b = 10.2782$  (14) Å $c = 9.0788$  (13) Å $\beta = 109.421$  (5)° $V = 1105.3$  (3) Å<sup>3</sup>*Z* = 4Mo  $K\alpha$  radiation $\mu = 0.32$  mm<sup>-1</sup>*T* = 90 K

0.57 × 0.22 × 0.03 mm

## Data collection

Bruker APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2006)

 $T_{\min} = 0.743$ ,  $T_{\max} = 1.000$ 

6385 measured reflections

2045 independent reflections

1475 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.047$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.041$  $wR(F^2) = 0.104$ *S* = 1.04

2045 reflections

148 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.29$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>

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>
N1–H1···O1 <sup>1</sup>	0.88 (2)	1.99 (2)	2.841 (2)	163 (2)
C7–H7···O1 <sup>1</sup>	0.95	2.45	3.228 (3)	139
C13–H13···O1 <sup>1</sup>	0.95	2.71	3.301 (3)	121

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

Data collection: APEX2 (Bruker 2006); cell refinement: APEX2 and SAINT (Bruker 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) and TITAN2000 (Hunter & Simpson, 1999); molecular graphics: SHELXTL (Sheldrick, 2008) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: SHELXL97, enCIFer (Allen *et al.*, 2004), PLATON (Spek, 2009) and publCIF (Westrip, 2010).

We thank the University of Otago for purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5145).

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## supporting information

*Acta Cryst.* (2010). E66, o2808–o2809 [https://doi.org/10.1107/S1600536810040262]

## A monoclinic polymorph of *N*-(3-chlorophenyl)benzamide

Aamer Saeed, Muhammad Arshad and Jim Simpson

### S1. Comment

*N*-substituted benzamides have numerous pharmaceutical and synthetic applications (Saeed *et al.*, 2010). The title compound, (I), is a monoclinic polymorph of the structure of this benzamide derivative which crystallizes in the space group P21/c. An alternative structure, (II), in the orthorhombic space group Pbc<sub>a</sub> was reported previously by Gowda, Tokarčík *et al.*, (2008).

Bond distances in the molecule are normal (Allen *et al.*, 1987) and very similar to those in the orthorhombic polymorph and in closely related chlorophenylbenzamide derivatives (Gowda *et al.*, 2007*a,b,c*); Gowda, Foro *et al.*, 2008). However, the two polymorphs differ markedly in the relative orientations of the C2···C6 phenyl and C8···C13 benzene rings [dihedral angles 8.90 (13) for (I) and 61.0 (1) for (II)]. The inclination of the amide –C1O1N1H1– units to the C2···C6 ring is more similar [15.8 (7) for (I) and 18.2 (2) for (II)]. The N1–H1 bonds in both forms are *anti* to the Cl1 substituents of the C8···C13 aniline rings. This behaviour parallels that observed with *N*-(2-chlorophenyl)benzamide (Gowda *et al.*, 2007*a*) and *N*-(3,4-dichlorophenyl)benzamide (Gowda *et al.*, 2007*c*), whereas the a *syn* conformation is favoured in *N*-(2,3-dichlorophenyl)benzamide (Gowda *et al.*, 2007*b*)

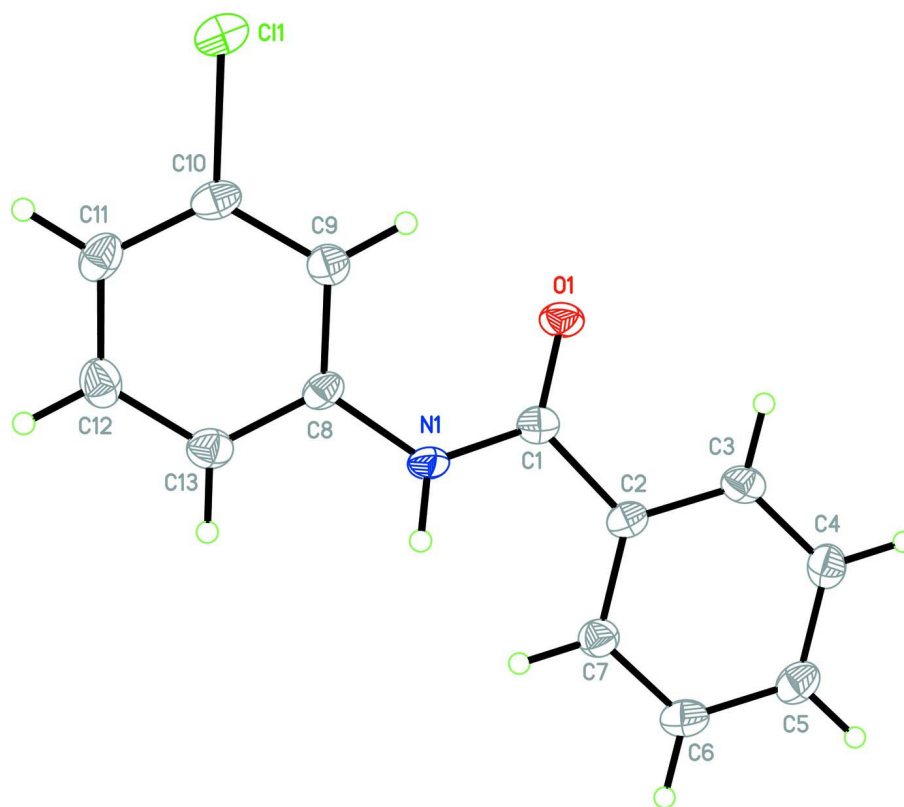
In the crystal structure, Fig. 2, intermolecular N1–H1···O1 hydrogen bonds form C4 chains along the *c* axis (Bernstein *et al.* 1995). These chains are further stabilized by weak C7–H5···O1 and C13–H13···O1 interactions that generate an  $R_2^1(7)$  motif involving the an *ortho*-H atom from the phenyl ring and an  $R_2^1(6)$  motif incorporating an *ortho*-H atom from the chlorobenzene ring respectively.

### S2. Experimental

Freshly distilled benzoyl chloride (1 mmol) in CHCl<sub>3</sub> was treated with 3-chloroaniline (3.5 mmol) under a nitrogen atmosphere at reflux for 2.5 h. Upon cooling, the reaction mixture was diluted with CHCl<sub>3</sub> and washed consecutively with 1 *M* aq HCl and saturated aq NaHCO<sub>3</sub>. The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. Crystallization of the residue from ethanol afforded the title compound (83%) as colourless crystals: Anal. calcd. for C<sub>13</sub>H<sub>10</sub>ClN<sub>2</sub>O: C, 67.39; H, 4.35; N, 6.05; found: C, 67.21; H, 4.42; N, 6.10%.

### S3. Refinement

The H atom bound to N1 was located in a difference map and refined isotropically. All other H-atoms were positioned geometrically and refined using a riding model with d(C–H) = 0.95 Å,  $U_{\text{iso}} = 1.2U_{\text{eq}}$  (C).



**Figure 1**

The structure of (I) with displacement ellipsoids for the non-hydrogen atoms drawn at the 50% probability level.

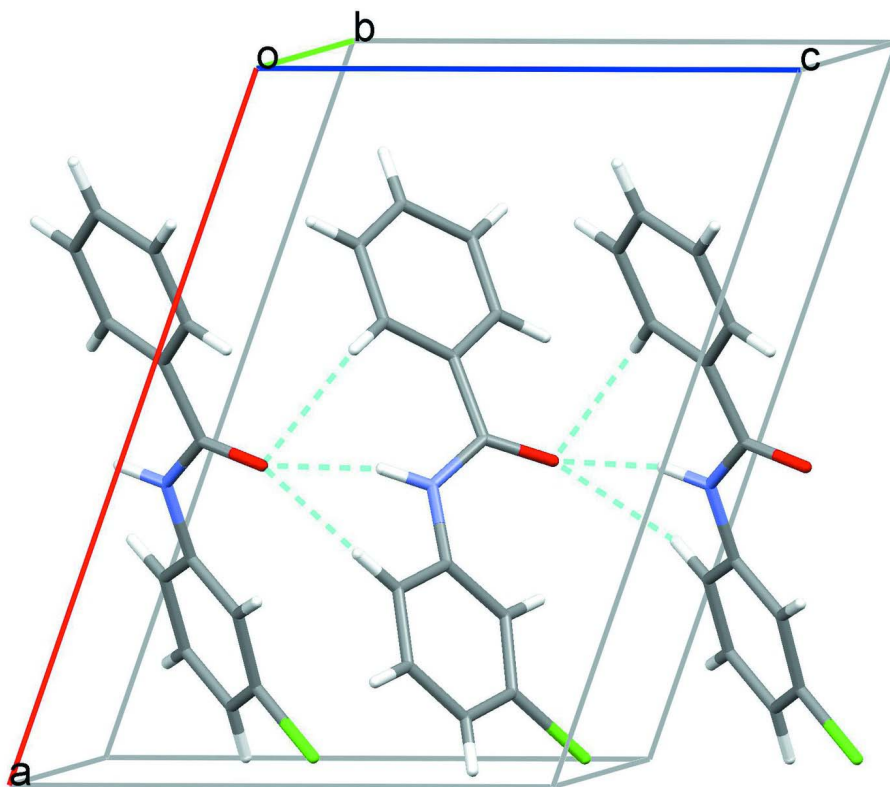


Figure 2

Crystal packing for (I) viewed along the *b* axis with hydrogen bonds drawn as dashed lines.

### *N*-(3-chlorophenyl)benzamide

#### Crystal data

$C_{13}H_{10}ClNO$

$M_r = 231.67$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2_1/c$

$a = 12.5598$  (17) Å

$b = 10.2782$  (14) Å

$c = 9.0788$  (13) Å

$\beta = 109.421$  (5)°

$V = 1105.3$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 480$

$D_x = 1.392$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1106 reflections

$\theta = 2.6$ – $25.3$ °

$\mu = 0.32$  mm<sup>-1</sup>

$T = 90$  K

Rectangular plate, colourless

$0.57 \times 0.22 \times 0.03$  mm

#### Data collection

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2006)

$T_{\min} = 0.743$ ,  $T_{\max} = 1.000$

6385 measured reflections

2045 independent reflections

1475 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 25.5$ °,  $\theta_{\min} = 3.1$ °

$h = -15$ → $15$

$k = -12$ → $12$

$l = -9$ → $10$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.041$  $wR(F^2) = 0.104$  $S = 1.04$ 

2045 reflections

148 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sitesH atoms treated by a mixture of independent  
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.2503P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 0.29 \text{ e } \text{Å}^{-3}$  $\Delta\rho_{\min} = -0.26 \text{ e } \text{Å}^{-3}$ *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 ( $\text{Å}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.58959 (16)	0.22008 (18)	0.5519 (2)	0.0208 (4)
H1	0.5656 (19)	0.202 (2)	0.451 (3)	0.025*
C1	0.52721 (18)	0.3046 (2)	0.6039 (3)	0.0187 (5)
O1	0.55935 (12)	0.34896 (15)	0.73758 (17)	0.0223 (4)
C2	0.41446 (18)	0.3409 (2)	0.4894 (2)	0.0180 (5)
C3	0.35873 (19)	0.4469 (2)	0.5262 (3)	0.0217 (5)
H3	0.3924	0.4929	0.6211	0.026*
C4	0.25445 (19)	0.4852 (2)	0.4248 (3)	0.0233 (5)
H4	0.2167	0.5572	0.4509	0.028*
C5	0.20480 (19)	0.4194 (2)	0.2856 (3)	0.0251 (6)
H5	0.1341	0.4473	0.2151	0.030*
C7	0.36288 (18)	0.2734 (2)	0.3510 (3)	0.0215 (5)
H7	0.3992	0.1998	0.3257	0.026*
C8	0.69653 (18)	0.1670 (2)	0.6371 (2)	0.0188 (5)
C9	0.77139 (19)	0.2275 (2)	0.7677 (3)	0.0204 (5)
H9	0.7516	0.3062	0.8071	0.025*
C10	0.87564 (19)	0.1698 (2)	0.8386 (3)	0.0220 (5)
C11	0.9070 (2)	0.0546 (2)	0.7871 (3)	0.0242 (5)
H11	0.9786	0.0165	0.8393	0.029*
C12	0.8316 (2)	-0.0039 (2)	0.6576 (3)	0.0256 (6)
H12	0.8516	-0.0832	0.6200	0.031*
C13	0.72744 (19)	0.0511 (2)	0.5819 (3)	0.0232 (5)
H13	0.6767	0.0101	0.4921	0.028*
Cl1	0.96981 (5)	0.24773 (6)	1.00047 (7)	0.0317 (2)

C6	0.25863 (19)	0.3132 (2)	0.2499 (3)	0.0259 (6)
H6	0.2241	0.2669	0.1554	0.031*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0224 (10)	0.0271 (10)	0.0112 (10)	0.0031 (8)	0.0033 (9)	-0.0010 (9)
C1	0.0210 (12)	0.0203 (11)	0.0161 (12)	-0.0029 (9)	0.0079 (10)	0.0025 (10)
O1	0.0246 (9)	0.0287 (8)	0.0135 (9)	0.0002 (7)	0.0063 (7)	-0.0022 (7)
C2	0.0179 (11)	0.0220 (11)	0.0152 (12)	-0.0020 (9)	0.0067 (9)	0.0035 (9)
C3	0.0295 (13)	0.0221 (11)	0.0153 (12)	-0.0033 (10)	0.0100 (10)	0.0011 (10)
C4	0.0261 (13)	0.0247 (12)	0.0228 (13)	0.0053 (10)	0.0131 (11)	0.0038 (10)
C5	0.0184 (12)	0.0322 (13)	0.0237 (14)	0.0022 (10)	0.0056 (10)	0.0041 (11)
C7	0.0193 (12)	0.0248 (12)	0.0199 (12)	0.0003 (9)	0.0057 (10)	-0.0008 (10)
C8	0.0195 (12)	0.0229 (12)	0.0134 (12)	0.0015 (9)	0.0048 (10)	0.0052 (9)
C9	0.0252 (13)	0.0213 (12)	0.0161 (12)	0.0014 (9)	0.0085 (10)	0.0027 (10)
C10	0.0226 (12)	0.0285 (12)	0.0138 (12)	-0.0027 (10)	0.0043 (10)	0.0043 (10)
C11	0.0217 (12)	0.0309 (13)	0.0207 (13)	0.0052 (10)	0.0081 (10)	0.0081 (11)
C12	0.0329 (14)	0.0221 (12)	0.0244 (14)	0.0059 (10)	0.0129 (12)	0.0040 (10)
C13	0.0272 (13)	0.0240 (12)	0.0179 (12)	-0.0010 (10)	0.0068 (10)	0.0019 (10)
Cl1	0.0246 (3)	0.0376 (4)	0.0256 (4)	0.0006 (3)	-0.0013 (3)	-0.0033 (3)
C6	0.0227 (13)	0.0319 (13)	0.0204 (13)	-0.0038 (11)	0.0034 (10)	-0.0022 (11)

*Geometric parameters (Å, °)*

N1—C1	1.356 (3)	C7—H7	0.9500
N1—C8	1.418 (3)	C8—C9	1.392 (3)
N1—H1	0.88 (2)	C8—C13	1.396 (3)
C1—O1	1.232 (2)	C9—C10	1.386 (3)
C1—C2	1.499 (3)	C9—H9	0.9500
C2—C7	1.392 (3)	C10—C11	1.378 (3)
C2—C3	1.394 (3)	C10—Cl1	1.745 (2)
C3—C4	1.384 (3)	C11—C12	1.378 (3)
C3—H3	0.9500	C11—H11	0.9500
C4—C5	1.386 (3)	C12—C13	1.381 (3)
C4—H4	0.9500	C12—H12	0.9500
C5—C6	1.378 (3)	C13—H13	0.9500
C5—H5	0.9500	C6—H6	0.9500
C7—C6	1.387 (3)		
C1—N1—C8	127.52 (19)	C9—C8—C13	119.9 (2)
C1—N1—H1	117.3 (16)	C9—C8—N1	122.8 (2)
C8—N1—H1	114.8 (16)	C13—C8—N1	117.3 (2)
O1—C1—N1	122.8 (2)	C10—C9—C8	118.1 (2)
O1—C1—C2	121.1 (2)	C10—C9—H9	121.0
N1—C1—C2	116.09 (19)	C8—C9—H9	121.0
C7—C2—C3	119.1 (2)	C11—C10—C9	122.9 (2)
C7—C2—C1	123.4 (2)	C11—C10—Cl1	119.38 (18)

C3—C2—C1	117.5 (2)	C9—C10—C11	117.77 (18)
C4—C3—C2	120.1 (2)	C12—C11—C10	118.1 (2)
C4—C3—H3	119.9	C12—C11—H11	120.9
C2—C3—H3	119.9	C10—C11—H11	120.9
C3—C4—C5	120.5 (2)	C11—C12—C13	121.0 (2)
C3—C4—H4	119.8	C11—C12—H12	119.5
C5—C4—H4	119.8	C13—C12—H12	119.5
C6—C5—C4	119.6 (2)	C12—C13—C8	120.1 (2)
C6—C5—H5	120.2	C12—C13—H13	120.0
C4—C5—H5	120.2	C8—C13—H13	120.0
C6—C7—C2	120.2 (2)	C5—C6—C7	120.4 (2)
C6—C7—H7	119.9	C5—C6—H6	119.8
C2—C7—H7	119.9	C7—C6—H6	119.8

*Hydrogen-bond geometry (Å, °)*

<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>
N1—H1 $\cdots$ O1 <sup>i</sup>	0.88 (2)	1.99 (2)	2.841 (2)	163 (2)
C7—H7 $\cdots$ O1 <sup>i</sup>	0.95	2.45	3.228 (3)	139
C13—H13 $\cdots$ O1 <sup>i</sup>	0.95	2.71	3.301 (3)	121

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