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A new polymorph of physcion

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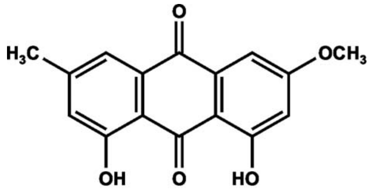
The structure of the title compound, 7-methoxy-2-methyl-4,5-dihydroxyanthracene-9,10-dione, $C_{16}H_{12}O_5$, was originally reported by Ulický *et al.* [*Acta Cryst.* (1991). C**47**, 1879–1881] in the space group $P2_12_12_1$ [polymorph (I*o*)]. The new polymorph, (I*m*), crystallizes in the space group $P2_1/c$. The molecular structures are closely similar, with both –OH groups forming intramolecular hydrogen bonds to one of the neighbouring quinone O atoms, thus slightly lengthening this C═O bond; the pattern of C—C bond lengths in the ring system is consistent with some contribution from a resonance form with a negative charge at the hydrogen-bonded quinone O atom and an aromatic region around its neighbouring C atoms. The packing of (I*m*) is simpler than the extensively crosslinked pattern of (I*o*), with molecular tapes connected by classical (but three-centre) and 'weak' hydrogen bonds, parallel to $[20\overline{1}]$.

Comment

Physcion (7-methoxy-2-methyl-4,5-dihydroxyanthracene-9,10-dione), (I), is a secondary metabolite occurring in many plants and fungi (Podojil *et al.*, 1979). It was studied as early as the end of the 19th century (Zopf, 1898; Hesse, 1912). Ulický *et al.* (1991) crystallized it from chloroform and reported its structure in the solid state, in the space group $P2_12_12_1$ [polymorph (I*o*)]. In a project aimed at isolating naturally occurring dyestuffs from plants, we have obtained this anthraquinone derivative from *Xanthoria parietine*, a lichen growing on numerous shrubs and trees in northern Germany. Our sample was obtained from lichen growing on elder trees (*Sambucus nigra*). In our hands, recrystallization from ethyl acetate led to a new polymorph, (I*m*), in the space group $P2_1/c$, the structure of which is reported here.

The molecule of (I*m*) is shown in Fig. 1. Its dimensions are determined rather more precisely than in the previous analysis, which may be attributed to the low-temperature data collection, the increased $2\theta_{\max}$ and the more favourable data-to-parameter ratio for a centrosymmetric structure. The major features, however, remain essentially those reported by Ulický *et al.* (1991) and may be summarized as follows: (i) both –OH groups form intramolecular hydrogen bonds to the quinone O5 atom (Table 2); (ii) this lengthens the C10═O5 bond slightly compared to C9═O4 (for bond lengths see Table 1); (iii) the C10—C4*A* and C10—C10*A* bonds are lengthened slightly compared to C9—C9*A* and C9—C8*A*, associated with an increased contribution from a resonance form with a more aromatic region around C10 and a negative charge at O5; (iv) the molecule is slightly bent around the central C9···C10 axis, with an interplanar angle of 3.57 (6)° between the planes C1–C4/C4*A*/C9*A*/C9/C10 and C5–C8/C8*A*/C10*A*/C9/C10 (r.m.s. deviations from planarity = 0.006 and 0.009 Å, respectively).



(I)

The molecular packing of (I*m*) involves tapes of molecules parallel to $[20\overline{1}]$ (Fig. 2). The molecules are linked by various hydrogen bonds (Table 2); around atom O5, the intramolecular systems are extended to asymmetric three-centre hydrogen bonds across the inversion centre $0, \frac{1}{2}, 0$, and the opposite ends of the molecules are linked by $H1\cdots O4^{iv}$ [symmetry code: (iv) $-x, -y + 1, -z + 1$] across the inversion centre $0, \frac{1}{2}, \frac{1}{2}$. $H12B\cdots O2^{ii}$ [2.65 Å; symmetry code: (ii) $x - 1, -y + \frac{3}{2}, z + \frac{1}{2}$] contacts may also be structurally significant, but are not shown explicitly in Fig. 2 (for 'weak' hydrogen bonds, the cut-off criterion $H\cdots O \leq 2.66$ Å was used). Neighbouring tapes subtend an angle of 9.30 (2)°, so that all molecules in a layer consisting of such tapes are approximately parallel to each other. The tapes are linked in the third dimension by the contact $H12C\cdots O3^{iii}$ [symmetry code: (iii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$].

In order to analyse the packing of the previously determined $P2_12_12_1$ polymorph (I*o*) (Ulický *et al.*, 1991), we have taken the coordinates from the Cambridge Structural Database (CSD, *ConQuest* Version 1.14; Allen, 2002; refcode

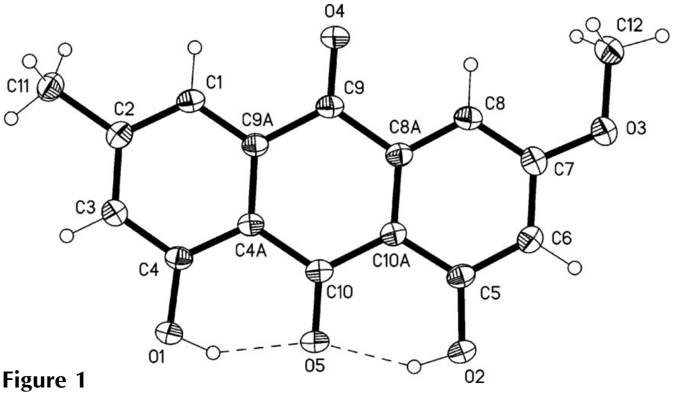


Figure 1
The molecular structure of polymorph (I*m*). Displacement ellipsoids are drawn at the 50% probability level.

doi:10.1107/S010827011203020X

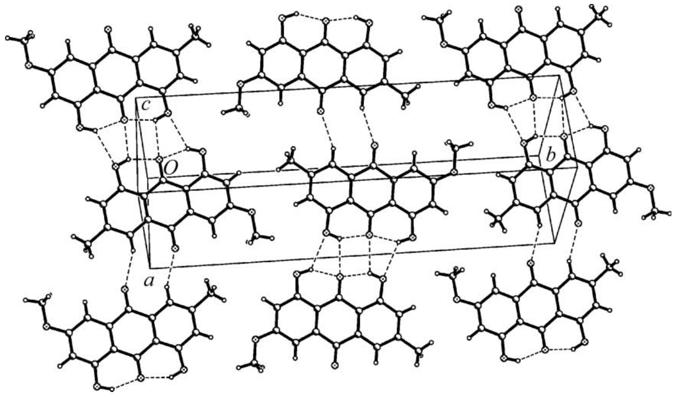


Figure 2
Packing diagram of the title compound [$P2_1/c$, polymorph (I*m*)], viewed parallel to the molecular plane of the asymmetric unit [approximately perpendicular to (102)], showing molecular tapes parallel to [20$\bar{1}$]. Classical and 'weak' hydrogen bonds are indicated by thin dashed lines.

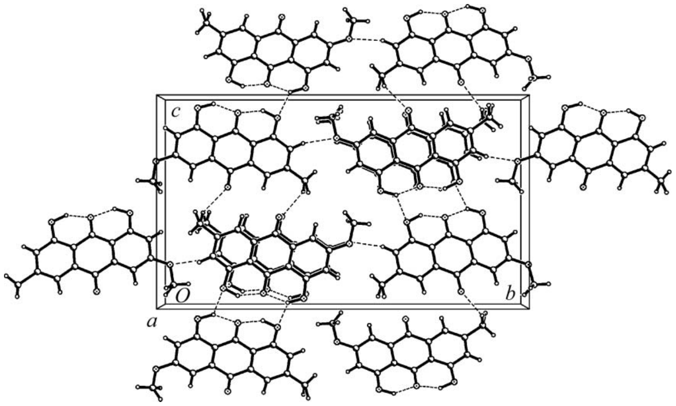


Figure 3
Packing diagram of previously determined polymorph (I*o*) ($P2_12_12_1$), viewed parallel to the short *a* axis. Classical and 'weak' hydrogen bonds are indicated by thin dashed lines.

SOHXAO), renumbered the atoms and standardized the C—H bond lengths (which showed a considerable scatter, associated with the free refinement of the H atoms) to conform with this paper. The following hydrogen bonds are found: (i) O2—H2···O1, with H2···O1 = 2.40 Å and angle = 118° (symmetry code for the acceptor atom: $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 2$); (ii) C3—H3···O3, with H3···O3 = 2.59 Å and angle = 154° (symmetry code for the acceptor atom: $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$); (iii) C11—H11*C*···O4, with H11*C*···O4 = 2.65 Å and angle = 152° (symmetry code for the acceptor atom: $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$). Qualitatively, the intermolecular contacts of (I*o*) differ appreciably from those of (I*m*): (*a*) only one of the intramolecular hydrogen bonds is extended intermolecularly to form a three-centre system; (*b*) atoms H1 and H8, next to the C9═O4 double bond, do not act as hydrogen-bond donors; (*c*) atom O4 accepts a hydrogen bond from a methyl H atom; (*d*) there is a 'side-to-side' H3···O3 linkage. Despite these differences, at first sight, the packing (Fig. 3) of (I*o*) appears similar to that of (I*m*), with apparent molecular tapes parallel to the *c* axis. However, this is an illusion arising from the projection along the short *a* axis. Where two molecules related by *a*-axis translation are seen (there are two such pairs in Fig. 3), it is clear that the linkages to neighbouring molecules in the *c* direction take place at different heights; in other words, the molecules are extensively crosslinked in the third dimension and are by no means parallel, with interplanar angles of *ca* 50° between neighbouring molecules in the *c* direction.

We noted a similar effect for two concomitant polymorphs of *N*,*N*′-bis[4-(diethylamino)phenyl]terephthaldiamide (Kuś *et al.*, 2010); the triclinic polymorph consisted of essentially planar ribbons of molecules linked by classical hydrogen bonds, whereas the monoclinic polymorph consisted of an extensively crosslinked system. For both compounds, it is tempting to surmise that the crosslinked packing represents an island of kinetic stability *en route* to the simpler and presumably more efficient parallel packing. The density of (I*m*) is higher than that of (I*o*) (1.539 compared to 1.51 Mg m^{-3}), but the different measurement temperatures do not allow a meaningful comparison. However, no corresponding energy calculations have been carried out and in neither case was the amount of material sufficient to undertake extensive experimental investigations of possible transformations between polymorphs, *e.g.* at different temperatures or in different solvent systems.

Experimental

The lichen was scraped off twigs and branches of elder trees, and the specimens were extracted with boiling ethyl acetate. The dark-yellow solution was filtered and the solvent removed by rotary evaporation; the remaining brown–yellow oil was subjected to column chromatography on silica gel with ethyl acetate as the eluent. From one of the fractions, shown to be pure by thin-layer chromatography, thin orange plates were obtained when the solvent was left to evaporate slowly at room temperature.

Crystal data

$C_{16}H_{12}O_5$
M_r = 284.26
Monoclinic, $P2_1/c$
a = 6.1986 (3) Å
b = 24.5470 (12) Å
c = 8.1004 (4) Å
β = 95.370 (4)°
V = 1227.12 (10) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.97 mm^{-1}
T = 130 K
0.25 × 0.10 × 0.04 mm

Data collection

Agilent Technologies Xcalibur (Atlas, Nova) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{\min}$ = 0.646, $T_{\max}$ = 1.000
43156 measured reflections
2553 independent reflections
2314 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
S = 1.05
2553 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.31 e Å^{-3}
$\Delta\rho_{\min}$ = −0.25 e Å^{-3}

The hydroxy H atoms were refined freely. Methyl H atoms were identified in difference syntheses; the geometry was idealized (C—H = 0.98 Å and H—C—H = 109.5°) and the methyl groups

Table 1
Selected bond lengths (Å).

C4*A*—C9*A*	1.4127 (18)	C9—O4	1.2211 (17)
C4*A*—C10	1.4652 (18)	C9—C9*A*	1.4843 (19)
C8*A*—C10*A*	1.4048 (18)	C10—O5	1.2542 (16)
C8*A*—C9	1.4921 (19)	C10—C10*A*	1.4651 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H01⋯O5	0.86 (2)	1.81 (2)	2.5947 (14)	150 (2)
O2—H02⋯O5	0.89 (3)	1.82 (3)	2.6192 (14)	148 (2)
O1—H01⋯O5^{i}	0.86 (2)	2.36 (2)	2.8191 (15)	113.4 (19)
O2—H02⋯O1^{i}	0.89 (3)	2.44 (2)	3.0934 (15)	130.3 (18)
C12—H12*B*⋯O2^{ii}	0.98	2.65	3.4091 (18)	134
C12—H12*C*⋯O3^{iii}	0.98	2.64	3.4878 (19)	145
C1—H1⋯O4^{iv}	0.95	2.40	3.2900 (17)	157

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

refined as rigid groups allowed to rotate but not tip. For all methyl H atoms, $U_{\rm iso}$(H) values were set at $1.5U_{\rm eq}$(C). Other H atoms were included in the refinement using a riding model starting from calculated positions, with aromatic C—H bond lengths of 0.98 Å and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MX3079). Services for accessing these data are described at the back of the journal.

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supplementary materials

Acta Cryst. (2012). C**68**, o317–o319 [doi:10.1107/S010827011203020X]

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$c = 8.1004$ (4) Å
$\beta = 95.370$ (4)°
$V = 1227.12$ (10) Å^3
$Z = 4$

$F(000) = 592$
$D_x = 1.539$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 17945 reflections
$\theta = 3.6$–75.7°
$\mu = 0.97$ mm^{-1}
$T = 130$ K
Plate, orange
0.25 × 0.10 × 0.04 mm

Data collection

Agilent Technologies Xcalibur (Atlas, Nova) diffractometer
Radiation source: Nova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 10.3543 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2012)
$T_{\min} = 0.646$, $T_{\max} = 1.000$

43156 measured reflections
2553 independent reflections
2314 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.036$
$\theta_{\max} = 75.9$°, $\theta_{\min} = 3.6$°
$h = -7 \rightarrow 7$
$k = -30 \rightarrow 30$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.126$
$S = 1.05$
2553 reflections
200 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
$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2 + 0.6508P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

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.
Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
2.9925 (0.0010) x - 1.9903 (0.0028) y + 6.6665 (0.0010) z = 2.1195 (0.0017)
* -0.0011 (0.0012) C1 * -0.0526 (0.0012) C2 * -0.0515 (0.0012) C3 * -0.0097 (0.0012) C4 * 0.0333 (0.0012) C4A * -0.0044 (0.0012) C5 * -0.0480 (0.0013) C6 * -0.0231 (0.0013) C7 * 0.0295 (0.0013) C8 * 0.0580 (0.0012) C8A * 0.0859 (0.0013) C9 * 0.0383 (0.0012) C9A * 0.0533 (0.0012) C10 * 0.0478 (0.0012) C10A * -0.1123 (0.0012) C11 * -0.0991 (0.0013) C12 * -0.0371 (0.0010) O1 * -0.0299 (0.0010) O2 * -0.0714 (0.0011) O3 * 0.1404 (0.0011) O4 * 0.0539 (0.0009) O5
Rms deviation of fitted atoms = 0.0618
2.9946 (0.0023) x - 2.8088 (0.0087) y + 6.6344 (0.0021) z = 1.7346 (0.0043)
Angle to previous plane (with approximate e.s.d.) = 1.92 (0.04)
* -0.0015 (0.0011) C1 * -0.0083 (0.0010) C2 * 0.0011 (0.0010) C3 * 0.0078 (0.0011) C4 * 0.0059 (0.0011) C4A * 0.0028 (0.0012) C9A * 0.0037 (0.0009) C9 * -0.0114 (0.0008) C10 - 0.0084 (0.0016) O1 - 0.0301 (0.0020) C11
Rms deviation of fitted atoms = 0.0063
3.0774 (0.0023) x - 1.3155 (0.0085) y + 6.6110 (0.0021) z = 2.5977 (0.0054)
Angle to previous plane (with approximate e.s.d.) = 3.57 (0.06)
* 0.0056 (0.0010) C5 * -0.0117 (0.0011) C6 * -0.0036 (0.0011) C7 * 0.0058 (0.0011) C8 * 0.0084 (0.0011) C8A * 0.0139 (0.0011) C10A * -0.0096 (0.0009) C9 * -0.0088 (0.0009) C10 - 0.0013 (0.0016) O2 - 0.0237 (0.0017) O3
Rms deviation of fitted atoms = 0.0090

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	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.2545 (2)	0.45799 (6)	0.34025 (17)	0.0214 (3)
H1	0.1301	0.4629	0.3988	0.026*
C2	0.3121 (2)	0.40551 (6)	0.29103 (17)	0.0216 (3)
C3	0.4950 (2)	0.39912 (6)	0.20716 (17)	0.0221 (3)
H3	0.5351	0.3637	0.1743	0.027*
C4	0.6220 (2)	0.44367 (6)	0.16971 (16)	0.0202 (3)
C4A	0.5647 (2)	0.49666 (5)	0.21771 (16)	0.0187 (3)
C5	0.7359 (2)	0.64581 (6)	0.17976 (17)	0.0210 (3)
C6	0.6626 (2)	0.69719 (6)	0.22145 (18)	0.0229 (3)
H6	0.7374	0.7288	0.1903	0.028*
C7	0.4803 (2)	0.70252 (6)	0.30861 (17)	0.0226 (3)
C8	0.3673 (2)	0.65641 (6)	0.35343 (17)	0.0219 (3)
H8	0.2429	0.6600	0.4127	0.026*
C8A	0.4392 (2)	0.60543 (6)	0.31022 (16)	0.0197 (3)
C9	0.3110 (2)	0.55739 (6)	0.35761 (17)	0.0213 (3)
C9A	0.3783 (2)	0.50264 (6)	0.30392 (16)	0.0194 (3)
C10	0.6933 (2)	0.54407 (5)	0.17714 (16)	0.0189 (3)
C10A	0.6236 (2)	0.59851 (5)	0.22387 (16)	0.0188 (3)
C11	0.1749 (2)	0.35733 (6)	0.32927 (19)	0.0264 (3)
H11A	0.2113	0.3262	0.2615	0.040*
H11B	0.0214	0.3665	0.3043	0.040*

H11C	0.2026	0.3480	0.4469	0.040*
C12	0.2300 (3)	0.76133 (6)	0.4271 (2)	0.0298 (3)
H12A	0.1050	0.7468	0.3581	0.045*
H12B	0.2073	0.8002	0.4477	0.045*
H12C	0.2467	0.7417	0.5329	0.045*
O1	0.79500 (17)	0.43385 (4)	0.08503 (13)	0.0252 (3)
H01	0.856 (4)	0.4649 (10)	0.074 (3)	0.061 (7)*
O2	0.91320 (17)	0.64404 (4)	0.09580 (13)	0.0256 (3)
H02	0.938 (4)	0.6089 (10)	0.076 (3)	0.054 (6)*
O3	0.42231 (18)	0.75449 (4)	0.34290 (14)	0.0281 (3)
O4	0.15523 (18)	0.56347 (4)	0.43754 (14)	0.0307 (3)
O5	0.85900 (15)	0.53826 (4)	0.10111 (12)	0.0235 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0201 (7)	0.0264 (7)	0.0182 (6)	−0.0014 (5)	0.0043 (5)	0.0005 (5)
C2	0.0219 (7)	0.0231 (7)	0.0193 (7)	−0.0020 (5)	−0.0005 (5)	0.0019 (5)
C3	0.0224 (7)	0.0215 (7)	0.0223 (7)	0.0011 (5)	0.0013 (5)	−0.0007 (5)
C4	0.0183 (7)	0.0252 (7)	0.0172 (6)	0.0022 (5)	0.0021 (5)	0.0005 (5)
C4A	0.0170 (6)	0.0225 (7)	0.0165 (6)	0.0001 (5)	0.0014 (5)	0.0007 (5)
C5	0.0175 (6)	0.0272 (7)	0.0180 (6)	−0.0021 (5)	0.0008 (5)	0.0011 (5)
C6	0.0236 (7)	0.0225 (7)	0.0227 (7)	−0.0029 (5)	0.0019 (6)	0.0012 (5)
C7	0.0245 (7)	0.0220 (7)	0.0207 (7)	0.0021 (5)	−0.0013 (5)	−0.0009 (5)
C8	0.0205 (7)	0.0265 (7)	0.0192 (7)	0.0005 (5)	0.0041 (5)	−0.0009 (5)
C8A	0.0181 (7)	0.0241 (7)	0.0171 (6)	−0.0009 (5)	0.0016 (5)	−0.0002 (5)
C9	0.0197 (7)	0.0255 (7)	0.0193 (6)	−0.0007 (5)	0.0047 (5)	−0.0004 (5)
C9A	0.0185 (7)	0.0227 (7)	0.0173 (6)	0.0000 (5)	0.0023 (5)	0.0001 (5)
C10	0.0166 (6)	0.0247 (7)	0.0153 (6)	0.0002 (5)	0.0009 (5)	0.0006 (5)
C10A	0.0182 (6)	0.0220 (7)	0.0159 (6)	−0.0007 (5)	0.0008 (5)	0.0003 (5)
C11	0.0273 (8)	0.0235 (7)	0.0285 (8)	−0.0038 (6)	0.0039 (6)	0.0016 (6)
C12	0.0281 (8)	0.0274 (7)	0.0346 (8)	0.0029 (6)	0.0062 (6)	−0.0035 (6)
O1	0.0213 (5)	0.0240 (5)	0.0317 (6)	0.0017 (4)	0.0099 (4)	−0.0017 (4)
O2	0.0219 (5)	0.0261 (6)	0.0303 (6)	−0.0032 (4)	0.0100 (4)	0.0008 (4)
O3	0.0307 (6)	0.0217 (5)	0.0326 (6)	0.0013 (4)	0.0072 (5)	−0.0021 (4)
O4	0.0287 (6)	0.0275 (5)	0.0389 (6)	−0.0025 (4)	0.0186 (5)	−0.0041 (4)
O5	0.0191 (5)	0.0267 (5)	0.0256 (5)	0.0000 (4)	0.0081 (4)	−0.0002 (4)

Geometric parameters (Å, º)

C1—C9A	1.3852 (19)	C9—O4	1.2211 (17)
C1—C2	1.4046 (19)	C9—C9A	1.4843 (19)
C2—C3	1.3843 (19)	C10—O5	1.2542 (16)
C2—C11	1.5055 (19)	C10—C10A	1.4651 (18)
C3—C4	1.3974 (19)	C12—O3	1.4374 (18)
C4—O1	1.3475 (16)	C1—H1	0.9500
C4—C4A	1.4124 (19)	C3—H3	0.9500
C4A—C9A	1.4127 (18)	C6—H6	0.9500
C4A—C10	1.4652 (18)	C8—H8	0.9500
C5—O2	1.3464 (17)	C11—H11A	0.9800

C5—C6	1.3927 (19)	C11—H11B	0.9800
C5—C10A	1.4166 (18)	C11—H11C	0.9800
C6—C7	1.394 (2)	C12—H12A	0.9800
C7—O3	1.3609 (16)	C12—H12B	0.9800
C7—C8	1.397 (2)	C12—H12C	0.9800
C8—C8A	1.3844 (19)	O1—H01	0.86 (2)
C8A—C10A	1.4048 (18)	O2—H02	0.89 (3)
C8A—C9	1.4921 (19)		

C9A—C1—C2	120.33 (13)	O5—C10—C4A	120.49 (12)
C3—C2—C1	118.99 (12)	C10A—C10—C4A	119.13 (12)
C3—C2—C11	121.09 (13)	C8A—C10A—C5	117.92 (12)
C1—C2—C11	119.92 (13)	C8A—C10A—C10	120.81 (12)
C2—C3—C4	121.47 (13)	C5—C10A—C10	121.24 (12)
O1—C4—C3	117.51 (12)	C7—O3—C12	116.94 (11)
O1—C4—C4A	122.51 (12)	C9A—C1—H1	119.8
C3—C4—C4A	119.97 (13)	C2—C1—H1	119.8
C4—C4A—C9A	118.05 (12)	C2—C3—H3	119.3
C4—C4A—C10	120.91 (12)	C4—C3—H3	119.3
C9A—C4A—C10	121.03 (12)	C5—C6—H6	119.8
O2—C5—C6	116.87 (12)	C7—C6—H6	119.8
O2—C5—C10A	123.00 (12)	C8A—C8—H8	120.5
C6—C5—C10A	120.13 (13)	C7—C8—H8	120.5
C5—C6—C7	120.41 (13)	C2—C11—H11A	109.5
O3—C7—C6	115.69 (12)	C2—C11—H11B	109.5
O3—C7—C8	123.93 (13)	H11A—C11—H11B	109.5
C6—C7—C8	120.37 (13)	C2—C11—H11C	109.5
C8A—C8—C7	119.07 (13)	H11A—C11—H11C	109.5
C8—C8A—C10A	122.09 (12)	H11B—C11—H11C	109.5
C8—C8A—C9	117.23 (12)	O3—C12—H12A	109.5
C10A—C8A—C9	120.68 (12)	O3—C12—H12B	109.5
O4—C9—C9A	121.59 (12)	H12A—C12—H12B	109.5
O4—C9—C8A	120.43 (12)	O3—C12—H12C	109.5
C9A—C9—C8A	117.98 (12)	H12A—C12—H12C	109.5
C1—C9A—C4A	121.18 (13)	H12B—C12—H12C	109.5
C1—C9A—C9	118.54 (12)	C4—O1—H01	106.1 (16)
C4A—C9A—C9	120.28 (12)	C5—O2—H02	106.4 (14)
O5—C10—C10A	120.37 (12)		

C9A—C1—C2—C3	0.6 (2)	C10—C4A—C9A—C1	179.01 (12)
C9A—C1—C2—C11	−179.24 (12)	C4—C4A—C9A—C9	179.86 (11)
C1—C2—C3—C4	−0.4 (2)	C10—C4A—C9A—C9	−0.98 (19)
C11—C2—C3—C4	179.35 (13)	O4—C9—C9A—C1	2.7 (2)
C2—C3—C4—O1	−178.79 (12)	C8A—C9—C9A—C1	−176.96 (12)
C2—C3—C4—C4A	0.0 (2)	O4—C9—C9A—C4A	−177.28 (13)
O1—C4—C4A—C9A	179.03 (12)	C8A—C9—C9A—C4A	3.03 (19)
C3—C4—C4A—C9A	0.3 (2)	C4—C4A—C10—O5	−1.14 (19)
O1—C4—C4A—C10	−0.1 (2)	C9A—C4A—C10—O5	179.72 (12)
C3—C4—C4A—C10	−178.89 (12)	C4—C4A—C10—C10A	177.75 (12)

O2—C5—C6—C7	−179.89 (12)	C9A—C4A—C10—C10A	−1.38 (19)
C10A—C5—C6—C7	0.8 (2)	C8—C8A—C10A—C5	−0.5 (2)
C5—C6—C7—O3	−179.93 (12)	C9—C8A—C10A—C5	178.83 (12)
C5—C6—C7—C8	−0.8 (2)	C8—C8A—C10A—C10	−178.84 (12)
O3—C7—C8—C8A	179.15 (12)	C9—C8A—C10A—C10	0.49 (19)
C6—C7—C8—C8A	0.1 (2)	O2—C5—C10A—C8A	−179.43 (12)
C7—C8—C8A—C10A	0.6 (2)	C6—C5—C10A—C8A	−0.20 (19)
C7—C8—C8A—C9	−178.78 (12)	O2—C5—C10A—C10	−1.1 (2)
C8—C8A—C9—O4	−3.1 (2)	C6—C5—C10A—C10	178.12 (12)
C10A—C8A—C9—O4	177.51 (13)	O5—C10—C10A—C8A	−179.48 (12)
C8—C8A—C9—C9A	176.57 (12)	C4A—C10—C10A—C8A	1.63 (19)
C10A—C8A—C9—C9A	−2.80 (19)	O5—C10—C10A—C5	2.2 (2)
C2—C1—C9A—C4A	−0.3 (2)	C4A—C10—C10A—C5	−176.65 (11)
C2—C1—C9A—C9	179.72 (12)	C6—C7—O3—C12	177.68 (12)
C4—C4A—C9A—C1	−0.2 (2)	C8—C7—O3—C12	−1.4 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H01···O5	0.86 (2)	1.81 (2)	2.5947 (14)	150 (2)
O2—H02···O5	0.89 (3)	1.82 (3)	2.6192 (14)	148 (2)
O1—H01···O5^{i}	0.86 (2)	2.36 (2)	2.8191 (15)	113.4 (19)
O2—H02···O1^{i}	0.89 (3)	2.44 (2)	3.0934 (15)	130.3 (18)
C12—H12*B*···O2^{ii}	0.98	2.65	3.4091 (18)	134
C12—H12*C*···O3^{iii}	0.98	2.64	3.4878 (19)	145
C1—H1···O4^{iv}	0.95	2.40	3.2900 (17)	157

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