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## Structure Reports

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# (E)-4-Chloro-N'-(5-hydroxy-2-nitrobenzylidene)benzohydrazide

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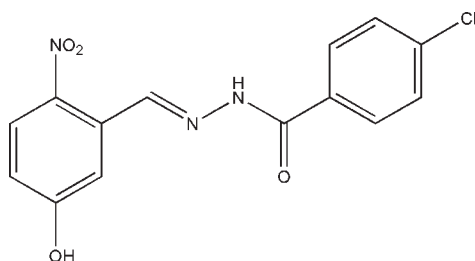
Received 24 August 2009; accepted 4 September 2009

 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.041;  $wR$  factor = 0.112; data-to-parameter ratio = 14.4.

The title compound,  $\text{C}_{14}\text{H}_{10}\text{ClN}_3\text{O}_4$ , was synthesized by the reaction of 5-hydroxy-2-nitrobenzaldehyde with an equimolar quantity of 4-chlorobenzohydrazide in methanol. The molecule displays an *E* configuration about the  $\text{C}=\text{N}$  bond. The dihedral angle between the two benzene rings is  $3.9(2)^\circ$ . In the crystal structure, molecules are linked through intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, forming chains running along the *b* axis.

## Related literature

For examples of the crystal structures of hydrazone compounds, see: Mohd Lair *et al.* (2009); Fun *et al.* (2008); Li & Ban (2009); Zhu *et al.* (2009); Yang (2007); You *et al.* (2008). For the hydrazone compounds previously reported by our group, see: Qu *et al.* (2008); Yang *et al.* (2008), Cao & Lu (2009*a,b*), Cao (2009*a,b*).



## Experimental

## Crystal data

$\text{C}_{14}\text{H}_{10}\text{ClN}_3\text{O}_4$   
 $M_r = 319.70$   
 Triclinic,  $P\bar{1}$   
 $a = 7.5386(2)$  Å  
 $b = 8.1677(2)$  Å

$c = 12.3435(4)$  Å  
 $\alpha = 90.820(3)^\circ$   
 $\beta = 106.056(2)^\circ$   
 $\gamma = 109.014(2)^\circ$   
 $V = 685.95(4)$  Å<sup>3</sup>

$Z = 2$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.30$  mm<sup>-1</sup>

$T = 298$  K  
 $0.23 \times 0.20 \times 0.20$  mm

## Data collection

Bruker SMART 1K diffractometer  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2001)  
 $T_{\min} = 0.934$ ,  $T_{\max} = 0.942$

4267 measured reflections  
 2933 independent reflections  
 2444 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.013$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.112$   
 $S = 1.04$   
 2933 reflections  
 203 parameters  
 1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.37$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}3-\text{H}3\cdots\text{O}4^{\text{i}}$	0.82	1.89	2.7093 (17)	174
$\text{N}2-\text{H}2\cdots\text{O}1^{\text{ii}}$	0.896 (10)	2.290 (11)	3.1649 (18)	165 (2)

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

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2273).

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## supporting information

*Acta Cryst.* (2009). E65, o2415 [doi:10.1107/S1600536809035740]

**(E)-4-Chloro-N'-(5-hydroxy-2-nitrobenzylidene)benzohydrazide**

Guo-Biao Cao

**S1. Comment**

In the last few years, the crystal structures of a large number of hydrazone compounds have been reported (Mohd Lair *et al.*, 2009; Fun *et al.*, 2008; Li & Ban, 2009; Zhu *et al.*, 2009; Yang, 2007; You *et al.*, 2008). As a continuation of our work in this area (Qu *et al.*, 2008; Yang *et al.*, 2008; Cao & Lu, 2009a,b), the title new hydrazone compound, derived from the reaction of 5-hydroxy-2-nitrobenzaldehyde with an equimolar quantity of 4-chlorobenzohydrazide, is reported.

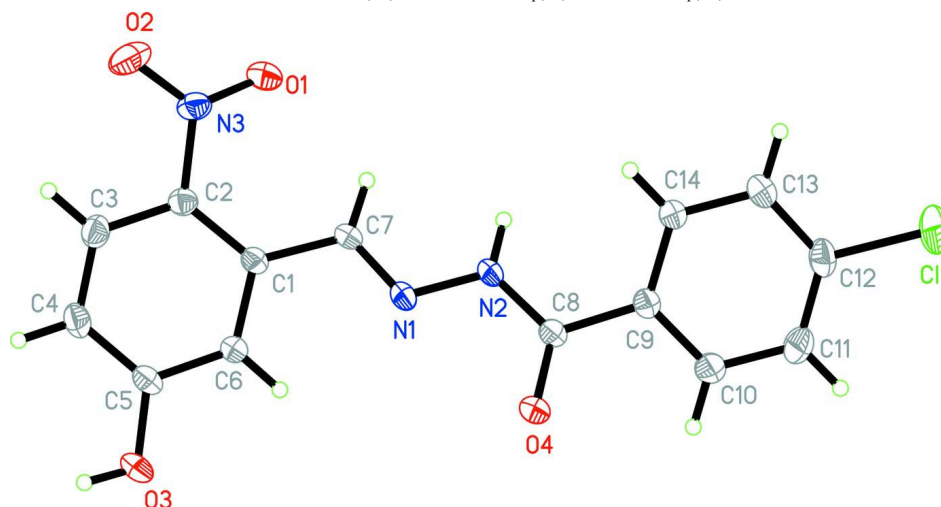
In the title compound, Fig. 1, the dihedral angle between the two benzene rings is  $3.9(2)^\circ$ . The molecule displays an *E* configuration about the C=N bond. In the crystal structure, molecules are linked through intermolecular N—H $\cdots$ O and O—H $\cdots$ O hydrogen bonds, Table 1, to form chains running along the *b* axis, Fig. 2.

**S2. Experimental**

The compound was prepared by refluxing equimolar quantities of 5-hydroxy-2-nitrobenzaldehyde with 4-chlorobenzohydrazide in methanol. Colorless block-like crystals were formed by slow evaporation of the solution in air.

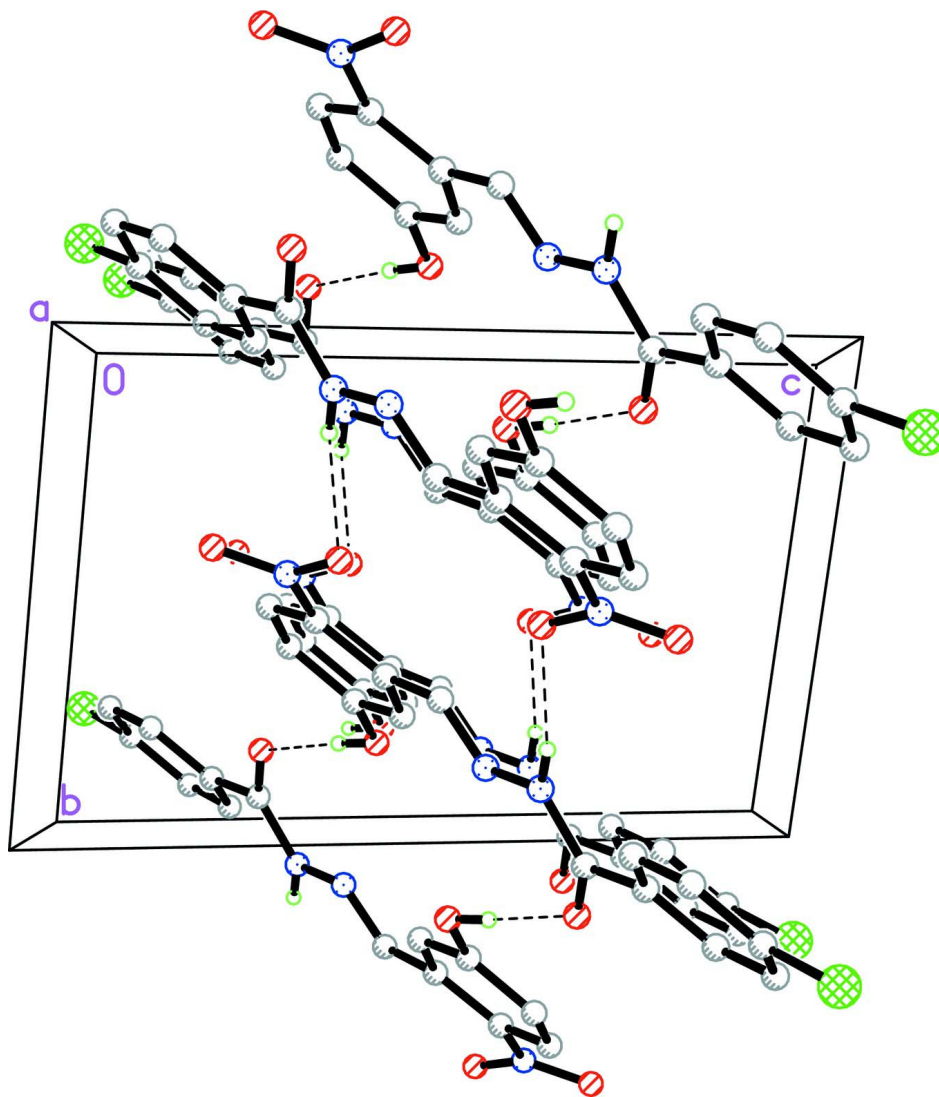
**S3. Refinement**

H2 was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. The other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 Å, O—H distance of 0.82 Å, and with  $U_{\text{iso}}(\text{H})$  set at  $1.2U_{\text{eq}}(\text{C})$  and  $1.5U_{\text{eq}}(\text{O})$ .



**Figure 1**

The molecular structure of the title compound with ellipsoids drawn at the 30% probability level.



**Figure 2**

The molecular packing of the title compound, viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

**(*E*)-4-Chloro-*N'*-(5-hydroxy-2-nitrobenzylidene)benzohydrazide**

*Crystal data*

$C_{14}H_{10}ClN_3O_4$

$M_r = 319.70$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 7.5386\ (2)\ \text{\AA}$

$b = 8.1677\ (2)\ \text{\AA}$

$c = 12.3435\ (4)\ \text{\AA}$

$\alpha = 90.820\ (3)^\circ$

$\beta = 106.056\ (2)^\circ$

$\gamma = 109.014\ (2)^\circ$

$V = 685.95\ (4)\ \text{\AA}^3$

$Z = 2$

$F(000) = 328$

$D_x = 1.548\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2079 reflections

$\theta = 2.6\text{--}30.1^\circ$

$\mu = 0.30\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, colorless

$0.23 \times 0.20 \times 0.20\ \text{mm}$

*Data collection*

Bruker SMART 1K  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\omega$  scans  
Absorption correction: multi-scan  
(*SADABS*; Bruker, 2001)  
 $T_{\min} = 0.934$ ,  $T_{\max} = 0.942$

4267 measured reflections  
2933 independent reflections  
2444 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.013$   
 $\theta_{\max} = 27.0^\circ$ ,  $\theta_{\min} = 1.7^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -10 \rightarrow 10$   
 $l = -15 \rightarrow 15$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.041$   
 $wR(F^2) = 0.112$   
 $S = 1.04$   
2933 reflections  
203 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 atoms treated by a mixture of independent  
and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.2581P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	1.99310 (8)	1.22131 (10)	1.02766 (6)	0.0873 (3)
N1	0.89654 (18)	0.85347 (17)	0.58743 (11)	0.0363 (3)
N2	1.08279 (18)	0.88906 (18)	0.65933 (12)	0.0365 (3)
N3	0.6482 (2)	0.46355 (18)	0.30087 (13)	0.0424 (3)
O1	0.7870 (2)	0.43682 (18)	0.36768 (12)	0.0570 (4)
O2	0.5932 (3)	0.4105 (2)	0.20048 (13)	0.0756 (5)
O3	0.22232 (17)	0.82467 (17)	0.43739 (11)	0.0489 (3)
H3	0.1313	0.8286	0.3839	0.073*
O4	1.08646 (17)	1.14913 (16)	0.72801 (11)	0.0458 (3)
C1	0.6246 (2)	0.67228 (19)	0.44254 (13)	0.0299 (3)
C2	0.5411 (2)	0.55749 (19)	0.34209 (13)	0.0332 (3)
C3	0.3510 (2)	0.5302 (2)	0.27424 (14)	0.0407 (4)
H3A	0.2995	0.4535	0.2079	0.049*
C4	0.2379 (2)	0.6149 (2)	0.30395 (15)	0.0407 (4)
H4	0.1098	0.5944	0.2590	0.049*

C5	0.3180 (2)	0.7317 (2)	0.40214 (14)	0.0352 (3)
C6	0.5077 (2)	0.75750 (19)	0.46992 (13)	0.0322 (3)
H6	0.5584	0.8346	0.5361	0.039*
C7	0.8272 (2)	0.7162 (2)	0.51835 (13)	0.0326 (3)
H7	0.9012	0.6456	0.5154	0.039*
C8	1.1687 (2)	1.0418 (2)	0.72753 (13)	0.0337 (3)
C9	1.3720 (2)	1.0754 (2)	0.80277 (13)	0.0335 (3)
C10	1.4371 (3)	1.1911 (2)	0.90032 (14)	0.0438 (4)
H10	1.3523	1.2403	0.9185	0.053*
C11	1.6278 (3)	1.2339 (3)	0.97075 (15)	0.0511 (5)
H11	1.6716	1.3107	1.0366	0.061*
C12	1.7510 (3)	1.1612 (3)	0.94169 (15)	0.0474 (4)
C13	1.6913 (2)	1.0459 (2)	0.84568 (15)	0.0437 (4)
H13	1.7774	0.9981	0.8278	0.052*
C14	1.5000 (2)	1.0026 (2)	0.77637 (14)	0.0362 (3)
H14	1.4565	0.9239	0.7115	0.043*
H2	1.141 (3)	0.809 (2)	0.660 (2)	0.080*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0458 (3)	0.1068 (5)	0.0802 (4)	0.0259 (3)	-0.0248 (3)	-0.0205 (4)
N1	0.0269 (6)	0.0390 (7)	0.0413 (7)	0.0161 (5)	0.0021 (5)	-0.0002 (6)
N2	0.0270 (6)	0.0385 (7)	0.0425 (7)	0.0179 (5)	0.0005 (5)	-0.0022 (6)
N3	0.0471 (8)	0.0356 (7)	0.0476 (8)	0.0178 (6)	0.0149 (7)	-0.0033 (6)
O1	0.0527 (8)	0.0596 (8)	0.0656 (9)	0.0360 (7)	0.0091 (7)	-0.0089 (7)
O2	0.0986 (13)	0.0879 (11)	0.0505 (9)	0.0534 (10)	0.0138 (8)	-0.0181 (8)
O3	0.0364 (6)	0.0568 (8)	0.0598 (8)	0.0295 (6)	0.0082 (6)	0.0016 (6)
O4	0.0368 (6)	0.0450 (7)	0.0554 (7)	0.0237 (5)	0.0024 (5)	-0.0080 (5)
C1	0.0283 (7)	0.0294 (7)	0.0337 (7)	0.0125 (6)	0.0087 (6)	0.0057 (6)
C2	0.0349 (8)	0.0296 (7)	0.0366 (8)	0.0133 (6)	0.0100 (6)	0.0028 (6)
C3	0.0387 (9)	0.0365 (8)	0.0385 (9)	0.0094 (7)	0.0029 (7)	-0.0023 (7)
C4	0.0280 (7)	0.0411 (9)	0.0454 (9)	0.0100 (7)	0.0014 (7)	0.0048 (7)
C5	0.0293 (7)	0.0343 (8)	0.0452 (9)	0.0151 (6)	0.0110 (6)	0.0093 (7)
C6	0.0297 (7)	0.0334 (7)	0.0345 (8)	0.0140 (6)	0.0074 (6)	0.0015 (6)
C7	0.0290 (7)	0.0356 (8)	0.0371 (8)	0.0178 (6)	0.0081 (6)	0.0030 (6)
C8	0.0296 (7)	0.0394 (8)	0.0344 (8)	0.0159 (6)	0.0080 (6)	0.0024 (6)
C9	0.0294 (7)	0.0370 (8)	0.0330 (8)	0.0126 (6)	0.0060 (6)	0.0035 (6)
C10	0.0402 (9)	0.0535 (10)	0.0380 (9)	0.0191 (8)	0.0088 (7)	-0.0043 (7)
C11	0.0483 (10)	0.0603 (11)	0.0339 (9)	0.0147 (9)	0.0008 (8)	-0.0090 (8)
C12	0.0338 (8)	0.0557 (11)	0.0412 (9)	0.0133 (8)	-0.0040 (7)	0.0026 (8)
C13	0.0320 (8)	0.0511 (10)	0.0471 (10)	0.0187 (7)	0.0053 (7)	0.0038 (8)
C14	0.0320 (8)	0.0390 (8)	0.0360 (8)	0.0145 (6)	0.0050 (6)	-0.0011 (6)

*Geometric parameters (Å, °)*

C11—C12	1.7399 (17)	C4—C5	1.389 (2)
N1—C7	1.266 (2)	C4—H4	0.9300

N1—N2	1.3714 (17)	C5—C6	1.388 (2)
N2—C8	1.351 (2)	C6—H6	0.9300
N2—H2	0.896 (10)	C7—H7	0.9300
N3—O2	1.2160 (19)	C8—C9	1.489 (2)
N3—O1	1.2245 (19)	C9—C10	1.387 (2)
N3—C2	1.456 (2)	C9—C14	1.390 (2)
O3—C5	1.3447 (19)	C10—C11	1.385 (2)
O3—H3	0.8200	C10—H10	0.9300
O4—C8	1.2285 (18)	C11—C12	1.372 (3)
C1—C6	1.391 (2)	C11—H11	0.9300
C1—C2	1.401 (2)	C12—C13	1.375 (3)
C1—C7	1.476 (2)	C13—C14	1.383 (2)
C2—C3	1.388 (2)	C13—H13	0.9300
C3—C4	1.375 (2)	C14—H14	0.9300
C3—H3A	0.9300		
C7—N1—N2	116.59 (12)	C1—C6—H6	118.8
C8—N2—N1	118.41 (12)	N1—C7—C1	117.56 (13)
C8—N2—H2	122.9 (16)	N1—C7—H7	121.2
N1—N2—H2	118.7 (16)	C1—C7—H7	121.2
O2—N3—O1	121.99 (15)	O4—C8—N2	122.68 (14)
O2—N3—C2	118.32 (15)	O4—C8—C9	120.98 (14)
O1—N3—C2	119.67 (14)	N2—C8—C9	116.33 (13)
C5—O3—H3	109.5	C10—C9—C14	119.36 (14)
C6—C1—C2	116.51 (13)	C10—C9—C8	117.71 (14)
C6—C1—C7	117.54 (13)	C14—C9—C8	122.85 (14)
C2—C1—C7	125.89 (13)	C11—C10—C9	120.34 (16)
C3—C2—C1	121.43 (14)	C11—C10—H10	119.8
C3—C2—N3	115.96 (14)	C9—C10—H10	119.8
C1—C2—N3	122.59 (14)	C12—C11—C10	118.85 (16)
C4—C3—C2	120.83 (15)	C12—C11—H11	120.6
C4—C3—H3A	119.6	C10—C11—H11	120.6
C2—C3—H3A	119.6	C11—C12—C13	122.33 (16)
C3—C4—C5	119.00 (14)	C11—C12—C11	118.73 (14)
C3—C4—H4	120.5	C13—C12—C11	118.92 (15)
C5—C4—H4	120.5	C12—C13—C14	118.43 (16)
O3—C5—C6	116.31 (14)	C12—C13—H13	120.8
O3—C5—C4	123.86 (14)	C14—C13—H13	120.8
C6—C5—C4	119.83 (14)	C13—C14—C9	120.67 (15)
C5—C6—C1	122.38 (14)	C13—C14—H14	119.7
C5—C6—H6	118.8	C9—C14—H14	119.7

*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>
O3—H3 $\cdots$ O4 <sup>i</sup>	0.82	1.89	2.7093 (17)	174

N2—H2···O1 <sup>ii</sup>	0.90 (1)	2.29 (1)	3.1649 (18)	165 (2)
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Symmetry codes: (i)  $-x+1, -y+2, -z+1$ ; (ii)  $-x+2, -y+1, -z+1$ .