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Crystal structure of 3,4-dimethoxyphenol

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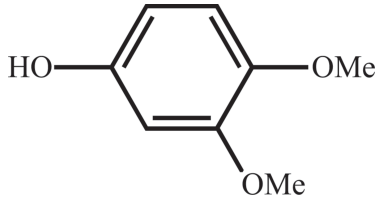
The title compound, $C_8H_{10}O_3$, has two planar molecules in the asymmetric unit possessing mean deviations from planarity of 0.051 and 0.071 Å. In the crystal, there are two distinct infinite chains, both along [010]. The chains are formed by O—H···O interactions between the phenol and both the 3-methoxy and the 4-methoxy groups.

Keywords: crystal structure; hydrogen bonding; phenols.

CCDC reference: 1439495

1. Related literature

For the crystal structure of the related 4-[(2,3-dimethylbut-3-en-2-yl)oxy]-3-methoxyphenol, see: Yamamoto *et al.* (2014). For the crystal structure of 3,4,5-trimethoxyphenol, see: Jia *et al.* (2012). For background and crystal structures solved during the study, see: McDonald *et al.* (2015); Nguyen *et al.* (2015).



2. Experimental

2.1. Crystal data

$C_8H_{10}O_3$
M_r = 154.16
Orthorhombic, *Pbca*
a = 8.7477 (4) Å
b = 13.8218 (7) Å
c = 26.6422 (13) Å
V = 3221.3 (3) Å^3
Z = 16
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 K
0.5 × 0.4 × 0.4 mm

2.2. Data collection

Bruker Venture D8 CMOS diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.700, T_{max} = 0.746
29914 measured reflections
3996 independent reflections
3360 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 1.04
3996 reflections
205 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.86 (1)	2.25 (1)	2.9663 (12)	141 (2)
O1—H1···O3^{i}	0.86 (1)	2.13 (1)	2.8834 (13)	145 (2)
O4—H4···O5^{i}	0.86 (1)	2.15 (2)	2.8384 (13)	137 (2)
O4—H4···O6^{i}	0.86 (1)	2.37 (1)	3.1107 (14)	145 (2)

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015) and *olex2.refine* (Bourhis *et al.*, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* and *publCIF* (Westrip, 2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: FF2145).

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supporting information

Acta Cryst. (2015). E**71**, o1019 [https://doi.org/10.1107/S2056989015022860]

Crystal structure of 3,4-dimethoxyphenol

Heather A. Mills-Robles, Vasumathi Desikan, James A. Golen and David R. Manke

S1. Comment

In a continuing collaborative study of the solid state structure of aromatic alcohols between UMass Dartmouth and Massasoit Community College (McDonald *et al.*, 2015; Nguyen *et al.*, 2015), we report herein the structure of 3,4-dimethoxyphenol. A similar 3,4-dialkoxyphenol complex has been structurally characterized (Yamamoto *et al.*, 2014) and demonstrates tip-to-tail hydrogen bonding with the 4-alkoxy group. The structure of the trisubstituted 3,4,5-trimethoxyphenol demonstrates a similar interaction, with just the 4-methoxy group involved in hydrogen bonding (Jia *et al.*, 2012). In contrast, the title compound exhibits hydrogen bonding chains with interactions involving the methoxy groups at both the 3 and 4 positions.

The molecular structure of the title compound is shown in Figure 1 .There are two molecules in the asymmetric unit, with non-hydrogen atoms possessing mean deviations from the plane of 0.051 Å and 0.071 Å. There are two distinct hydrogen bonding chains which both propagate along [010]. One is formed by O1–H1···O2 and O1–H1···O3 interactions, and the other by O4–H4···O5 and O4–H4···O6 interactions. The packing of the title compound indicating hydrogen bonding is shown in Figure 2.

S2. Experimental

A commercial sample (Aldrich) was used for crystallization. Single crystals suitable for X-ray diffraction studies were grown by slow evaporation of a methylene chloride solution.

S3. Refinement

All non-hydrogen atoms were refined anisotropically (Olex2) by full matrix least squares on F^2. Hydrogen atoms H1 and H4 were found from a Fourier difference map, and refined with a fixed distance of 0.86 (0.005) Å and isotropic displacement parameters of 1.50 times U_{eq} of the parent O atoms. The remaining hydrogen atoms were placed in calculated positions and then refined with a riding model with C–H lengths of 0.95 Å (sp^2) and 0.98 Å (sp^3) with isotropic displacement parameters set to 1.20 (sp^2) and 1.50 (sp^3) times U_{eq} of the parent C atom.

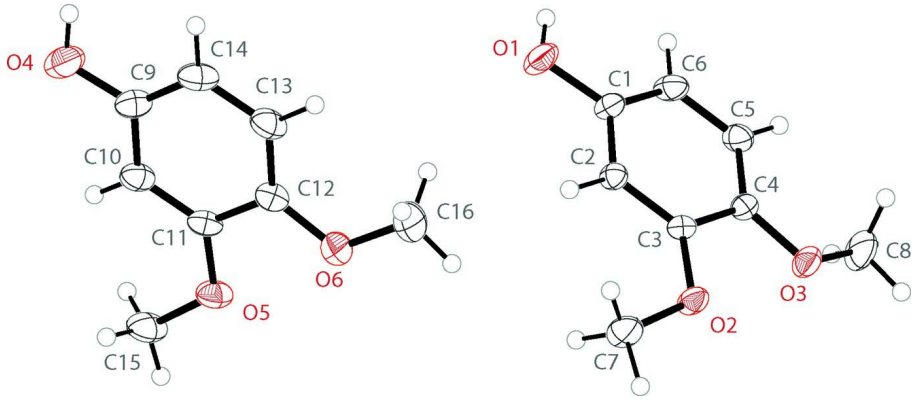

Figure 1

Molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius.

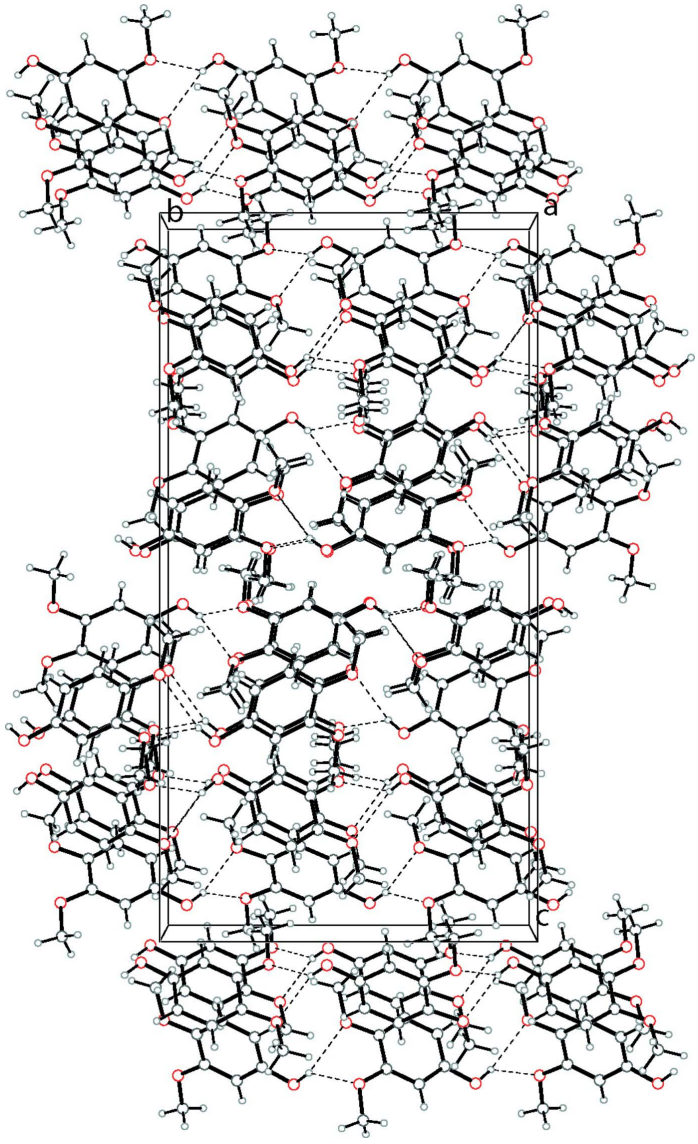

Figure 2

Molecular packing of the title compound with hydrogen bonding shown as dashed lines.

3,4-Dimethoxyphenol

Crystal data

$C_8H_{10}O_3$
M_r = 154.16
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
a = 8.7477 (4) Å
b = 13.8218 (7) Å
c = 26.6422 (13) Å
V = 3221.3 (3) Å^3
Z = 16

$F(000)$ = 1312
D_x = 1.271 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9890 reflections
θ = 3.0–28.3°
μ = 0.10 mm^{-1}
T = 120 K
Block, brown
0.5 × 0.4 × 0.4 mm

Data collection

Bruker Venture D8 CMOS diffractometer
Radiation source: Mo
TRIUMPH monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.700, T_{max} = 0.746

29914 measured reflections
3996 independent reflections
3360 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 28.4°, θ_{min} = 2.9°
h = −11→10
k = −18→18
l = −35→34

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 1.04
3996 reflections
205 parameters
2 restraints

Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 1.1413P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Special details

Experimental. Absorption correction: SADABS-2014/4 (Bruker,2014/4) was used for absorption correction. wR2(int) was 0.0791 before and 0.0531 after correction. The Ratio of minimum to maximum transmission is 0.9391. The $\lambda/2$ correction factor is 0.00150.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.31642 (12)	0.55792 (6)	0.46341 (4)	0.0364 (2)
H1	0.2656 (18)	0.6010 (10)	0.4472 (6)	0.055*
O2	0.41171 (10)	0.21648 (5)	0.45901 (3)	0.0274 (2)
O3	0.23732 (11)	0.19341 (6)	0.38304 (3)	0.0322 (2)
C1	0.29128 (13)	0.46989 (8)	0.44100 (4)	0.0244 (2)
C2	0.36772 (12)	0.39078 (7)	0.46188 (4)	0.0215 (2)
H2	0.4337	0.3994	0.4899	0.026*
C3	0.34642 (12)	0.29982 (7)	0.44142 (4)	0.0198 (2)
C4	0.25063 (13)	0.28729 (8)	0.39961 (4)	0.0224 (2)
C5	0.17669 (14)	0.36617 (8)	0.37932 (4)	0.0267 (2)
H5	0.1123	0.3579	0.3509	0.032*
C6	0.19567 (14)	0.45826 (8)	0.40017 (5)	0.0280 (3)
H6	0.1433	0.5123	0.3864	0.034*
C7	0.49570 (17)	0.22270 (9)	0.50473 (5)	0.0374 (3)
H7A	0.5365	0.1588	0.5133	0.056*
H7B	0.5803	0.2685	0.5006	0.056*
H7C	0.4280	0.2451	0.5317	0.056*
C8	0.1208 (2)	0.17531 (11)	0.34658 (6)	0.0538 (5)
H8A	0.1214	0.1066	0.3375	0.081*

H8B	0.0209	0.1924	0.3607	0.081*
H8C	0.1402	0.2145	0.3166	0.081*
O4	0.27270 (14)	0.84893 (7)	0.72098 (4)	0.0463 (3)
H4	0.319 (2)	0.8911 (12)	0.7029 (7)	0.069*
O5	0.06921 (10)	0.52789 (6)	0.71498 (3)	0.0313 (2)
O6	0.20065 (12)	0.49211 (7)	0.63132 (3)	0.0370 (2)
C9	0.26184 (15)	0.76288 (9)	0.69618 (5)	0.0313 (3)
C10	0.17021 (14)	0.69220 (9)	0.71881 (5)	0.0288 (3)
H10	0.1198	0.7055	0.7496	0.035*
C11	0.15354 (13)	0.60291 (8)	0.69608 (4)	0.0250 (2)
C12	0.22572 (14)	0.58329 (9)	0.65010 (4)	0.0278 (3)
C13	0.31638 (15)	0.65389 (10)	0.62848 (5)	0.0337 (3)
H13	0.3660	0.6411	0.5975	0.040*
C14	0.33589 (15)	0.74365 (10)	0.65161 (5)	0.0346 (3)
H14	0.3998	0.7912	0.6367	0.042*
C15	0.01260 (18)	0.53863 (10)	0.76497 (5)	0.0403 (3)
H15A	−0.0456	0.4808	0.7744	0.060*
H15B	0.0986	0.5469	0.7881	0.060*
H15C	−0.0540	0.5955	0.7667	0.060*
C16	0.2624 (2)	0.47148 (13)	0.58306 (6)	0.0560 (5)
H16A	0.2374	0.4048	0.5737	0.084*
H16B	0.2187	0.5161	0.5583	0.084*
H16C	0.3737	0.4794	0.5839	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0473 (6)	0.0169 (4)	0.0450 (5)	0.0030 (4)	−0.0158 (4)	−0.0031 (4)
O2	0.0317 (4)	0.0177 (4)	0.0326 (4)	0.0028 (3)	−0.0104 (3)	0.0003 (3)
O3	0.0436 (5)	0.0208 (4)	0.0322 (4)	−0.0027 (4)	−0.0134 (4)	−0.0036 (3)
C1	0.0267 (5)	0.0170 (5)	0.0295 (6)	−0.0015 (4)	−0.0012 (4)	0.0002 (4)
C2	0.0210 (5)	0.0201 (5)	0.0234 (5)	−0.0016 (4)	−0.0024 (4)	0.0003 (4)
C3	0.0189 (5)	0.0179 (5)	0.0227 (5)	0.0003 (4)	0.0007 (4)	0.0030 (4)
C4	0.0248 (5)	0.0195 (5)	0.0229 (5)	−0.0038 (4)	−0.0008 (4)	0.0002 (4)
C5	0.0282 (6)	0.0260 (5)	0.0260 (5)	−0.0040 (5)	−0.0078 (4)	0.0042 (4)
C6	0.0292 (6)	0.0212 (5)	0.0337 (6)	0.0008 (4)	−0.0067 (5)	0.0070 (4)
C7	0.0450 (7)	0.0256 (6)	0.0416 (7)	0.0050 (5)	−0.0209 (6)	0.0027 (5)
C8	0.0796 (12)	0.0334 (7)	0.0484 (9)	−0.0055 (8)	−0.0363 (8)	−0.0082 (6)
O4	0.0618 (7)	0.0258 (5)	0.0513 (6)	−0.0033 (5)	0.0108 (5)	0.0018 (4)
O5	0.0317 (4)	0.0287 (4)	0.0335 (5)	−0.0021 (3)	0.0081 (4)	0.0079 (3)
O6	0.0491 (6)	0.0340 (5)	0.0279 (4)	−0.0052 (4)	0.0066 (4)	−0.0012 (4)
C9	0.0318 (6)	0.0236 (5)	0.0385 (6)	0.0032 (5)	0.0006 (5)	0.0063 (5)
C10	0.0289 (6)	0.0269 (6)	0.0306 (6)	0.0062 (5)	0.0052 (5)	0.0065 (4)
C11	0.0207 (5)	0.0261 (5)	0.0283 (6)	0.0021 (4)	0.0008 (4)	0.0097 (4)
C12	0.0277 (6)	0.0288 (6)	0.0269 (6)	0.0013 (5)	0.0000 (4)	0.0056 (5)
C13	0.0346 (7)	0.0366 (6)	0.0300 (6)	0.0012 (5)	0.0094 (5)	0.0081 (5)
C14	0.0313 (6)	0.0311 (6)	0.0413 (7)	−0.0012 (5)	0.0077 (5)	0.0124 (5)
C15	0.0448 (8)	0.0339 (6)	0.0421 (7)	0.0050 (6)	0.0214 (6)	0.0106 (6)

C16	0.0853 (13)	0.0480 (9)	0.0348 (7)	−0.0094 (9)	0.0195 (8)	−0.0073 (7)

Geometric parameters (Å, º)

O1—H1	0.859 (5)	O4—H4	0.860 (5)
O1—C1	1.3731 (13)	O4—C9	1.3638 (16)
O2—C3	1.3684 (12)	O5—C11	1.3686 (14)
O2—C7	1.4251 (14)	O5—C15	1.4285 (15)
O3—C4	1.3754 (13)	O6—C12	1.3735 (15)
O3—C8	1.4302 (16)	O6—C16	1.4236 (17)
C1—C2	1.3971 (15)	C9—C10	1.4002 (17)
C1—C6	1.3816 (16)	C9—C14	1.3786 (18)
C2—H2	0.9500	C10—H10	0.9500
C2—C3	1.3829 (14)	C10—C11	1.3824 (17)
C3—C4	1.4048 (15)	C11—C12	1.4045 (16)
C4—C5	1.3781 (16)	C12—C13	1.3831 (17)
C5—H5	0.9500	C13—H13	0.9500
C5—C6	1.3986 (16)	C13—C14	1.3957 (19)
C6—H6	0.9500	C14—H14	0.9500
C7—H7A	0.9800	C15—H15A	0.9800
C7—H7B	0.9800	C15—H15B	0.9800
C7—H7C	0.9800	C15—H15C	0.9800
C8—H8A	0.9800	C16—H16A	0.9800
C8—H8B	0.9800	C16—H16B	0.9800
C8—H8C	0.9800	C16—H16C	0.9800
C1—O1—H1	108.2 (13)	C9—O4—H4	110.7 (14)
C3—O2—C7	117.19 (9)	C11—O5—C15	116.82 (10)
C4—O3—C8	116.31 (10)	C12—O6—C16	116.88 (11)
O1—C1—C2	116.34 (10)	O4—C9—C10	116.09 (11)
O1—C1—C6	122.85 (10)	O4—C9—C14	123.55 (12)
C6—C1—C2	120.81 (10)	C14—C9—C10	120.35 (12)
C1—C2—H2	120.3	C9—C10—H10	120.2
C3—C2—C1	119.35 (10)	C11—C10—C9	119.65 (11)
C3—C2—H2	120.3	C11—C10—H10	120.2
O2—C3—C2	125.04 (9)	O5—C11—C10	124.89 (11)
O2—C3—C4	114.63 (9)	O5—C11—C12	114.65 (10)
C2—C3—C4	120.33 (9)	C10—C11—C12	120.47 (11)
O3—C4—C3	114.92 (9)	O6—C12—C11	115.02 (10)
O3—C4—C5	125.51 (10)	O6—C12—C13	125.96 (11)
C5—C4—C3	119.56 (10)	C13—C12—C11	119.00 (11)
C4—C5—H5	119.7	C12—C13—H13	119.6
C4—C5—C6	120.57 (10)	C12—C13—C14	120.89 (12)
C6—C5—H5	119.7	C14—C13—H13	119.6
C1—C6—C5	119.37 (10)	C9—C14—C13	119.62 (11)
C1—C6—H6	120.3	C9—C14—H14	120.2
C5—C6—H6	120.3	C13—C14—H14	120.2
O2—C7—H7A	109.5	O5—C15—H15A	109.5

O2—C7—H7B	109.5	O5—C15—H15B	109.5
O2—C7—H7C	109.5	O5—C15—H15C	109.5
H7A—C7—H7B	109.5	H15A—C15—H15B	109.5
H7A—C7—H7C	109.5	H15A—C15—H15C	109.5
H7B—C7—H7C	109.5	H15B—C15—H15C	109.5
O3—C8—H8A	109.5	O6—C16—H16A	109.5
O3—C8—H8B	109.5	O6—C16—H16B	109.5
O3—C8—H8C	109.5	O6—C16—H16C	109.5
H8A—C8—H8B	109.5	H16A—C16—H16B	109.5
H8A—C8—H8C	109.5	H16A—C16—H16C	109.5
H8B—C8—H8C	109.5	H16B—C16—H16C	109.5
O1—C1—C2—C3	179.40 (10)	O4—C9—C10—C11	−179.88 (11)
O1—C1—C6—C5	179.69 (11)	O4—C9—C14—C13	−179.08 (12)
O2—C3—C4—O3	0.15 (14)	O5—C11—C12—O6	−0.06 (15)
O2—C3—C4—C5	178.86 (10)	O5—C11—C12—C13	−178.59 (11)
O3—C4—C5—C6	178.24 (11)	O6—C12—C13—C14	−178.68 (12)
C1—C2—C3—O2	−178.48 (10)	C9—C10—C11—O5	178.92 (11)
C1—C2—C3—C4	0.94 (16)	C9—C10—C11—C12	−1.08 (17)
C2—C1—C6—C5	−0.61 (18)	C10—C9—C14—C13	1.42 (19)
C2—C3—C4—O3	−179.33 (10)	C10—C11—C12—O6	179.94 (11)
C2—C3—C4—C5	−0.62 (16)	C10—C11—C12—C13	1.41 (17)
C3—C4—C5—C6	−0.32 (18)	C11—C12—C13—C14	−0.32 (19)
C4—C5—C6—C1	0.93 (18)	C12—C13—C14—C9	−1.1 (2)
C6—C1—C2—C3	−0.32 (17)	C14—C9—C10—C11	−0.34 (19)
C7—O2—C3—C2	6.52 (16)	C15—O5—C11—C10	−9.16 (17)
C7—O2—C3—C4	−172.93 (11)	C15—O5—C11—C12	170.84 (11)
C8—O3—C4—C3	169.24 (12)	C16—O6—C12—C11	175.56 (13)
C8—O3—C4—C5	−9.38 (18)	C16—O6—C12—C13	−6.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.86 (1)	2.25 (1)	2.9663 (12)	141 (2)
O1—H1···O3^{i}	0.86 (1)	2.13 (1)	2.8834 (13)	145 (2)
O4—H4···O5^{i}	0.86 (1)	2.15 (2)	2.8384 (13)	137 (2)
O4—H4···O6^{i}	0.86 (1)	2.37 (1)	3.1107 (14)	145 (2)

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