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N-(4-Hydroxy-3-methoxybenzyl)-benzamide

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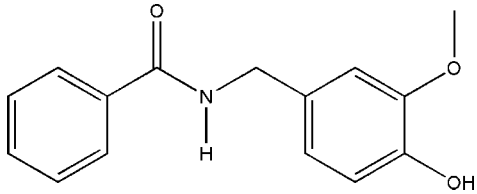
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.064; wR factor = 0.186; data-to-parameter ratio = 12.9.

In the molecular structure of the title compound, $C_{15}H_{15}NO_3$, the two benzene rings are twisted with respect to each other, making a dihedral angle of 75.11 (10)°. In the amide fragment, the C=O and C—N bond distances are 1.248 (3) and 1.321 (3) Å, respectively, indicating electron delocalization. A partially ovelapped arrangement between parallel hydroxy-methoxybenzene rings is observed in the crystal structure, and the face-to-face distance of 3.531 (16) Å suggests the existence of weak π–π stacking. N—H···O and O—H···O hydrogen bonding is also present in the crystal structure.

Related literature

The title compound was obtained during an investigation of capsaicin and its derivatives. For the biological activity of capsaicin, see: Kaga *et al.* (1989). For related structures, see: Luo & Huang (2004); Tong *et al.* (2008).



Experimental

Crystal data

$C_{15}H_{15}NO_3$ M_r = 257.28
Monoclinic, $P2_1/n$
a = 7.2292 (18) Å
b = 21.057 (5) Å
c = 9.031 (2) Å
β = 106.849 (12)°
V = 1315.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 K
0.40 × 0.28 × 0.26 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: none
8839 measured reflections
2353 independent reflections
1304 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.186
S = 1.00
2353 reflections
182 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O3^{i}	0.85 (3)	2.42 (3)	3.145 (6)	143 (2)
O3—H3*A*···O1^{ii}	0.98 (4)	1.80 (4)	2.745 (5)	160 (5)

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2556).

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supporting information

Acta Cryst. (2009). E**65**, o1899 [doi:10.1107/S1600536809027500]

N-(4-Hydroxy-3-methoxybenzyl)benzamide

Liang-You Xia, Wen-Long Wang, Shan-Heng Wang, Yan-Lan Huang and Shang Shan

S1. Comment

Capsaicin, a pungent principle of capsicums, has been known to exhibit a variety of biological activities, including recent findings concerning its mutagenicity (Kaga *et al.* 1989). During the investigation on syntheses of capsaicin and its derivatives, the title compound has recently been obtained and its crystal structure is reported here.

In the molecular structure of the title compound (Fig. 1), two benzene rings are twisted to each other with a dihedral angle of 75.11 (10)°, which is similar to 72.1 (2)° found in *N*-2-chlorobenzylbenzamide (Luo & Huang, 2004) but is somewhat larger than 56.32 (17)° found in *N*-(4-Cyanobenzyl)benzamide (Tong *et al.* 2008). The amide flagment is nearly coplanar with the C1-benzene ring [dihedral angle 5.0 (4)°]. The C7═O1 and C7—N1 bond distances are 1.248 (3) and 1.321 (3) Å, respectively, showing the electron delocalization in the amide fragment.

A partially ovelapped arrangement between parallel C9-benzene and C9^{i}-benzene rings is observed in the crystal structure (Fig. 2), the face-to-face distance of 3.531 (16) Å suggests the existence of weak π-π stacking between them [symmetry code: (i) 1 - *x*,1 - *y*,-*z*]. The N—H···O and O—H···O hydrogen bonding is present in the crystal structure (Table 1 and Fig. 2), which helps to stabilize the crystal structure.

S2. Experimental

4-Hydroxy-3-methoxy benzylamine HCl salt (4.7 g, 25 mmol) and dimethylformamide (25 ml) were added to a 100 ml 3-necked flask equipped with an additional funnel, a thermometer and a magnetic stirrer. Water solution (10 ml) of NaOH (2.0 g) was added at room temperature. The mixture was stirred at 308 K for 30 min and then cooled to 273 K. An ether solution (10 ml) of benzoyl chloride (3.5 g, 25 mmol) was added dropwise at about 273 K over 20 min. After stirred for 2 h at room temperature the mixture was poured into water, and then extracted with ethyl acetate. The ethyl acetate extract was washed with 1 *M* HCl followed by saturated $NaHCO_3$ and brine. The extract was then dried over anhydrous Na_2SO_4 and filtered. Solvents were removed under vacuum at about 308 K to give a solid crude. Recrystallization was performed twice with an absolute ethyl acetate to obtain single crystals of the title compound.

S3. Refinement

Hydroxy and imino H atoms were located in a difference Fourier map and were refined isotropically. Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and torsion angle was refined to fit the electron density, U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic) and 0.97 Å (methylene), and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C).

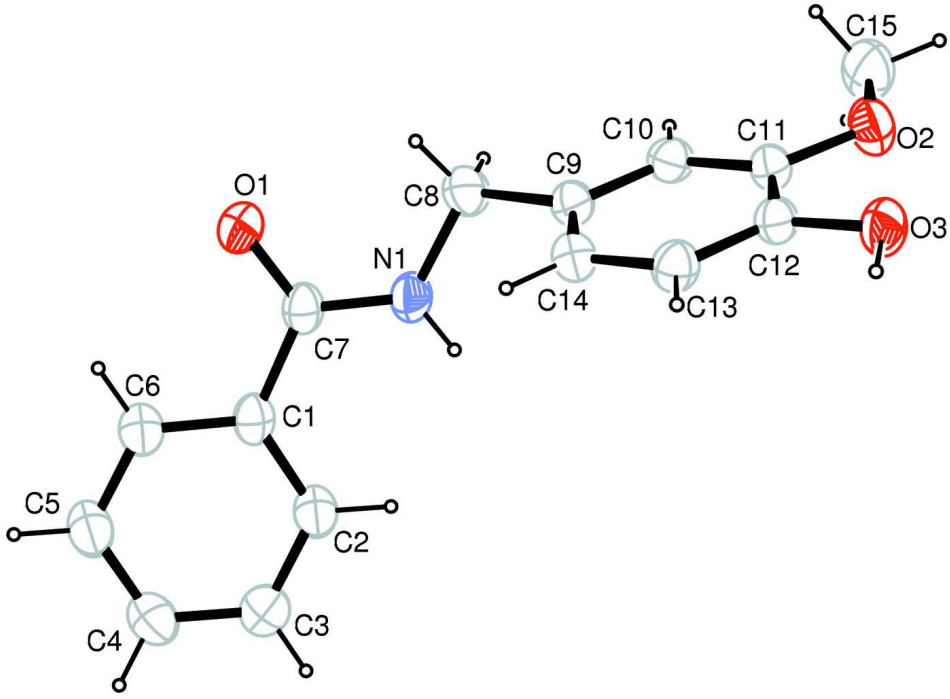

Figure 1

The molecular structure of the title compound with 40% probability displacement (arbitrary spheres for H atoms).

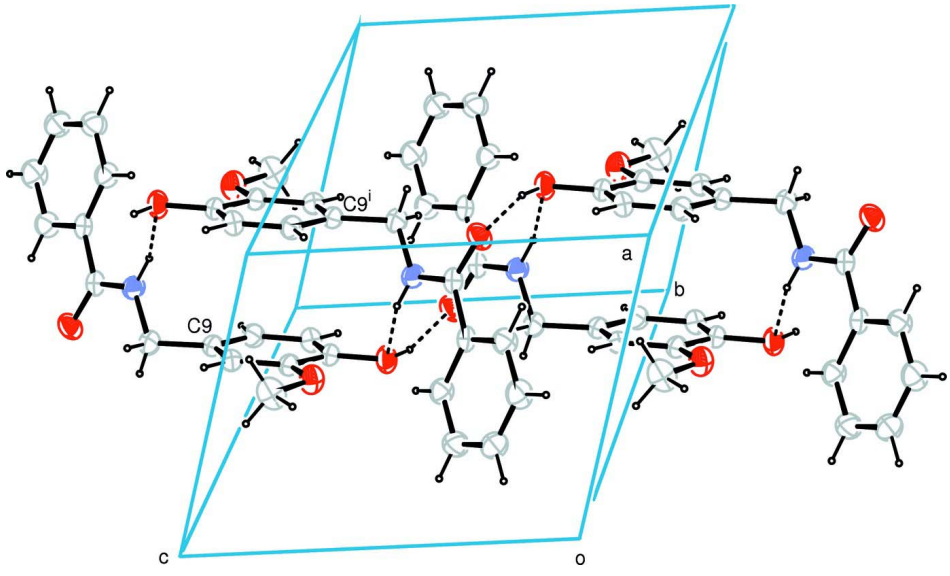

Figure 2

The unit cell packing diagram showing π-π stacking between C9-benzene rings [symmetry code: (i) 1 - *x*, 1 - *y*, -*z*]. Dashed lines indicate the hydrogen bonding.

N-(4-Hydroxy-3-methoxybenzyl)benzamide

Crystal data

$C_{15}H_{15}NO_3$
$M_r = 257.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 7.2292$ (18) Å
$b = 21.057$ (5) Å
$c = 9.031$ (2) Å
$\beta = 106.849$ (12)°
$V = 1315.7$ (5) Å^3
$Z = 4$
$F(000) = 544$
$D_x = 1.299$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4326 reflections
$\theta = 2.2$–$25.1°$
$\mu = 0.09$ mm^{-1}
$T = 294$ K
Prism, colorless
0.40 × 0.28 × 0.26 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
ω scans
8839 measured reflections
2353 independent reflections
1304 reflections with $I > 2\sigma(I)$
$R_{int} = 0.060$
$\theta_{max} = 25.2°$, $\theta_{min} = 1.9°$
$h = -8 \rightarrow 8$
$k = -25 \rightarrow 25$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.064$
$wR(F^2) = 0.186$
$S = 1.00$
2353 reflections
182 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.024 (4)

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}
N1	0.4418 (4)	0.57597 (12)	0.3750 (3)	0.0609 (8)
O1	0.2826 (3)	0.61857 (10)	0.5293 (2)	0.0704 (7)
O2	0.2344 (3)	0.40542 (9)	−0.1741 (2)	0.0699 (7)
O3	0.2085 (3)	0.50638 (11)	−0.3439 (2)	0.0681 (7)

C1	0.5991 (4)	0.65751 (12)	0.5602 (3)	0.0501 (7)
C2	0.7756 (4)	0.65520 (14)	0.5270 (3)	0.0624 (8)
H2	0.7941	0.6257	0.4560	0.075*
C3	0.9237 (5)	0.69650 (15)	0.5988 (4)	0.0730 (10)
H3	1.0410	0.6947	0.5760	0.088*
C4	0.8970 (5)	0.74018 (16)	0.7039 (4)	0.0774 (10)
H4	0.9962	0.7680	0.7517	0.093*
C5	0.7239 (5)	0.74283 (16)	0.7384 (4)	0.0789 (10)
H5	0.7063	0.7722	0.8101	0.095*
C6	0.5766 (5)	0.70172 (13)	0.6662 (4)	0.0656 (9)
H6	0.4597	0.7039	0.6895	0.079*
C7	0.4317 (4)	0.61524 (13)	0.4864 (3)	0.0526 (8)
C8	0.2814 (5)	0.53450 (15)	0.2939 (3)	0.0640 (9)
H8A	0.1614	0.5516	0.3050	0.077*
H8B	0.3004	0.4927	0.3413	0.077*
C9	0.2655 (4)	0.52835 (13)	0.1243 (3)	0.0512 (8)
C10	0.2606 (4)	0.46920 (13)	0.0575 (3)	0.0530 (8)
H10	0.2709	0.4331	0.1186	0.064*
C11	0.2408 (4)	0.46267 (13)	−0.0981 (3)	0.0508 (8)
C12	0.2250 (4)	0.51616 (14)	−0.1907 (3)	0.0511 (7)
C13	0.2304 (4)	0.57554 (13)	−0.1242 (3)	0.0569 (8)
H13	0.2204	0.6117	−0.1851	0.068*
C14	0.2505 (4)	0.58145 (14)	0.0317 (3)	0.0576 (8)
H14	0.2540	0.6217	0.0750	0.069*
C15	0.2403 (6)	0.34901 (15)	−0.0863 (4)	0.0872 (11)
H15A	0.3633	0.3459	−0.0092	0.131*
H15B	0.2221	0.3128	−0.1537	0.131*
H15C	0.1393	0.3502	−0.0369	0.131*
H1N	0.546 (4)	0.5707 (13)	0.350 (3)	0.063 (10)*
H3A	0.204 (7)	0.548 (2)	−0.393 (5)	0.148 (18)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0560 (17)	0.0776 (18)	0.0497 (16)	−0.0013 (14)	0.0162 (14)	−0.0173 (13)
O1	0.0796 (16)	0.0755 (15)	0.0682 (15)	−0.0099 (12)	0.0404 (12)	−0.0126 (11)
O2	0.0938 (16)	0.0555 (13)	0.0618 (14)	−0.0044 (11)	0.0247 (12)	−0.0115 (11)
O3	0.0854 (16)	0.0777 (16)	0.0449 (13)	−0.0053 (12)	0.0247 (11)	−0.0056 (11)
C1	0.0671 (19)	0.0473 (16)	0.0358 (15)	0.0076 (14)	0.0149 (14)	0.0045 (13)
C2	0.068 (2)	0.0613 (19)	0.0585 (19)	0.0032 (16)	0.0192 (16)	−0.0100 (15)
C3	0.069 (2)	0.079 (2)	0.074 (2)	−0.0035 (18)	0.0259 (18)	−0.0077 (18)
C4	0.082 (3)	0.076 (2)	0.072 (2)	−0.0169 (19)	0.0189 (19)	−0.0176 (19)
C5	0.099 (3)	0.068 (2)	0.075 (2)	−0.010 (2)	0.034 (2)	−0.0211 (18)
C6	0.073 (2)	0.0619 (19)	0.065 (2)	−0.0006 (16)	0.0253 (17)	−0.0104 (16)
C7	0.068 (2)	0.0524 (17)	0.0390 (16)	0.0050 (15)	0.0181 (15)	0.0010 (13)
C8	0.069 (2)	0.076 (2)	0.0461 (18)	−0.0121 (16)	0.0144 (15)	−0.0081 (16)
C9	0.0497 (17)	0.0587 (18)	0.0450 (17)	−0.0037 (13)	0.0134 (14)	−0.0059 (14)
C10	0.0529 (18)	0.0561 (18)	0.0498 (18)	−0.0038 (13)	0.0144 (14)	0.0005 (14)

C11	0.0516 (18)	0.0556 (17)	0.0453 (17)	−0.0030 (13)	0.0141 (13)	−0.0088 (14)
C12	0.0521 (18)	0.0618 (18)	0.0391 (17)	−0.0039 (14)	0.0129 (13)	−0.0042 (14)
C13	0.065 (2)	0.0556 (18)	0.0521 (19)	0.0024 (14)	0.0206 (15)	0.0029 (15)
C14	0.0630 (19)	0.0570 (18)	0.0545 (19)	−0.0045 (14)	0.0198 (15)	−0.0098 (15)
C15	0.118 (3)	0.056 (2)	0.099 (3)	−0.004 (2)	0.050 (2)	−0.0084 (19)

Geometric parameters (Å, º)

N1—C7	1.321 (3)	C5—H5	0.9300
N1—C8	1.467 (4)	C6—H6	0.9300
N1—H1N	0.85 (3)	C8—C9	1.508 (4)
O1—C7	1.248 (3)	C8—H8A	0.9700
O2—C11	1.382 (3)	C8—H8B	0.9700
O2—C15	1.422 (4)	C9—C10	1.380 (4)
O3—C12	1.370 (3)	C9—C14	1.382 (4)
O3—H3A	0.98 (4)	C10—C11	1.377 (4)
C1—C6	1.378 (4)	C10—H10	0.9300
C1—C2	1.393 (4)	C11—C12	1.388 (4)
C1—C7	1.494 (4)	C12—C13	1.382 (4)
C2—C3	1.386 (4)	C13—C14	1.379 (4)
C2—H2	0.9300	C13—H13	0.9300
C3—C4	1.374 (4)	C14—H14	0.9300
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.375 (4)	C15—H15B	0.9600
C4—H4	0.9300	C15—H15C	0.9600
C5—C6	1.381 (4)		
C7—N1—C8	122.9 (3)	C9—C8—H8A	109.2
C7—N1—H1N	122 (2)	N1—C8—H8B	109.2
C8—N1—H1N	114.9 (19)	C9—C8—H8B	109.2
C11—O2—C15	117.4 (2)	H8A—C8—H8B	107.9
C12—O3—H3A	108 (3)	C10—C9—C14	118.6 (3)
C6—C1—C2	118.2 (3)	C10—C9—C8	120.4 (3)
C6—C1—C7	117.9 (3)	C14—C9—C8	121.0 (3)
C2—C1—C7	123.9 (2)	C11—C10—C9	121.2 (3)
C3—C2—C1	120.6 (3)	C11—C10—H10	119.4
C3—C2—H2	119.7	C9—C10—H10	119.4
C1—C2—H2	119.7	C10—C11—O2	124.9 (3)
C4—C3—C2	119.9 (3)	C10—C11—C12	120.0 (3)
C4—C3—H3	120.0	O2—C11—C12	115.1 (2)
C2—C3—H3	120.0	O3—C12—C13	123.9 (3)
C3—C4—C5	120.2 (3)	O3—C12—C11	117.0 (3)
C3—C4—H4	119.9	C13—C12—C11	119.1 (3)
C5—C4—H4	119.9	C14—C13—C12	120.4 (3)
C4—C5—C6	119.7 (3)	C14—C13—H13	119.8
C4—C5—H5	120.2	C12—C13—H13	119.8
C6—C5—H5	120.2	C13—C14—C9	120.8 (3)
C1—C6—C5	121.4 (3)	C13—C14—H14	119.6

C1—C6—H6	119.3	C9—C14—H14	119.6
C5—C6—H6	119.3	O2—C15—H15A	109.5
O1—C7—N1	121.0 (3)	O2—C15—H15B	109.5
O1—C7—C1	119.3 (2)	H15A—C15—H15B	109.5
N1—C7—C1	119.7 (3)	O2—C15—H15C	109.5
N1—C8—C9	112.0 (2)	H15A—C15—H15C	109.5
N1—C8—H8A	109.2	H15B—C15—H15C	109.5

C6—C1—C2—C3	−0.1 (4)	N1—C8—C9—C14	−54.6 (4)
C7—C1—C2—C3	179.2 (3)	C14—C9—C10—C11	−0.1 (4)
C1—C2—C3—C4	0.0 (5)	C8—C9—C10—C11	178.2 (2)
C2—C3—C4—C5	0.3 (5)	C9—C10—C11—O2	−179.9 (3)
C3—C4—C5—C6	−0.5 (5)	C9—C10—C11—C12	−0.1 (4)
C2—C1—C6—C5	−0.1 (4)	C15—O2—C11—C10	2.9 (4)
C7—C1—C6—C5	−179.5 (3)	C15—O2—C11—C12	−176.9 (3)
C4—C5—C6—C1	0.4 (5)	C10—C11—C12—O3	179.1 (2)
C8—N1—C7—O1	0.5 (4)	O2—C11—C12—O3	−1.1 (4)
C8—N1—C7—C1	−178.1 (2)	C10—C11—C12—C13	0.3 (4)
C6—C1—C7—O1	−4.3 (4)	O2—C11—C12—C13	−179.9 (2)
C2—C1—C7—O1	176.4 (3)	O3—C12—C13—C14	−178.9 (3)
C6—C1—C7—N1	174.4 (2)	C11—C12—C13—C14	−0.2 (4)
C2—C1—C7—N1	−5.0 (4)	C12—C13—C14—C9	0.0 (5)
C7—N1—C8—C9	142.7 (3)	C10—C9—C14—C13	0.2 (4)
N1—C8—C9—C10	127.1 (3)	C8—C9—C14—C13	−178.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O3^{i}	0.85 (3)	2.42 (3)	3.145 (6)	143 (2)
O3—H3*A*···O1^{ii}	0.98 (4)	1.80 (4)	2.745 (5)	160 (5)

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