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(*E*)-4-Hydroxy-*N*′-(2-hydroxy-4-methoxybenzylidene)benzohydrazide

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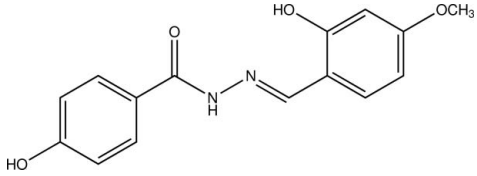
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.043; wR factor = 0.122; data-to-parameter ratio = 26.4.

In the title compound, $C_{15}H_{14}N_2O_4$, the dihedral angle between the benzene rings is 40.59 (4)° and an intramolecular O—H···N hydrogen bond generates an S(6) ring. In the crystal, N—H···O, O—H···O and C—H···O interactions link the molecules into a three-dimensional network.

Related literature

For a related sturucture and background to the properties and uses of hydrazones, see: Tameem *et al.* (2008).



Experimental

Crystal data

$C_{15}H_{14}N_2O_4$
M_r = 286.28
Monoclinic, $P2_1/c$
a = 15.1982 (2) Å
b = 8.2416 (1) Å
c = 10.7900 (1) Å
β = 101.173 (1)°
V = 1325.91 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.43 × 0.28 × 0.18 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.956, T_{max} = 0.982
21103 measured reflections
5357 independent reflections
4446 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.122
S = 1.05
5357 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···O3^{i}	0.859 (15)	2.342 (15)	3.0800 (10)	144.3 (14)
O3—H1*O*3···N2	0.866 (18)	1.851 (18)	2.6271 (10)	148.3 (17)
O2—H1*O*2···O1^{ii}	0.914 (18)	1.766 (18)	2.6713 (9)	170.3 (16)
C5—H5*A*···O2^{iii}	0.95	2.54	3.2036 (11)	128
C15—H15*A*···O1^{iv}	0.98	2.53	3.2646 (12)	132

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6854).

References

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Tameem, A. A., Saad, B., Salhin, A. M., Jebas, S. R. & Fun, H.-K. (2008). *Acta Cryst.* E**64**, o679–o680.

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S1. Comment

Continuing our interest on the synthesis and application of hydrazone and hydrazide derivatives (Tameem *et al.*, 2008), compound (I) (Fig. 1) was hereby synthesized based on the condensation reaction of 4-hydroxybenzhydrazide and 2-hydroxy-4-methoxybenzaldehyde.

All parameters in (I), are within normal ranges. The dihedral angle between C1—C6 and C9—C14 is 40.59 (4)°. In the molecule, intramolecular interaction of O3—H1O3···N2 form an S(6) hydrogen ring motif. In the crystal structure, the molecules are arranged into a three-dimensional network, connected by N1—H1N1···O3^{i}, O2—H1O2···O1^{ii}, C5—H5A···O2^{iii} and C15—H15A···O1^{iv} interactions (Table 1).

S2. Experimental

A solution of 2-hydroxy-4-methoxybenzaldehyde (152 mg, 1 mmol) in methanol (10 ml) was added dropwise to a methanolic solution (10 ml) of 4-hydroxybenzhydrazide (152 mg, 1 mmol) and the mixture was refluxed for 2 h. The resulting solution was condensed on a steam bath to 5 ml and cooled to room temperature. Yellow blocks were separated out, washed with cooled methanol and dried in air.

S3. Refinement

N and O bound H atoms were located from a difference Fourier map and freely refined. The remaining H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms. A rotating group model was applied to the methyl group.

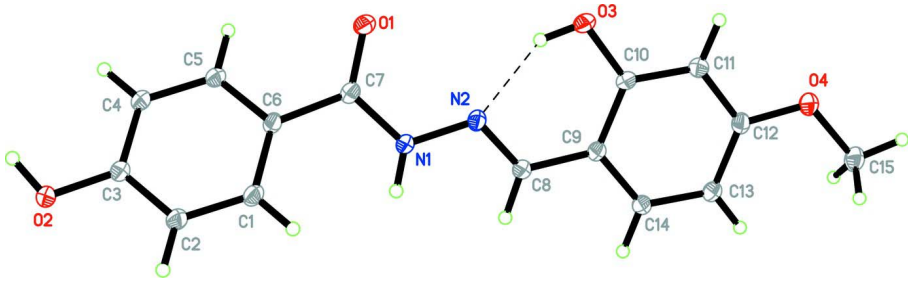

Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids. Hydrogen atoms are shown as spheres of arbitrary radius.

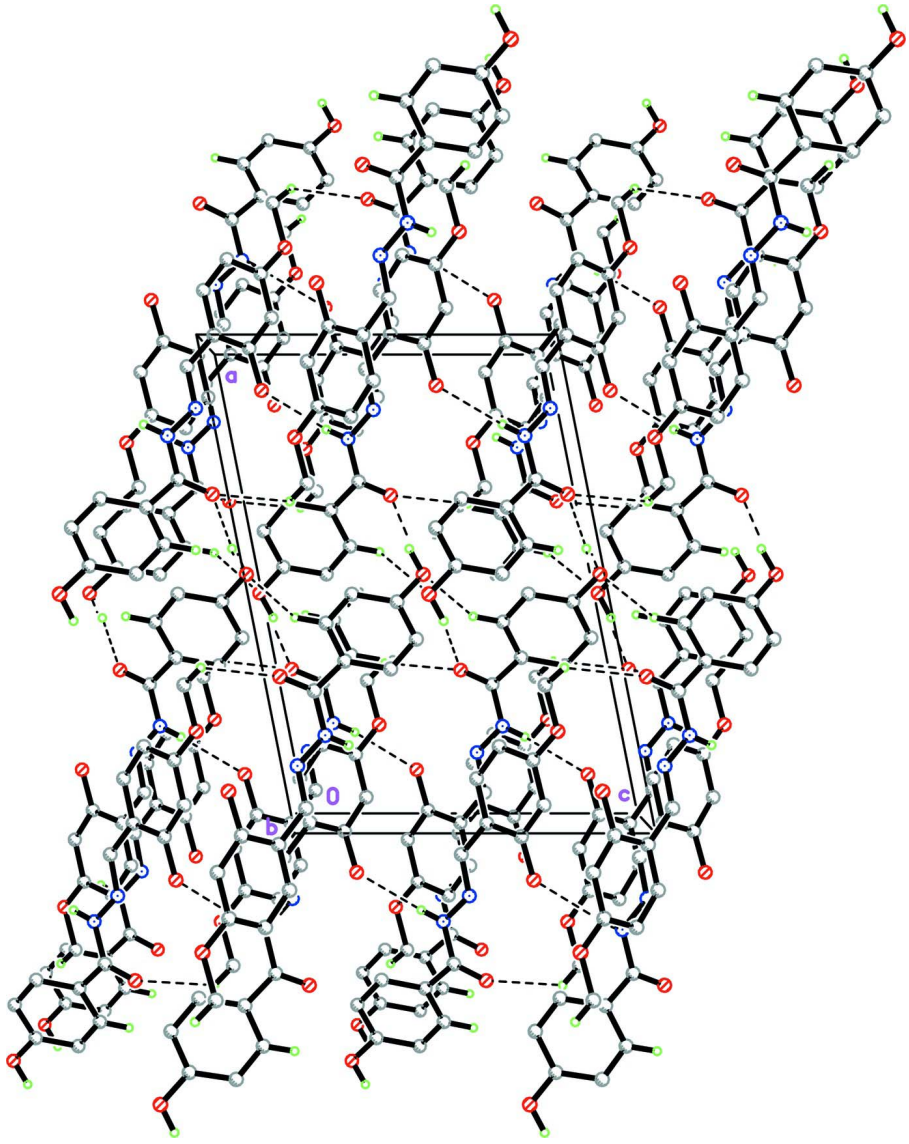

Figure 2

The crystal packing of (I). Dashed lines indicate hydrogen bonds. H atoms not involved in the hydrogen bond interactions have been omitted for clarity.

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$c = 10.7900$ (1) Å
$\beta = 101.173$ (1)°
$V = 1325.91$ (3) Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.434$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9753 reflections
$\theta = 2.7$–$35.0°$
$\mu = 0.11$ mm^{-1}
$T = 100$ K
Block, yellow
0.43 × 0.28 × 0.18 mm

Data collection

Bruker SMART APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.956, T_{max} = 0.982
21103 measured reflections
5357 independent reflections
4446 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 34.0°, θ_{min} = 2.7°
h = −23→22
k = −10→12
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.122
S = 1.05
5357 reflections
203 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
$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.4059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.31723 (4)	0.31882 (9)	0.55171 (6)	0.01663 (14)
O2	0.51966 (4)	0.76437 (9)	0.99737 (6)	0.01740 (14)
O3	0.09841 (4)	0.09665 (9)	0.36222 (6)	0.01737 (14)
O4	−0.20519 (4)	−0.02273 (9)	0.21628 (6)	0.01959 (14)
N1	0.19965 (5)	0.38654 (10)	0.64137 (7)	0.01474 (14)
N2	0.14157 (5)	0.29962 (10)	0.55201 (7)	0.01408 (14)
C1	0.32956 (6)	0.51526 (12)	0.85518 (8)	0.01642 (16)
H1A	0.2780	0.4682	0.8784	0.020*
C2	0.38846 (6)	0.60648 (12)	0.94252 (8)	0.01661 (17)
H2A	0.3774	0.6206	1.0255	0.020*
C3	0.46390 (6)	0.67757 (11)	0.90881 (8)	0.01411 (15)
C4	0.47939 (6)	0.65803 (11)	0.78597 (8)	0.01494 (16)
H4A	0.5297	0.7085	0.7618	0.018*
C5	0.42107 (6)	0.56481 (11)	0.69974 (8)	0.01445 (15)

H5A	0.4324	0.5500	0.6170	0.017*
C6	0.34575 (5)	0.49233 (11)	0.73292 (8)	0.01322 (15)
C7	0.28800 (5)	0.39174 (11)	0.63549 (8)	0.01329 (15)
C8	0.05729 (6)	0.31976 (11)	0.55158 (8)	0.01372 (15)
H8A	0.0393	0.3916	0.6109	0.016*
C9	−0.01035 (5)	0.23455 (10)	0.46203 (8)	0.01278 (15)
C10	0.01179 (6)	0.12335 (11)	0.37274 (8)	0.01310 (15)
C11	−0.05540 (6)	0.03854 (11)	0.29367 (8)	0.01457 (15)
H11A	−0.0402	−0.0385	0.2357	0.017*
C12	−0.14544 (6)	0.06598 (11)	0.29894 (8)	0.01454 (15)
C13	−0.16902 (6)	0.17757 (11)	0.38464 (8)	0.01555 (16)
H13A	−0.2302	0.1976	0.3872	0.019*
C14	−0.10109 (6)	0.25820 (11)	0.46569 (8)	0.01491 (16)
H14A	−0.1166	0.3319	0.5257	0.018*
C15	−0.29779 (6)	−0.01043 (14)	0.22528 (9)	0.02144 (19)
H15A	−0.3334	−0.0854	0.1651	0.032*
H15B	−0.3187	0.1008	0.2058	0.032*
H15C	−0.3046	−0.0383	0.3112	0.032*
H1N1	0.1785 (10)	0.4363 (19)	0.6991 (14)	0.029 (4)*
H1O3	0.1324 (12)	0.155 (2)	0.4189 (18)	0.047 (5)*
H1O2	0.5723 (12)	0.788 (2)	0.9722 (16)	0.039 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0124 (3)	0.0221 (3)	0.0158 (3)	0.0004 (2)	0.0038 (2)	−0.0021 (2)
O2	0.0132 (3)	0.0228 (3)	0.0166 (3)	−0.0040 (2)	0.0039 (2)	−0.0049 (2)
O3	0.0116 (3)	0.0226 (3)	0.0186 (3)	0.0009 (2)	0.0045 (2)	−0.0035 (2)
O4	0.0142 (3)	0.0253 (4)	0.0187 (3)	−0.0046 (2)	0.0017 (2)	−0.0060 (3)
N1	0.0103 (3)	0.0188 (4)	0.0152 (3)	−0.0015 (2)	0.0027 (2)	−0.0035 (3)
N2	0.0116 (3)	0.0161 (3)	0.0141 (3)	−0.0019 (2)	0.0016 (2)	−0.0008 (2)
C1	0.0126 (3)	0.0224 (4)	0.0149 (3)	−0.0037 (3)	0.0043 (3)	0.0006 (3)
C2	0.0143 (4)	0.0223 (4)	0.0139 (3)	−0.0030 (3)	0.0044 (3)	−0.0002 (3)
C3	0.0113 (3)	0.0161 (4)	0.0149 (3)	−0.0001 (3)	0.0025 (3)	−0.0004 (3)
C4	0.0121 (3)	0.0178 (4)	0.0157 (3)	−0.0022 (3)	0.0046 (3)	−0.0008 (3)
C5	0.0125 (3)	0.0176 (4)	0.0140 (3)	−0.0012 (3)	0.0043 (3)	−0.0002 (3)
C6	0.0105 (3)	0.0155 (4)	0.0135 (3)	−0.0010 (3)	0.0018 (2)	0.0008 (3)
C7	0.0115 (3)	0.0150 (4)	0.0132 (3)	−0.0004 (3)	0.0022 (2)	0.0022 (3)
C8	0.0119 (3)	0.0153 (4)	0.0139 (3)	−0.0003 (3)	0.0025 (2)	−0.0010 (3)
C9	0.0112 (3)	0.0136 (4)	0.0134 (3)	−0.0002 (3)	0.0022 (2)	0.0001 (3)
C10	0.0117 (3)	0.0151 (4)	0.0130 (3)	0.0009 (3)	0.0036 (2)	0.0011 (3)
C11	0.0151 (4)	0.0152 (4)	0.0138 (3)	−0.0010 (3)	0.0035 (3)	−0.0010 (3)
C12	0.0136 (3)	0.0160 (4)	0.0135 (3)	−0.0025 (3)	0.0015 (3)	0.0005 (3)
C13	0.0112 (3)	0.0179 (4)	0.0176 (4)	−0.0004 (3)	0.0028 (3)	−0.0017 (3)
C14	0.0122 (3)	0.0158 (4)	0.0168 (4)	0.0002 (3)	0.0030 (3)	−0.0026 (3)
C15	0.0145 (4)	0.0278 (5)	0.0210 (4)	−0.0049 (3)	0.0008 (3)	−0.0033 (4)

Geometric parameters (Å, º)

O1—C7	1.2375 (11)	C4—H4A	0.9500
O2—C3	1.3525 (10)	C5—C6	1.3979 (12)
O2—H1O2	0.915 (18)	C5—H5A	0.9500
O3—C10	1.3610 (10)	C6—C7	1.4849 (12)
O3—H1O3	0.866 (19)	C8—C9	1.4487 (11)
O4—C12	1.3568 (10)	C8—H8A	0.9500
O4—C15	1.4329 (12)	C9—C14	1.4009 (12)
N1—C7	1.3571 (11)	C9—C10	1.4166 (12)
N1—N2	1.3756 (10)	C10—C11	1.3862 (12)
N1—H1N1	0.858 (16)	C11—C12	1.3990 (12)
N2—C8	1.2907 (11)	C11—H11A	0.9500
C1—C2	1.3885 (12)	C12—C13	1.3992 (13)
C1—C6	1.4008 (12)	C13—C14	1.3865 (12)
C1—H1A	0.9500	C13—H13A	0.9500
C2—C3	1.3971 (12)	C14—H14A	0.9500
C2—H2A	0.9500	C15—H15A	0.9800
C3—C4	1.3997 (12)	C15—H15B	0.9800
C4—C5	1.3864 (12)	C15—H15C	0.9800
C3—O2—H1O2	111.6 (11)	N2—C8—C9	121.03 (8)
C10—O3—H1O3	107.8 (12)	N2—C8—H8A	119.5
C12—O4—C15	117.28 (7)	C9—C8—H8A	119.5
C7—N1—N2	119.35 (7)	C14—C9—C10	118.30 (8)
C7—N1—H1N1	122.1 (10)	C14—C9—C8	119.27 (8)
N2—N1—H1N1	118.5 (10)	C10—C9—C8	122.40 (8)
C8—N2—N1	115.92 (7)	O3—C10—C11	118.44 (8)
C2—C1—C6	120.29 (8)	O3—C10—C9	121.49 (8)
C2—C1—H1A	119.9	C11—C10—C9	120.07 (8)
C6—C1—H1A	119.9	C10—C11—C12	120.21 (8)
C1—C2—C3	120.27 (8)	C10—C11—H11A	119.9
C1—C2—H2A	119.9	C12—C11—H11A	119.9
C3—C2—H2A	119.9	O4—C12—C11	114.94 (8)
O2—C3—C2	118.12 (8)	O4—C12—C13	124.33 (8)
O2—C3—C4	122.18 (8)	C11—C12—C13	120.73 (8)
C2—C3—C4	119.70 (8)	C14—C13—C12	118.49 (8)
C5—C4—C3	119.76 (8)	C14—C13—H13A	120.8
C5—C4—H4A	120.1	C12—C13—H13A	120.8
C3—C4—H4A	120.1	C13—C14—C9	122.16 (8)
C4—C5—C6	120.91 (8)	C13—C14—H14A	118.9
C4—C5—H5A	119.5	C9—C14—H14A	118.9
C6—C5—H5A	119.5	O4—C15—H15A	109.5
C5—C6—C1	119.05 (8)	O4—C15—H15B	109.5
C5—C6—C7	117.28 (7)	H15A—C15—H15B	109.5
C1—C6—C7	123.67 (8)	O4—C15—H15C	109.5
O1—C7—N1	121.15 (8)	H15A—C15—H15C	109.5
O1—C7—C6	122.80 (8)	H15B—C15—H15C	109.5

N1—C7—C6	116.04 (7)		
C7—N1—N2—C8	−169.85 (8)	N2—C8—C9—C14	179.21 (8)
C6—C1—C2—C3	0.66 (14)	N2—C8—C9—C10	1.28 (13)
C1—C2—C3—O2	−179.55 (8)	C14—C9—C10—O3	178.72 (8)
C1—C2—C3—C4	0.73 (14)	C8—C9—C10—O3	−3.34 (13)
O2—C3—C4—C5	178.60 (8)	C14—C9—C10—C11	−1.39 (13)
C2—C3—C4—C5	−1.68 (14)	C8—C9—C10—C11	176.55 (8)
C3—C4—C5—C6	1.27 (13)	O3—C10—C11—C12	−178.13 (8)
C4—C5—C6—C1	0.11 (13)	C9—C10—C11—C12	1.98 (13)
C4—C5—C6—C7	−178.74 (8)	C15—O4—C12—C11	174.52 (8)
C2—C1—C6—C5	−1.08 (14)	C15—O4—C12—C13	−5.41 (13)
C2—C1—C6—C7	177.70 (9)	C10—C11—C12—O4	179.35 (8)
N2—N1—C7—O1	−0.48 (13)	C10—C11—C12—C13	−0.72 (13)
N2—N1—C7—C6	178.13 (7)	O4—C12—C13—C14	178.82 (8)
C5—C6—C7—O1	28.68 (13)	C11—C12—C13—C14	−1.10 (13)
C1—C6—C7—O1	−150.12 (9)	C12—C13—C14—C9	1.70 (14)
C5—C6—C7—N1	−149.91 (8)	C10—C9—C14—C13	−0.47 (13)
C1—C6—C7—N1	31.29 (12)	C8—C9—C14—C13	−178.48 (8)
N1—N2—C8—C9	−179.89 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*1···O3^{i}	0.859 (15)	2.342 (15)	3.0800 (10)	144.3 (14)
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O2—H1*O*2···O1^{ii}	0.914 (18)	1.766 (18)	2.6713 (9)	170.3 (16)
C5—H5*A*···O2^{iii}	0.95	2.54	3.2036 (11)	128
C15—H15*A*···O1^{iv}	0.98	2.53	3.2646 (12)	132

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*+1, *y*+1/2, −*z*+3/2; (iii) *x*, −*y*+3/2, *z*−1/2; (iv) −*x*, *y*−1/2, −*z*+1/2.