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Redetermination of 3,5-dimethylphenol

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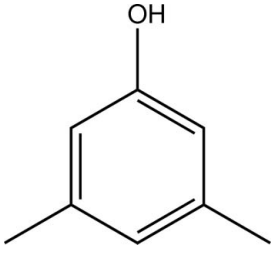
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.143; data-to-parameter ratio = 20.1.

The previous structure determination [Gillier-Pandraud *et al.* (1972). *C. R. Acad. Sci. Ser. C*, **275**, 1495] of the title compound, $C_8H_{10}O$, did not report atomic coordinates. There are two molecules in the asymmetric unit, *A* and *B*, which both show approximate non-crystallographic C_s symmetry. The intracyclic C—C—C angles cover the range 118.74 (12)–121.76 (13)°. In the crystal, molecules are linked by O—H···O hydrogen bonds, generating [001] $C_2^2(4)$ chains such that molecules *A* and *B* alternate. There is no aromatic π–π stacking in the crystal as the shortest centroid–centroid distance is greater than 4.74 Å.

Related literature

The compound has been deposited with the CSD (refcode: DMPHNL) but no three-dimensional-coordinates are available (Gillier-Pandraud *et al.*, 1972). For graph-set analysis of hydrogen bonds, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_{10}O$
M_r = 122.16
Monoclinic, $P2_1/c$
a = 11.9807 (6) Å
b = 13.8725 (7) Å
c = 8.5378 (4) Å
β = 90.000 (2)°
V = 1419.00 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 200 K
0.50 × 0.41 × 0.33 mm

Data collection

Bruker APEXII CCD diffractometer
12884 measured reflections
3392 independent reflections
2998 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 1.04
3392 reflections
169 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 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}	0.84	1.91	2.7463 (13)	171
O2—H2···O1^{ii}	0.84	1.90	2.7327 (13)	172

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

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

The authors thank Mrs Jenny Bell for helpful discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5838).

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supporting information

Acta Cryst. (2011). E**67**, o1152 [doi:10.1107/S1600536811013547]

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S1. Comment

Phenol and derivatives are interesting bonding partners for a variety of transition metals and elements from the *p*-block of the periodic system. They can act as neutral or – upon deprotonation – as anionic monodentate ligands. Upon variation of the substituents on the aromatic system, a seemingly endless series of symmetric as well as asymmetric phenol derivatives featuring different steric pretenses and acidities of the hydroxyl-group are available. At the beginning of a larger study aimed at elucidating the coordination behaviour of various phenol-derivatives in dependence of pH-value and substitution pattern on the phenyl moiety, it seemed of interest to determine the crystal structure of the title compound to enable comparisons with metric parameters in envisioned coordination compounds. Although the structure has been deposited with the Cambridge Structural Database (Gillier-Pandraud *et al.*, 1972), no three-dimensional-coordinates were provided.

The asymmetric unit comprises two molecules of the title compound which are nearly orientated perpendicular to each other. The least-squares planes defined by the C-atoms of the respective phenyl moieties intersect at an angle of 87.87 (4) °. Intracyclic C–C–C angles span a range of 119–122 ° with the biggest angles invariably found on the C-atoms bearing the hydroxyl group and the C-atoms in *para*-position to these, respectively. The H-atoms of both hydroxyl groups are approximately in plane with the aromatic systems (Fig. 1).

In the crystal structure, a set of cooperative hydrogen bonds connects the molecules to infinite chains along the crystallographic *c*-axis. Both molecules in the asymmetric unit participate alternately in these chains. In terms of graph-set analysis, the description of these intermolecular interactions necessitates a $C^2_2(4)$ descriptor on the binary level (Fig. 2). The closest distance between two centers of gravity was measured at 4.7437 (8) Å.

The packing of the title compound in the crystal structure is shown in Figure 3.

S2. Experimental

The compound was obtained commercially (Fluka). Crystals suitable for the X-ray diffraction study were taken directly from the provided compound.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.98 Å for the methyl groups and C—H 0.95 Å for aromatic carbon atoms) and were included in the refinement in the riding model approximation, with U(H) set to 1.5U_{eq}(C) for the methyl groups and 1.2U_{eq}(C) for aromatic carbon atoms. The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C—C bonds to best fit the experimental electron density (HFIX 137 in the *SHELX* program suite (Sheldrick, 2008)). The H atom of the hydroxyl groups were allowed to rotate with a fixed angle around the O—C bonds to best fit the experimental electron density (HFIX 147 in the *SHELX* program suite (Sheldrick, 2008)), their U(H) set to 1.5U_{eq}(O).

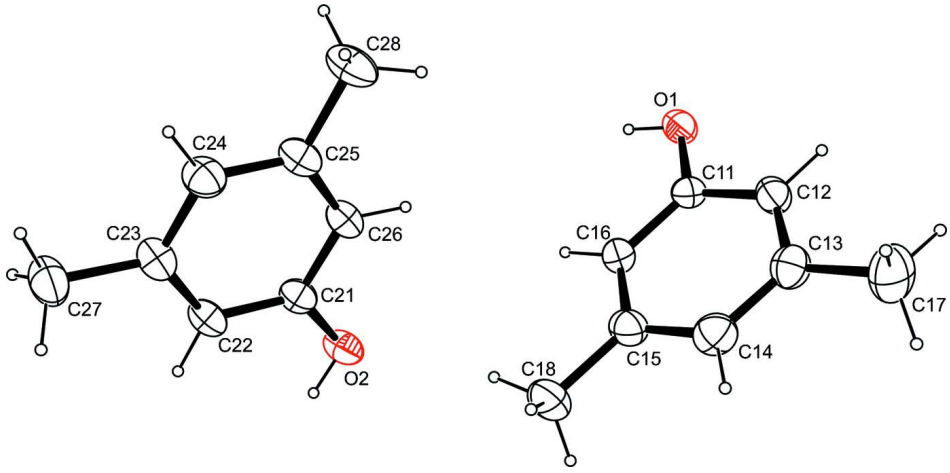

Figure 1

The molecular structure of the title compound, with anisotropic displacement ellipsoids drawn at 50% probability level.

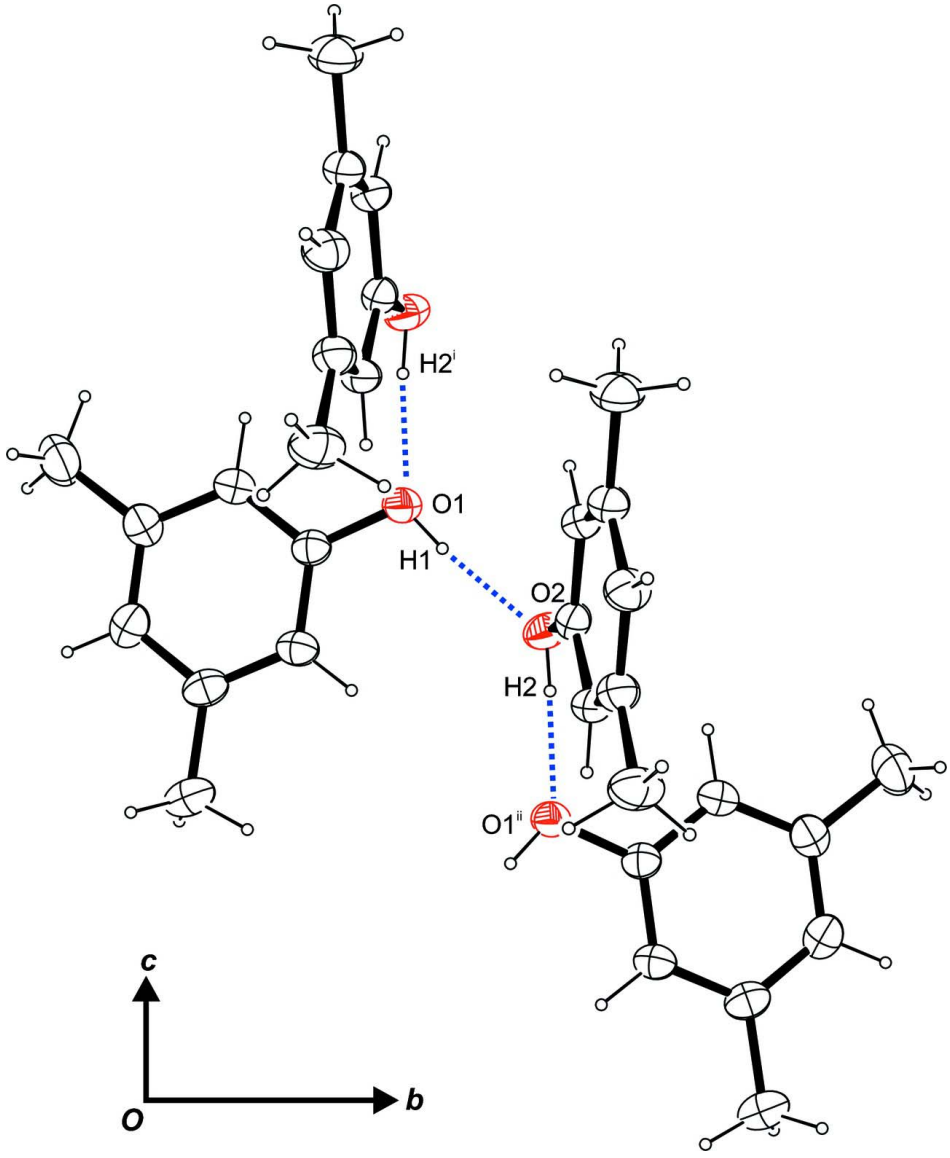

Figure 2

Intermolecular contacts, viewed along [-1 0 0]. Symmetry operators: [i] *x*, *y*, *z* + 1; [ii] *x*, *y*, *z* - 1.

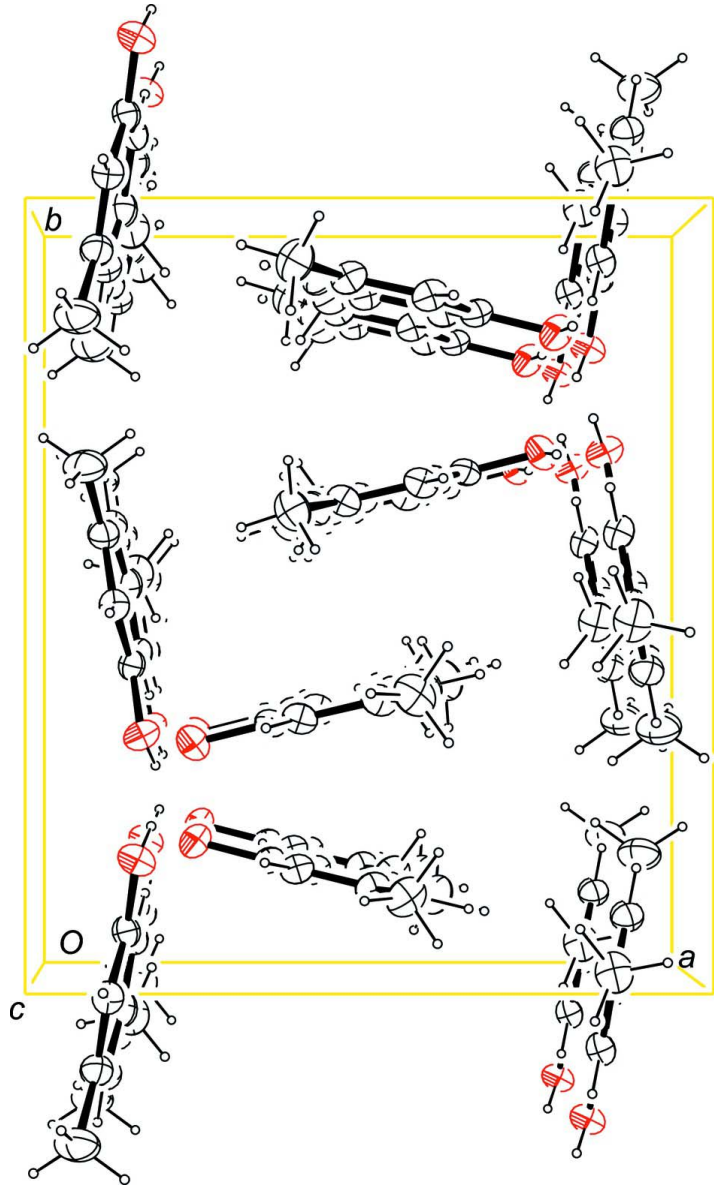

Figure 3

Molecular packing of the title compound, viewed along [0 0 - 1] (anisotropic displacement ellipsoids drawn at 50% probability level).

3,5-Dimethylphenol

Crystal data

$C_8H_{10}O$
M_r = 122.16
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.9807 (6) Å
b = 13.8725 (7) Å
c = 8.5378 (4) Å
β = 90.000 (2)°
V = 1419.00 (12) Å^3
Z = 8
F(000) = 528
D_x = 1.144 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8727 reflections
θ = 2.3–28.3°
μ = 0.07 mm^{-1}

T = 200 K
Block, colourless

0.50 × 0.41 × 0.33 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
12884 measured reflections
3392 independent reflections

2998 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 28.0°, θ_{min} = 3.3°
h = −15→9
k = −18→18
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.143
S = 1.04
3392 reflections
169 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0724P)^2 + 0.556P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.16676 (9)	0.32297 (7)	0.83341 (11)	0.0322 (2)
H1	0.1864	0.2833	0.7644	0.048*
C11	0.15120 (10)	0.41221 (9)	0.76595 (14)	0.0263 (3)
C12	0.12708 (11)	0.48876 (10)	0.86483 (15)	0.0294 (3)
H12	0.1224	0.4787	0.9747	0.035*
C13	0.10982 (12)	0.58017 (10)	0.80279 (17)	0.0334 (3)
C14	0.11818 (12)	0.59291 (10)	0.64172 (17)	0.0356 (3)
H14	0.1070	0.6554	0.5989	0.043*
C15	0.14249 (11)	0.51675 (10)	0.54173 (15)	0.0320 (3)
C16	0.15847 (11)	0.42544 (10)	0.60545 (14)	0.0292 (3)
H16	0.1744	0.3723	0.5390	0.035*
C17	0.08510 (17)	0.66459 (12)	0.9083 (2)	0.0502 (4)
H171	0.0653	0.6410	1.0129	0.075*
H172	0.0227	0.7017	0.8651	0.075*
H173	0.1512	0.7059	0.9155	0.075*
C18	0.15133 (14)	0.53221 (12)	0.36751 (17)	0.0419 (4)
H181	0.0772	0.5460	0.3246	0.063*
H182	0.1813	0.4739	0.3181	0.063*
H183	0.2012	0.5867	0.3465	0.063*
O2	0.24467 (8)	0.31707 (7)	0.13407 (11)	0.0327 (2)
H2	0.2264	0.3213	0.0393	0.049*
C21	0.35593 (11)	0.34152 (8)	0.15086 (14)	0.0269 (3)
C22	0.42454 (12)	0.35771 (9)	0.02291 (14)	0.0293 (3)
H22	0.3951	0.3537	−0.0802	0.035*

C23	0.53654 (12)	0.37975 (10)	0.04558 (15)	0.0321 (3)
C24	0.57689 (12)	0.38687 (10)	0.19756 (16)	0.0341 (3)
H24	0.6532	0.4026	0.2140	0.041*
C25	0.50780 (12)	0.37150 (9)	0.32656 (15)	0.0318 (3)
C26	0.39652 (12)	0.34879 (9)	0.30206 (14)	0.0295 (3)
H26	0.3483	0.3383	0.3887	0.035*
C27	0.61218 (14)	0.39382 (12)	−0.09337 (17)	0.0418 (4)
H271	0.6191	0.3330	−0.1508	0.063*
H272	0.6861	0.4145	−0.0572	0.063*
H273	0.5807	0.4432	−0.1625	0.063*
C28	0.55365 (15)	0.37856 (12)	0.49024 (17)	0.0434 (4)
H281	0.4918	0.3812	0.5652	0.065*
H282	0.5989	0.4371	0.4998	0.065*
H283	0.6001	0.3220	0.5123	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0423 (6)	0.0306 (5)	0.0236 (4)	0.0077 (4)	−0.0022 (4)	−0.0006 (3)
C11	0.0237 (6)	0.0303 (6)	0.0249 (6)	0.0020 (5)	−0.0022 (4)	0.0009 (5)
C12	0.0298 (6)	0.0339 (6)	0.0244 (6)	−0.0006 (5)	−0.0002 (5)	−0.0023 (5)
C13	0.0327 (7)	0.0312 (6)	0.0362 (7)	−0.0009 (5)	0.0021 (5)	−0.0036 (5)
C14	0.0360 (7)	0.0316 (6)	0.0392 (7)	0.0001 (5)	0.0024 (6)	0.0060 (5)
C15	0.0268 (6)	0.0412 (7)	0.0281 (6)	−0.0004 (5)	0.0006 (5)	0.0054 (5)
C16	0.0279 (6)	0.0360 (6)	0.0238 (6)	0.0041 (5)	0.0006 (5)	−0.0016 (5)
C17	0.0630 (11)	0.0348 (8)	0.0526 (10)	0.0025 (7)	0.0076 (8)	−0.0098 (7)
C18	0.0440 (8)	0.0520 (9)	0.0297 (7)	0.0017 (7)	0.0027 (6)	0.0105 (6)
O2	0.0341 (5)	0.0389 (5)	0.0252 (4)	−0.0077 (4)	−0.0050 (4)	0.0058 (4)
C21	0.0318 (7)	0.0234 (5)	0.0255 (6)	−0.0033 (5)	−0.0018 (5)	0.0020 (4)
C22	0.0380 (7)	0.0294 (6)	0.0206 (5)	−0.0045 (5)	−0.0022 (5)	0.0000 (4)
C23	0.0381 (7)	0.0319 (6)	0.0261 (6)	−0.0051 (5)	0.0019 (5)	0.0000 (5)
C24	0.0326 (7)	0.0381 (7)	0.0315 (7)	−0.0070 (6)	−0.0038 (5)	0.0001 (5)
C25	0.0403 (8)	0.0305 (6)	0.0246 (6)	−0.0039 (5)	−0.0050 (5)	0.0002 (5)
C26	0.0377 (7)	0.0288 (6)	0.0220 (6)	−0.0032 (5)	0.0003 (5)	0.0020 (4)
C27	0.0408 (8)	0.0537 (9)	0.0309 (7)	−0.0084 (7)	0.0069 (6)	0.0002 (6)
C28	0.0518 (9)	0.0508 (9)	0.0278 (7)	−0.0088 (7)	−0.0122 (6)	−0.0003 (6)

Geometric parameters (Å, º)

O1—C11	1.3780 (15)	O2—C21	1.3828 (16)
O1—H1	0.8400	O2—H2	0.8400
C11—C16	1.3853 (17)	C21—C26	1.3832 (17)
C11—C12	1.3871 (17)	C21—C22	1.3855 (18)
C12—C13	1.3897 (19)	C22—C23	1.390 (2)
C12—H12	0.9500	C22—H22	0.9500
C13—C14	1.390 (2)	C23—C24	1.3881 (19)
C13—C17	1.507 (2)	C23—C27	1.5056 (19)
C14—C15	1.389 (2)	C24—C25	1.3942 (19)

C14—H14	0.9500	C24—H24	0.9500
C15—C16	1.3918 (19)	C25—C26	1.386 (2)
C15—C18	1.5066 (18)	C25—C28	1.5048 (18)
C16—H16	0.9500	C26—H26	0.9500
C17—H171	0.9800	C27—H271	0.9800
C17—H172	0.9800	C27—H272	0.9800
C17—H173	0.9800	C27—H273	0.9800
C18—H181	0.9800	C28—H281	0.9800
C18—H182	0.9800	C28—H282	0.9800
C18—H183	0.9800	C28—H283	0.9800
C11—O1—H1	109.5	C21—O2—H2	109.5
O1—C11—C16	121.60 (11)	O2—C21—C26	116.97 (11)
O1—C11—C12	117.49 (11)	O2—C21—C22	122.00 (11)
C16—C11—C12	120.90 (12)	C26—C21—C22	121.03 (12)
C11—C12—C13	119.84 (12)	C21—C22—C23	119.91 (12)
C11—C12—H12	120.1	C21—C22—H22	120.0
C13—C12—H12	120.1	C23—C22—H22	120.0
C12—C13—C14	118.84 (12)	C24—C23—C22	118.82 (12)
C12—C13—C17	120.70 (13)	C24—C23—C27	121.18 (13)
C14—C13—C17	120.45 (13)	C22—C23—C27	119.99 (12)
C15—C14—C13	121.76 (13)	C23—C24—C25	121.38 (13)
C15—C14—H14	119.1	C23—C24—H24	119.3
C13—C14—H14	119.1	C25—C24—H24	119.3
C14—C15—C16	118.74 (12)	C26—C25—C24	119.13 (12)
C14—C15—C18	120.88 (13)	C26—C25—C28	120.41 (13)
C16—C15—C18	120.38 (13)	C24—C25—C28	120.46 (13)
C11—C16—C15	119.91 (12)	C21—C26—C25	119.71 (12)
C11—C16—H16	120.0	C21—C26—H26	120.1
C15—C16—H16	120.0	C25—C26—H26	120.1
C13—C17—H171	109.5	C23—C27—H271	109.5
C13—C17—H172	109.5	C23—C27—H272	109.5
H171—C17—H172	109.5	H271—C27—H272	109.5
C13—C17—H173	109.5	C23—C27—H273	109.5
H171—C17—H173	109.5	H271—C27—H273	109.5
H172—C17—H173	109.5	H272—C27—H273	109.5
C15—C18—H181	109.5	C25—C28—H281	109.5
C15—C18—H182	109.5	C25—C28—H282	109.5
H181—C18—H182	109.5	H281—C28—H282	109.5
C15—C18—H183	109.5	C25—C28—H283	109.5
H181—C18—H183	109.5	H281—C28—H283	109.5
H182—C18—H183	109.5	H282—C28—H283	109.5
O1—C11—C12—C13	−179.53 (12)	O2—C21—C22—C23	−178.39 (12)
C16—C11—C12—C13	0.0 (2)	C26—C21—C22—C23	1.3 (2)
C11—C12—C13—C14	−0.6 (2)	C21—C22—C23—C24	−1.2 (2)
C11—C12—C13—C17	−179.15 (14)	C21—C22—C23—C27	177.78 (13)
C12—C13—C14—C15	0.4 (2)	C22—C23—C24—C25	0.6 (2)

C17—C13—C14—C15	179.05 (14)	C27—C23—C24—C25	−178.36 (14)
C13—C14—C15—C16	0.2 (2)	C23—C24—C25—C26	−0.1 (2)
C13—C14—C15—C18	−179.91 (14)	C23—C24—C25—C28	179.34 (14)
O1—C11—C16—C15	−179.85 (12)	O2—C21—C26—C25	178.94 (12)
C12—C11—C16—C15	0.6 (2)	C22—C21—C26—C25	−0.76 (19)
C14—C15—C16—C11	−0.7 (2)	C24—C25—C26—C21	0.2 (2)
C18—C15—C16—C11	179.39 (13)	C28—C25—C26—C21	−179.28 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.84	1.91	2.7463 (13)	171
O2—H2···O1^{ii}	0.84	1.90	2.7327 (13)	172

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