Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(2-Hydroxy-3-methoxyphenyl)-1*H*-benzimidazol-3-ium perchlorate

Chuan Chen, Hong-Xing Li, Guang-Feng Hou and Guang-Ming Li*

School of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China
Correspondence e-mail: gmli_2000@163.com

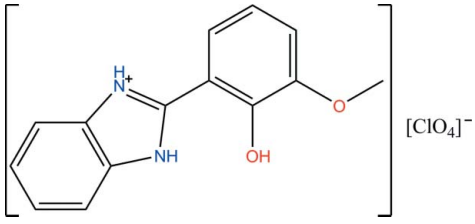
Received 12 May 2012; accepted 22 May 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.140; data-to-parameter ratio = 15.1.

In the title molecular salt, $C_{14}H_{13}N_2O_2^+\cdot ClO_4^-$, the ring systems in the cation are almost coplanar [dihedral angle = 5.53 (13)°]. Intramolecular N—H···O and O—H···O hydrogen bonds generate S(6) and S(5) rings, respectively. In the crystal, the two H atoms involved in the intramolecular hydrogen bonds also participate in intermolecular links to acceptor O atoms of the perchlorate anions. A simple intermolecular N—H···O bond also occurs. Together, these form a double-chain structure along [101].

Related literature

For a related structure, see: Yang *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{13}N_2O_2^+\cdot ClO_4^-$
M_r = 340.71
Monoclinic, $P2_1/c$
a = 7.7698 (16) Å
b = 20.462 (4) Å
c = 9.856 (2) Å
β = 113.09 (3)°
V = 1441.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 293 K
0.50 × 0.45 × 0.42 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.864, T_{max} = 0.884
13683 measured reflections
3285 independent reflections
2025 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 1.03
3285 reflections
218 parameters
15 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82 (1)	2.20 (3)	2.635 (3)	114 (3)
O1—H1···O4^{i}	0.82 (1)	2.13 (2)	2.869 (3)	151 (3)
N1—H101···O1	0.90 (1)	2.12 (3)	2.660 (3)	118 (3)
N1—H101···O5	0.90 (1)	2.04 (2)	2.819 (3)	144 (3)
N2—H102···O3^{ii}	0.89 (1)	2.06 (2)	2.857 (4)	149 (3)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalClear* (Rigaku/MSC, 2002); 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: *SHELXL97*.

This work was supported financially by the National Natural Science Foundation of China (Nos. 20872030 and 20972043), Heilongjiang Province (Nos. 2009RFXXG201, GC09A402 and 2010td03) and Heilongjiang University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6796).

References

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalClear*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yang, X. P., Jones, R. A., Wiester, M. J., Oye, M. M. & Wong, W. K. (2010). *Cryst. Growth Des.* **8**, 3430–3436.

supplementary materials

Acta Cryst. (2012). E**68**, o1920 [doi:10.1107/S1600536812023422]

2-(2-Hydroxy-3-methoxyphenyl)-1*H*-benzimidazol-3-ium perchlorate

Chuan Chen, Hong-Xing Li, Guang-Feng Hou and Guang-Ming Li

Comment

The title compound is an unexpected product during the process of preparing the N,N′-phenyl-bis(3-methoxy-salicylaldimine) and Ce complex. We speculate that the title ligand was produced form the decomposition of N,N′-phenyl-bis(3-methoxysalicylaldimine). A similar ligand and Yb cation constructed metallic cluster has been reproted by Yang's group (Yang *et al.* 2010).

In the title compound, $[C_{14}H_{13}N_2O_2][ClO_4]$, the phenyl ring and benzimidazol ring are almost conplane with small dihedral angle of 5.517 (1) ° (Figure 1). Intermolecular N—H···O and O—H···0 hydrogen bonds link these protonated ligands and perchlorate anions to form double chain structure along [101] (Figure 2, Table 1).

Experimental

A solution of N,N′-phenyl-bis(3-methoxysalicylaldimine) (0.1502 g, 0.4 mmol) in 10 mL CH_2Cl_2, was added dropwise to a solution of $[Ce(ClO_4)_3]\cdot 9H_2O$ (0.1984 g, 0.4 mmol) in 10 mL of CH_3OH solution, after then the mixsure was keeping stir about 12h. Pale brown blocks were obtained after seven days. Elemental Anal. Calc. for $C_{14}H_{13}ClN_2O_6$: C, 49.35; H, 3.85; Cl, 10.41; N, 8.22; O, 28.17 wt%, Found: C, 49.32; H, 3.86; Cl, 10.40; N, 8.24 O, 28.15 wt%.

Refinement

C-bound H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 / 0.96 Å (aromatic / methyl) and U_{iso}(H) = 1.2 \ 1.5U_{eq}(C). H atoms attached to N and O atoms were located in a differece Fourier map and refined with a restraint of N—H = 0.90 (1) Å and O—H = 0.82 (1), and with U_{iso}(H) = 1.5U_{eq}(N and O).

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO* (Rigaku, 1998); data reduction: *CrystalClear* (Rigaku/MSC, 2002); 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: *SHELXL97* (Sheldrick, 2008).

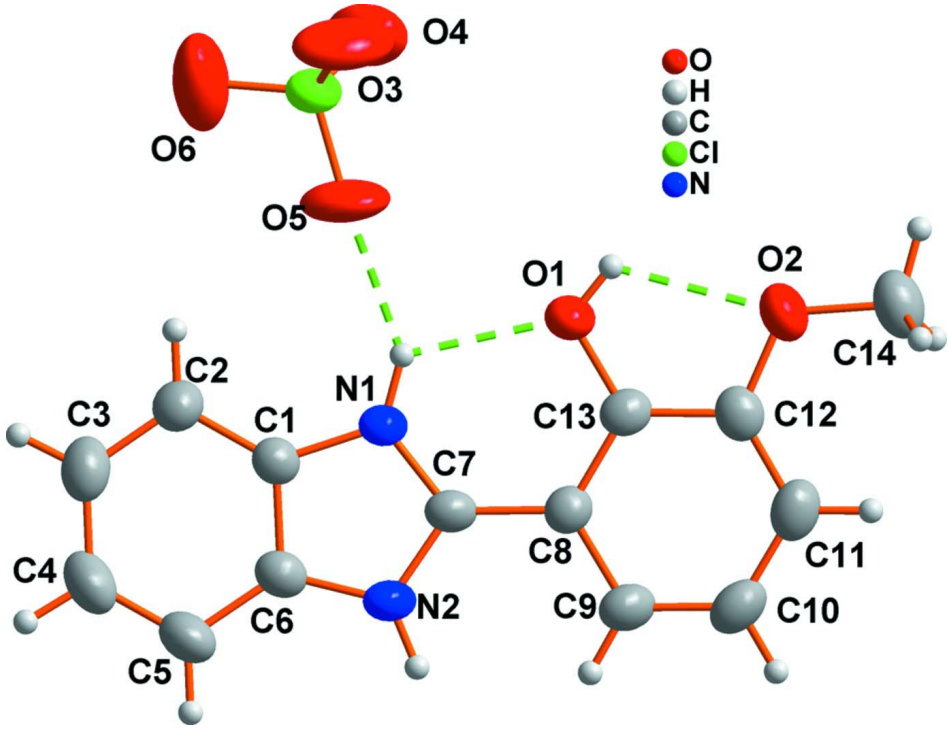


Figure 1

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

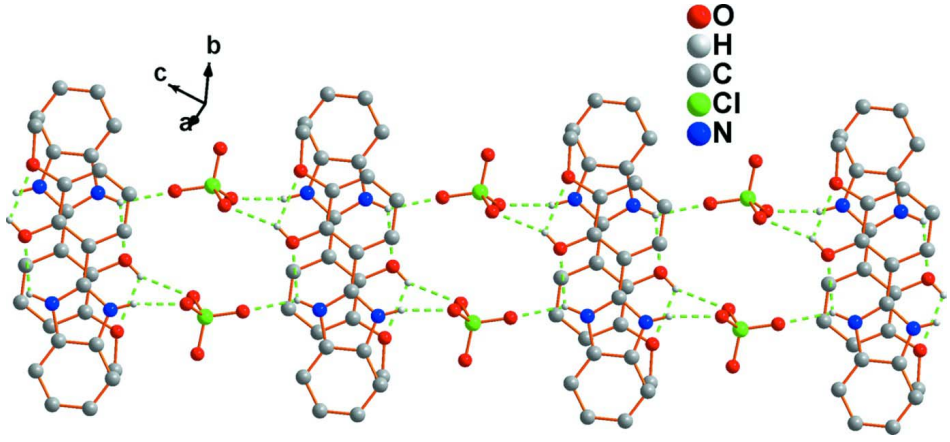


Figure 2

The double chain structure.

2-(2-Hydroxy-3-methoxyphenyl)-1*H*-benzimidazol-3-ium perchlorate

Crystal data

$C_{14}H_{13}N_2O_2^+\cdot ClO_4^-$
$M_r = 340.71$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.7698\ (16)$ Å
$b = 20.462\ (4)$ Å
$c = 9.856\ (2)$ Å
$\beta = 113.09\ (3)°$
$V = 1441.4\ (5)$ Å^3
$Z = 4$
$F(000) = 704$
$D_x = 1.570$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7910 reflections

θ = 3.0–27.4°
μ = 0.30 mm^{-1}
T = 293 K

Block, colorless
0.50 × 0.45 × 0.42 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.864, T_{max} = 0.884

13683 measured reflections
3285 independent reflections
2025 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −10→9
k = −26→26
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.140
S = 1.03
3285 reflections
218 parameters
15 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
$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.5284P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-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. isor 0.01 o5 o6 dfix 0.90 0.01 h101 n1 h102 n2 dfix 0.82 0.01 o1 h1 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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.2919 (3)	0.54938 (10)	0.6641 (2)	0.0448 (5)
H1	0.318 (5)	0.5726 (15)	0.737 (3)	0.067*
O2	0.1478 (3)	0.66205 (11)	0.6962 (2)	0.0536 (6)
O3	0.8344 (4)	0.46040 (16)	0.9925 (3)	0.0933 (10)
O4	0.5840 (4)	0.41460 (17)	1.0309 (3)	0.0886 (9)
O5	0.5714 (4)	0.43412 (17)	0.7935 (2)	0.0900 (10)
O6	0.7608 (6)	0.35259 (16)	0.9385 (4)	0.1203 (13)
C1	0.3647 (4)	0.39510 (13)	0.4381 (3)	0.0348 (6)
C2	0.4842 (4)	0.34335 (15)	0.5008 (3)	0.0451 (7)
H2	0.5525	0.3404	0.6020	0.054*
C3	0.4962 (4)	0.29644 (16)	0.4046 (4)	0.0528 (8)
H3	0.5726	0.2603	0.4424	0.063*

C4	0.3972 (4)	0.30136 (17)	0.2519 (4)	0.0556 (8)
H4	0.4126	0.2692	0.1910	0.067*
C5	0.2779 (5)	0.35259 (17)	0.1898 (3)	0.0540 (8)
H5	0.2119	0.3559	0.0884	0.065*
C6	0.2609 (4)	0.39922 (14)	0.2863 (3)	0.0401 (6)
C7	0.1917 (4)	0.48632 (14)	0.3912 (3)	0.0357 (6)
C8	0.1067 (4)	0.54718 (13)	0.4073 (3)	0.0361 (6)
C9	−0.0326 (4)	0.57649 (15)	0.2837 (3)	0.0458 (7)
H9	−0.0722	0.5561	0.1921	0.055*
C10	−0.1100 (4)	0.63489 (16)	0.2979 (3)	0.0513 (8)
H10	−0.2025	0.6537	0.2156	0.062*
C11	−0.0525 (4)	0.66657 (16)	0.4331 (3)	0.0481 (7)
H11	−0.1035	0.7068	0.4407	0.058*
C12	0.0804 (4)	0.63794 (14)	0.5558 (3)	0.0412 (7)
C13	0.1614 (4)	0.57819 (14)	0.5442 (3)	0.0371 (6)
C14	0.0861 (5)	0.72523 (17)	0.7194 (4)	0.0610 (9)
H14A	0.1256	0.7573	0.6665	0.091*
H14B	0.1392	0.7353	0.8228	0.091*
H14C	−0.0479	0.7255	0.6845	0.091*
Cl1	0.68517 (11)	0.41471 (4)	0.93839 (7)	0.0463 (2)
N1	0.3185 (3)	0.45039 (11)	0.4985 (2)	0.0351 (5)
H101	0.367 (4)	0.4594 (16)	0.5954 (13)	0.053*
N2	0.1566 (4)	0.45555 (13)	0.2634 (2)	0.0442 (6)
H102	0.079 (4)	0.4709 (17)	0.177 (2)	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0481 (12)	0.0439 (12)	0.0338 (9)	0.0034 (10)	0.0068 (9)	−0.0052 (8)
O2	0.0597 (14)	0.0452 (13)	0.0546 (12)	0.0044 (11)	0.0210 (11)	−0.0124 (10)
O3	0.0832 (19)	0.114 (3)	0.0514 (14)	−0.0493 (18)	−0.0079 (13)	0.0054 (15)
O4	0.0807 (19)	0.135 (3)	0.0592 (15)	0.0109 (19)	0.0376 (15)	0.0040 (16)
O5	0.0704 (18)	0.139 (3)	0.0349 (12)	−0.0005 (18)	−0.0075 (11)	0.0044 (14)
O6	0.186 (4)	0.062 (2)	0.146 (3)	0.038 (2)	0.100 (3)	0.0105 (19)
C1	0.0364 (14)	0.0327 (15)	0.0362 (12)	−0.0054 (11)	0.0151 (12)	−0.0020 (11)
C2	0.0398 (16)	0.0443 (18)	0.0456 (15)	0.0021 (13)	0.0107 (13)	−0.0011 (13)
C3	0.0423 (17)	0.0423 (19)	0.072 (2)	0.0015 (14)	0.0206 (16)	−0.0054 (15)
C4	0.0515 (19)	0.055 (2)	0.0638 (19)	−0.0042 (16)	0.0265 (17)	−0.0215 (16)
C5	0.058 (2)	0.059 (2)	0.0426 (15)	−0.0010 (17)	0.0173 (15)	−0.0135 (14)
C6	0.0444 (16)	0.0398 (17)	0.0359 (13)	−0.0059 (12)	0.0155 (12)	−0.0038 (11)
C7	0.0386 (15)	0.0365 (15)	0.0295 (12)	−0.0064 (12)	0.0106 (11)	0.0014 (10)
C8	0.0355 (14)	0.0323 (15)	0.0386 (13)	−0.0028 (12)	0.0123 (11)	0.0020 (11)
C9	0.0497 (17)	0.0418 (18)	0.0381 (14)	−0.0027 (14)	0.0089 (13)	0.0052 (12)
C10	0.0509 (19)	0.0452 (19)	0.0512 (17)	0.0056 (15)	0.0129 (15)	0.0152 (14)
C11	0.0478 (18)	0.0363 (17)	0.0629 (18)	0.0025 (14)	0.0245 (16)	0.0070 (14)
C12	0.0423 (16)	0.0353 (16)	0.0492 (15)	−0.0056 (12)	0.0214 (14)	−0.0032 (12)
C13	0.0330 (14)	0.0363 (16)	0.0382 (13)	−0.0067 (12)	0.0100 (11)	0.0022 (11)
C14	0.069 (2)	0.044 (2)	0.075 (2)	−0.0003 (17)	0.0338 (19)	−0.0128 (17)
Cl1	0.0494 (4)	0.0493 (4)	0.0333 (3)	−0.0014 (4)	0.0086 (3)	−0.0021 (3)
N1	0.0388 (12)	0.0355 (13)	0.0277 (10)	−0.0017 (10)	0.0094 (10)	−0.0003 (9)

N2	0.0529 (15)	0.0443 (15)	0.0278 (10)	0.0031 (12)	0.0075 (11)	0.0012 (9)

Geometric parameters (Å, º)

O1—C13	1.353 (3)	C6—N2	1.376 (4)
O1—H1	0.816 (10)	C7—N2	1.337 (3)
O2—C12	1.365 (3)	C7—N1	1.346 (3)
O2—C14	1.428 (4)	C7—C8	1.447 (4)
O3—Cl1	1.420 (3)	C8—C13	1.398 (4)
O4—Cl1	1.419 (3)	C8—C9	1.408 (4)
O5—Cl1	1.410 (2)	C9—C10	1.369 (4)
O6—Cl1	1.400 (3)	C9—H9	0.9300
C1—C2	1.384 (4)	C10—C11	1.389 (4)
C1—N1	1.389 (3)	C10—H10	0.9300
C1—C6	1.396 (4)	C11—C12	1.375 (4)
C2—C3	1.378 (4)	C11—H11	0.9300
C2—H2	0.9300	C12—C13	1.400 (4)
C3—C4	1.400 (4)	C14—H14A	0.9600
C3—H3	0.9300	C14—H14B	0.9600
C4—C5	1.374 (5)	C14—H14C	0.9600
C4—H4	0.9300	N1—H101	0.897 (10)
C5—C6	1.389 (4)	N2—H102	0.885 (10)
C5—H5	0.9300		
C13—O1—H1	111 (3)	C9—C10—H10	119.5
C12—O2—C14	118.0 (3)	C11—C10—H10	119.5
C2—C1—N1	132.2 (3)	C12—C11—C10	119.5 (3)
C2—C1—C6	122.0 (3)	C12—C11—H11	120.2
N1—C1—C6	105.8 (2)	C10—C11—H11	120.2
C3—C2—C1	116.1 (3)	O2—C12—C11	126.5 (3)
C3—C2—H2	121.9	O2—C12—C13	113.1 (3)
C1—C2—H2	121.9	C11—C12—C13	120.5 (3)
C2—C3—C4	122.2 (3)	O1—C13—C8	119.0 (3)
C2—C3—H3	118.9	O1—C13—C12	121.1 (2)
C4—C3—H3	118.9	C8—C13—C12	119.9 (3)
C5—C4—C3	121.6 (3)	O2—C14—H14A	109.5
C5—C4—H4	119.2	O2—C14—H14B	109.5
C3—C4—H4	119.2	H14A—C14—H14B	109.5
C4—C5—C6	116.6 (3)	O2—C14—H14C	109.5
C4—C5—H5	121.7	H14A—C14—H14C	109.5
C6—C5—H5	121.7	H14B—C14—H14C	109.5
N2—C6—C5	132.1 (3)	O6—Cl1—O5	110.6 (2)
N2—C6—C1	106.4 (2)	O6—Cl1—O4	109.7 (2)
C5—C6—C1	121.5 (3)	O5—Cl1—O4	111.48 (18)
N2—C7—N1	107.4 (2)	O6—Cl1—O3	108.5 (2)
N2—C7—C8	125.1 (2)	O5—Cl1—O3	106.87 (18)
N1—C7—C8	127.5 (2)	O4—Cl1—O3	109.64 (19)
C13—C8—C9	118.8 (3)	C7—N1—C1	109.9 (2)
C13—C8—C7	121.2 (2)	C7—N1—H101	127 (2)
C9—C8—C7	120.0 (2)	C1—N1—H101	123 (2)

C10—C9—C8	120.2 (3)	C7—N2—C6	110.5 (2)
C10—C9—H9	119.9	C7—N2—H102	123 (2)
C8—C9—H9	119.9	C6—N2—H102	126 (2)
C9—C10—C11	121.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82 (1)	2.20 (3)	2.635 (3)	114 (3)
O1—H1···O4^{i}	0.82 (1)	2.13 (2)	2.869 (3)	151 (3)
N1—H101···O1	0.90 (1)	2.12 (3)	2.660 (3)	118 (3)
N1—H101···O5	0.90 (1)	2.04 (2)	2.819 (3)	144 (3)
N2—H102···O3^{ii}	0.89 (1)	2.06 (2)	2.857 (4)	149 (3)

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