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1-(2,6-Dihydroxyphenyl)tetradecan-1-one: isolated from the fruit rinds of *Myristica malabarica*

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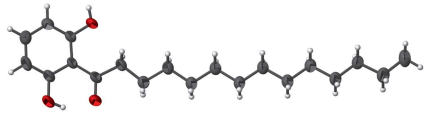
Keywords: crystal structure; 2,6-dihydroxyphenyl; tetradecyl ketone; isolation; *M. malabarica*; *Antileishmanial* activity; O—H···O hydrogen bonding.

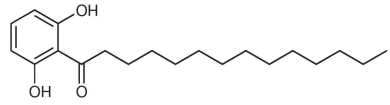
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{20}H_{32}O_3$, was isolated from the Indian spice *M. malabarica*. It is built up by a C—C linkage between a 2,6-dihydroxyphenyl moiety and the terminal carbonyl C atom of tetradecanal, which has an extended chain conformation. There is an intramolecular O—H···O hydrogen bond enclosing an *S*(6) ring motif. In the crystal, molecules are linked by O—H···O hydrogen bonds, forming zigzag chains propagating along [001]. The chains pack in a herringbone arrangement up the *a* axis.

3D view



Chemical scheme



Structure description

The origin of the title compound is fruit rinds of *M. malabarica*, popularly known as *Ram patri* in the local dialect in Mumbai. It is used as an exotic spice in various Indian cuisines and as a phytomedicine for the treatment of various kinds of ailments (Forrest & Heacock, 1972). It has been isolated for the first time from the diethyl ether extract by column chromatography over silica gel with gradient solvent elution. It is soluble in various organic solvents such as diethyl ether, chloroform, methanol *etc*. and undergoes reactions with different kind of chemical reagents such as dilute aqueous sodium hydroxide, neutral ferric chloride solution to exhibit a pale yellow and greenish blue colour due to the formation of the respective sodium salt and ferric complex of the phenol (Dean, 1963). This chemical test indicates the presence of the 3-hydroxy ketone moiety in this molecule, which is also confirmed by UV absorption by performing a bathochromic shift at around 30 nm upon the addition of $AlCl_3$ as shift reagent under the condition of acidic pH. The *antileishmanial* activity of the title molecule has been evaluated against *Leishmania donovani* by using the MTS–PMS assay (Manna *et al.*, 2012).

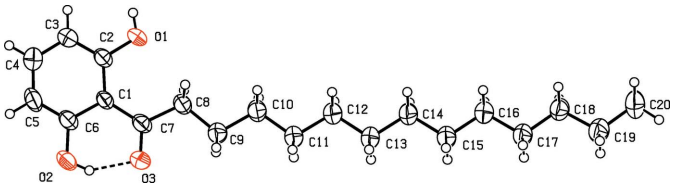


Figure 1
An view of molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

The experimental result of the bioassay revealed that it possesses very good inhibitory activity against the protozoan parasite *Leishmania donovani* (Sen *et al.*, 2007).

The molecular structure of the title compound is illustrated in Fig. 1. It is composed of a 2,6-dihydroxybenzene group linked to the carbonyl C atom, C7, of tetradecanal. The latter has an extended chain conformation. There is an intramolecular O—H···$O_{carbonyl}$ hydrogen bond forming an *S*(6) loop.

In the crystal, molecules are linked by O—H···O hydrogen bonds, forming zigzag chains propagating along the *c*-axis direction (Table 1 and Fig. 2). The chains pack in a herringbone arrangement up the *a* axis (Fig. 2). There are no other significant intermolecular interactions present in the crystal.

Synthesis and crystallization

The title molecule was isolated as a small trace quantity from a methanol extract of the fruit rind of *M. malabarica* by column chromatography over silica gel with gradient solvent elution by using a binary solvent mixture of methanol and chloroform. Suitable crystals for X-ray diffraction analysis were obtained by recrystallization (× 3) from hexane:ethyl acetate (4:1) at room temperature, by slow evaporation (m.p. 363 K). Spectroscopic analysis: ^{1}H NMR data ($CDCl_3$, 200 MHz): 12.80 (*s*, chelated-OH), 7.07 (*dd*, 1H, *J* = 8.2 Hz, H-4′), 6.22 (*d*, 2H, *J* = 8.2 Hz, H-3′ & H-5′), 2.99 (*dd*, 2H, *J* = 7.0 Hz, H-2), 1.67–1.40 (*m*, 4H, H-3 & H-13), 1.16 (*brs*, 18H, 9 × -CH_2–), 0.78 (*t*, 3H, *J* = 6.0 Hz, -CH_3). ^{13}C NMR data (50 MHz, $CDCl_3$): 209.59 (C-1, >C═O), 163.40 (C-2′ & C-6′, Ar—C—OH), 143.90 (C-1′, Ar—C—C), 111.35 (C-5′, Ar—C—H), 108.31 (C-3′, Ar—C—H), 45.70 (C-2, –CH_2—CO–), 30.52 (C-3, –CH_2—CH_3), 30.45 (C-5, –CH_2—CH_3), 30.27 (9 × C–CH_2–), 14.47 (–CH_3), 17.09 (C-8, –CH_2–). EIMS (70 ev) data: EIMS *m*/*z* (%) [M^+] 320 (12), 320 (14), 278 (2), 256 (3), 202 (4), 189 (7), 176 (5), 165 (12), 151 (37), 137 (100; base peak), 123 (12), 109 (9), 96 (14), 83 (11), 69 (5).

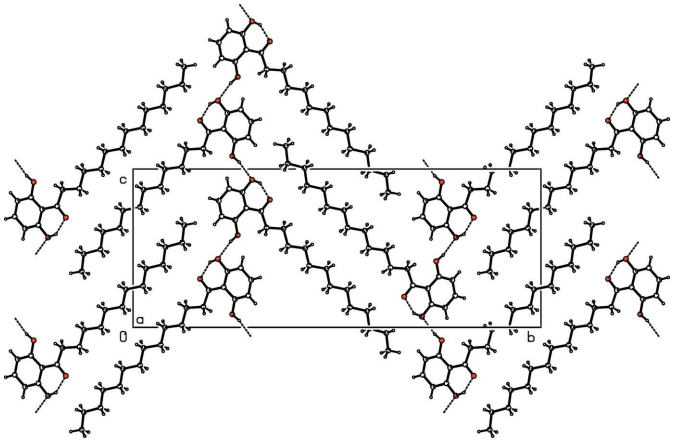


Figure 2
A view of the molecular packing of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O3	0.84 (2)	1.75 (3)	2.485 (4)	146 (4)
O1—H1*O*···O2^{i}	0.84 (2)	1.94 (2)	2.760 (3)	168 (4)

Symmetry code: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{32}O_3$
M_r	320.46
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	4.2047 (6), 34.146 (4), 13.347 (3)
β (°)	97.67 (1)
V (Å^3)	1899.1 (6)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.07
Crystal size (mm)	0.50 × 0.12 × 0.08
Data collection	
Diffractometer	Oxford Diffraction Xcalibur, Sapphire CCD
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min}, T_{max}	0.964, 0.994
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6324, 3396, 2217
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.086, 0.160, 1.30
No. of reflections	3396
No. of parameters	214
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.16

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{20}H_{32}O_3$
M_r = 320.46
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 4.2047 (6) Å
b = 34.146 (4) Å
c = 13.347 (3) Å
β = 97.67 (1)°
V = 1899.1 (6) Å^3
Z = 4
$F(000)$ = 704
D_x = 1.121 Mg m^{-3}
Melting point: 363 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1086 reflections
θ = 2.8–27.9°
μ = 0.07 mm^{-1}
T = 293 K
Needle, colourless
0.50 × 0.12 × 0.08 mm

Data collection

Oxford Diffraction Xcalibur, Sapphire CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Rotation method data acquisition using ω scans.
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)
T_{min} = 0.964, T_{max} = 0.994
6324 measured reflections
3396 independent reflections
2217 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.3°, θ_{min} = 2.8°
h = −5→2
k = −41→33
l = −16→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.160
S = 1.30
3396 reflections
214 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0204P)^2 + 1.6101P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Special details

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	−0.0113 (8)	0.72255 (9)	0.2460 (2)	0.0393 (8)
C2	−0.0688 (8)	0.74981 (10)	0.3212 (2)	0.0425 (8)
C3	−0.2542 (9)	0.78269 (10)	0.2988 (2)	0.0532 (9)
H3	−0.2905	0.8000	0.3498	0.064*
C4	−0.3858 (10)	0.78977 (12)	0.2000 (3)	0.0640 (11)
H4	−0.5121	0.8119	0.1847	0.077*
C5	−0.3317 (9)	0.76445 (11)	0.1246 (3)	0.0597 (10)
H5	−0.4209	0.7695	0.0583	0.072*
C6	−0.1467 (8)	0.73162 (10)	0.1462 (2)	0.0464 (9)
C7	0.1710 (9)	0.68584 (10)	0.2653 (2)	0.0470 (9)
C8	0.3023 (8)	0.67262 (9)	0.3702 (2)	0.0454 (8)
H8A	0.1333	0.6746	0.4128	0.054*
H8B	0.4726	0.6904	0.3971	0.054*
C9	0.4330 (9)	0.63108 (9)	0.3769 (2)	0.0492 (9)
H9A	0.2658	0.6130	0.3494	0.059*
H9B	0.6084	0.6289	0.3368	0.059*
C10	0.5516 (8)	0.62006 (10)	0.4857 (2)	0.0498 (9)
H10A	0.7275	0.6373	0.5109	0.060*
H10B	0.3797	0.6246	0.5261	0.060*
C11	0.6644 (9)	0.57805 (10)	0.5009 (2)	0.0512 (9)
H11A	0.8370	0.5734	0.4609	0.061*
H11B	0.4889	0.5606	0.4763	0.061*
C12	0.7816 (9)	0.56813 (10)	0.6106 (2)	0.0508 (9)
H12A	0.9618	0.5850	0.6340	0.061*
H12B	0.6114	0.5740	0.6507	0.061*
C13	0.8842 (9)	0.52598 (10)	0.6300 (3)	0.0534 (9)
H13A	0.7034	0.5090	0.6082	0.064*
H13B	1.0526	0.5198	0.5895	0.064*
C14	1.0047 (9)	0.51736 (10)	0.7395 (2)	0.0515 (9)
H14A	0.8372	0.5241	0.7799	0.062*
H14B	1.1870	0.5342	0.7607	0.062*
C15	1.1044 (9)	0.47527 (10)	0.7621 (3)	0.0540 (9)
H15A	0.9208	0.4584	0.7431	0.065*
H15B	1.2684	0.4682	0.7207	0.065*
C16	1.2325 (9)	0.46803 (10)	0.8721 (3)	0.0532 (9)

H16A	1.4150	0.4851	0.8908	0.064*
H16B	1.0679	0.4752	0.9132	0.064*
C17	1.3348 (9)	0.42633 (10)	0.8971 (3)	0.0533 (9)
H17A	1.4977	0.4188	0.8557	0.064*
H17B	1.1519	0.4091	0.8801	0.064*
C18	1.4666 (9)	0.42050 (10)	1.0074 (3)	0.0525 (9)
H18A	1.6473	0.4380	1.0242	0.063*
H18B	1.3025	0.4280	1.0484	0.063*
C19	1.5747 (10)	0.37927 (10)	1.0357 (3)	0.0635 (11)
H19A	1.3939	0.3617	1.0200	0.076*
H19B	1.7379	0.3716	0.9946	0.076*
C20	1.7081 (10)	0.37452 (12)	1.1458 (3)	0.0743 (12)
H20A	1.5470	0.3816	1.1871	0.111*
H20B	1.8919	0.3912	1.1616	0.111*
H20C	1.7701	0.3477	1.1587	0.111*
O1	0.0677 (7)	0.74329 (7)	0.41776 (17)	0.0621 (7)
H1O	0.008 (9)	0.7602 (9)	0.456 (2)	0.075*
O2	−0.0966 (7)	0.70821 (7)	0.06808 (17)	0.0638 (8)
H2O	0.016 (8)	0.6889 (8)	0.088 (3)	0.077*
O3	0.2155 (8)	0.66456 (7)	0.19388 (18)	0.0763 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.052 (2)	0.0385 (18)	0.0266 (16)	−0.0058 (15)	0.0025 (14)	0.0033 (14)
C2	0.053 (2)	0.0442 (19)	0.0296 (18)	−0.0059 (16)	0.0030 (15)	0.0015 (15)
C3	0.071 (3)	0.051 (2)	0.038 (2)	0.0043 (19)	0.0059 (17)	−0.0029 (17)
C4	0.077 (3)	0.060 (2)	0.053 (2)	0.019 (2)	0.003 (2)	0.008 (2)
C5	0.074 (3)	0.065 (3)	0.036 (2)	0.002 (2)	−0.0069 (18)	0.0120 (18)
C6	0.064 (2)	0.044 (2)	0.0304 (18)	−0.0107 (17)	0.0021 (16)	0.0013 (15)
C7	0.067 (2)	0.044 (2)	0.0305 (18)	−0.0081 (17)	0.0069 (16)	0.0006 (15)
C8	0.055 (2)	0.046 (2)	0.0340 (18)	−0.0023 (16)	0.0012 (15)	0.0036 (15)
C9	0.058 (2)	0.044 (2)	0.044 (2)	0.0003 (17)	0.0033 (16)	0.0028 (16)
C10	0.054 (2)	0.049 (2)	0.045 (2)	0.0015 (17)	0.0014 (16)	0.0065 (17)
C11	0.060 (2)	0.048 (2)	0.045 (2)	0.0040 (17)	0.0024 (17)	0.0059 (16)
C12	0.057 (2)	0.049 (2)	0.046 (2)	0.0035 (17)	0.0056 (17)	0.0072 (17)
C13	0.063 (2)	0.046 (2)	0.050 (2)	0.0060 (18)	0.0031 (17)	0.0081 (17)
C14	0.058 (2)	0.049 (2)	0.047 (2)	0.0050 (17)	0.0066 (17)	0.0089 (17)
C15	0.063 (2)	0.048 (2)	0.049 (2)	0.0026 (18)	0.0007 (18)	0.0065 (17)
C16	0.060 (2)	0.048 (2)	0.051 (2)	0.0050 (18)	0.0042 (18)	0.0065 (17)
C17	0.062 (2)	0.045 (2)	0.052 (2)	0.0017 (17)	0.0035 (18)	0.0066 (17)
C18	0.058 (2)	0.046 (2)	0.054 (2)	0.0039 (17)	0.0073 (18)	0.0075 (17)
C19	0.077 (3)	0.049 (2)	0.064 (3)	0.002 (2)	0.003 (2)	0.008 (2)
C20	0.084 (3)	0.068 (3)	0.068 (3)	0.008 (2)	0.000 (2)	0.023 (2)
O1	0.096 (2)	0.0599 (17)	0.0268 (13)	0.0189 (15)	−0.0037 (12)	−0.0071 (11)
O2	0.111 (2)	0.0497 (16)	0.0279 (13)	−0.0022 (15)	0.0005 (13)	−0.0004 (12)
O3	0.138 (3)	0.0541 (16)	0.0363 (15)	0.0235 (16)	0.0090 (15)	−0.0027 (13)

Geometric parameters (Å, º)

C1—C6	1.412 (4)	C12—H12B	0.9700
C1—C2	1.413 (4)	C13—C14	1.510 (4)
C1—C7	1.474 (4)	C13—H13A	0.9700
C2—O1	1.357 (4)	C13—H13B	0.9700
C2—C3	1.377 (5)	C14—C15	1.516 (4)
C3—C4	1.381 (5)	C14—H14A	0.9700
C3—H3	0.9300	C14—H14B	0.9700
C4—C5	1.369 (5)	C15—C16	1.516 (4)
C4—H4	0.9300	C15—H15A	0.9700
C5—C6	1.373 (5)	C15—H15B	0.9700
C5—H5	0.9300	C16—C17	1.512 (4)
C6—O2	1.352 (4)	C16—H16A	0.9700
C7—O3	1.233 (4)	C16—H16B	0.9700
C7—C8	1.503 (4)	C17—C18	1.515 (4)
C8—C9	1.520 (4)	C17—H17A	0.9700
C8—H8A	0.9700	C17—H17B	0.9700
C8—H8B	0.9700	C18—C19	1.512 (4)
C9—C10	1.519 (4)	C18—H18A	0.9700
C9—H9A	0.9700	C18—H18B	0.9700
C9—H9B	0.9700	C19—C20	1.510 (5)
C10—C11	1.516 (4)	C19—H19A	0.9700
C10—H10A	0.9700	C19—H19B	0.9700
C10—H10B	0.9700	C20—H20A	0.9600
C11—C12	1.519 (4)	C20—H20B	0.9600
C11—H11A	0.9700	C20—H20C	0.9600
C11—H11B	0.9700	O1—H1O	0.836 (18)
C12—C13	1.515 (4)	O2—H2O	0.835 (18)
C12—H12A	0.9700		
C6—C1—C2	116.1 (3)	C14—C13—C12	113.7 (3)
C6—C1—C7	119.1 (3)	C14—C13—H13A	108.8
C2—C1—C7	124.8 (3)	C12—C13—H13A	108.8
O1—C2—C3	119.7 (3)	C14—C13—H13B	108.8
O1—C2—C1	118.4 (3)	C12—C13—H13B	108.8
C3—C2—C1	121.9 (3)	H13A—C13—H13B	107.7
C2—C3—C4	119.6 (3)	C13—C14—C15	115.1 (3)
C2—C3—H3	120.2	C13—C14—H14A	108.5
C4—C3—H3	120.2	C15—C14—H14A	108.5
C5—C4—C3	120.4 (4)	C13—C14—H14B	108.5
C5—C4—H4	119.8	C15—C14—H14B	108.5
C3—C4—H4	119.8	H14A—C14—H14B	107.5
C4—C5—C6	120.4 (3)	C16—C15—C14	113.6 (3)
C4—C5—H5	119.8	C16—C15—H15A	108.9
C6—C5—H5	119.8	C14—C15—H15A	108.9
O2—C6—C5	117.6 (3)	C16—C15—H15B	108.9
O2—C6—C1	120.9 (3)	C14—C15—H15B	108.9

C5—C6—C1	121.5 (3)	H15A—C15—H15B	107.7
O3—C7—C1	119.7 (3)	C17—C16—C15	114.9 (3)
O3—C7—C8	117.9 (3)	C17—C16—H16A	108.5
C1—C7—C8	122.4 (3)	C15—C16—H16A	108.5
C7—C8—C9	114.9 (3)	C17—C16—H16B	108.5
C7—C8—H8A	108.6	C15—C16—H16B	108.5
C9—C8—H8A	108.6	H16A—C16—H16B	107.5
C7—C8—H8B	108.6	C16—C17—C18	113.2 (3)
C9—C8—H8B	108.6	C16—C17—H17A	108.9
H8A—C8—H8B	107.5	C18—C17—H17A	108.9
C10—C9—C8	111.0 (3)	C16—C17—H17B	108.9
C10—C9—H9A	109.4	C18—C17—H17B	108.9
C8—C9—H9A	109.4	H17A—C17—H17B	107.7
C10—C9—H9B	109.4	C19—C18—C17	115.1 (3)
C8—C9—H9B	109.4	C19—C18—H18A	108.5
H9A—C9—H9B	108.0	C17—C18—H18A	108.5
C11—C10—C9	114.8 (3)	C19—C18—H18B	108.5
C11—C10—H10A	108.6	C17—C18—H18B	108.5
C9—C10—H10A	108.6	H18A—C18—H18B	107.5
C11—C10—H10B	108.6	C20—C19—C18	113.8 (3)
C9—C10—H10B	108.6	C20—C19—H19A	108.8
H10A—C10—H10B	107.5	C18—C19—H19A	108.8
C10—C11—C12	113.3 (3)	C20—C19—H19B	108.8
C10—C11—H11A	108.9	C18—C19—H19B	108.8
C12—C11—H11A	108.9	H19A—C19—H19B	107.7
C10—C11—H11B	108.9	C19—C20—H20A	109.5
C12—C11—H11B	108.9	C19—C20—H20B	109.5
H11A—C11—H11B	107.7	H20A—C20—H20B	109.5
C13—C12—C11	115.2 (3)	C19—C20—H20C	109.5
C13—C12—H12A	108.5	H20A—C20—H20C	109.5
C11—C12—H12A	108.5	H20B—C20—H20C	109.5
C13—C12—H12B	108.5	C2—O1—H1O	110 (3)
C11—C12—H12B	108.5	C6—O2—H2O	111 (3)
H12A—C12—H12B	107.5		

C6—C1—C2—O1	−177.2 (3)	C6—C1—C7—C8	−175.5 (3)
C7—C1—C2—O1	4.1 (5)	C2—C1—C7—C8	3.2 (5)
C6—C1—C2—C3	1.8 (5)	O3—C7—C8—C9	−8.9 (5)
C7—C1—C2—C3	−176.9 (3)	C1—C7—C8—C9	170.0 (3)
O1—C2—C3—C4	178.3 (3)	C7—C8—C9—C10	−178.5 (3)
C1—C2—C3—C4	−0.7 (5)	C8—C9—C10—C11	175.8 (3)
C2—C3—C4—C5	−0.4 (6)	C9—C10—C11—C12	179.9 (3)
C3—C4—C5—C6	0.2 (6)	C10—C11—C12—C13	177.6 (3)
C4—C5—C6—O2	−178.6 (4)	C11—C12—C13—C14	179.0 (3)
C4—C5—C6—C1	1.0 (6)	C12—C13—C14—C15	179.0 (3)
C2—C1—C6—O2	177.7 (3)	C13—C14—C15—C16	178.4 (3)
C7—C1—C6—O2	−3.6 (5)	C14—C15—C16—C17	−179.9 (3)
C2—C1—C6—C5	−2.0 (5)	C15—C16—C17—C18	179.0 (3)

C7—C1—C6—C5	176.8 (3)	C16—C17—C18—C19	−179.5 (3)
C6—C1—C7—O3	3.4 (5)	C17—C18—C19—C20	179.4 (3)
C2—C1—C7—O3	−177.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O3	0.84 (2)	1.75 (3)	2.485 (4)	146 (4)
O1—H1*O*···O2^{i}	0.84 (2)	1.94 (2)	2.760 (3)	168 (4)

Symmetry code: (i) x, $-y+3/2$, $z+1/2$.