Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of 10a-hydroxy-9-(3-nitrophenyl)-3,6-diphenyl-3,4,5,6,7,8a,9,10a-octahydro-1*H*-xanthene-1,8(2*H*)-dione

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Received 28 October 2015
Accepted 9 November 2015

Edited by D.-J. Xu, Zhejiang University (Yuquan Campus), China

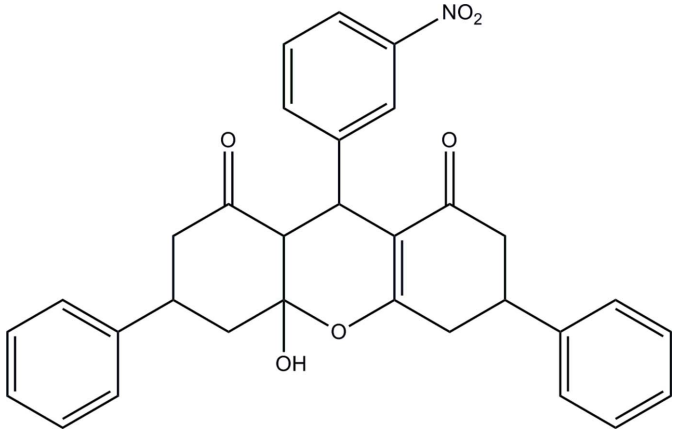
Keywords: crystal structure; xanthene; hydrogen bonding

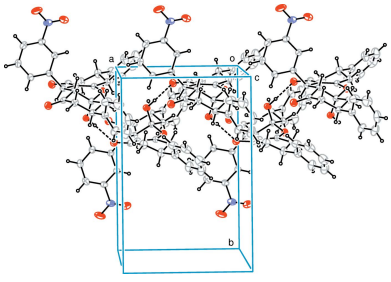
CCDC reference: 1435921
Supporting information: this article has supporting information at journals.iucr.org/e

In the octahydroxanthenedione unit of the title compound, $C_{31}H_{27}NO_6$, the central dihydropyran ring shows an envelope conformation, while the bilateral cyclohexene and cyclohexane rings adopt a half-boat conformation and a chair conformation, respectively. The nitrobenzene ring is twisted with respect to the two benzene rings, making dihedral angles of 63.1 (1) and 63.0 (1)°. In the crystal, O—H···O hydrogen bonds link the molecules into supramolecular chains propagating along the *a*-axis direction.

1. Chemical context

Xanthenes are important biologically active heterocyclic compounds, which possess anti-inflammatory, antibacterial and antiviral activities (Shakibaei *et al.*, 2007; Lambert *et al.*, 1997). Many studies have been carried out on xanthene derivatives (Knight & Little, 2001; Jha & Beal, 2004; Lu *et al.*, 2011; Cui *et al.*, 2012; Wang *et al.*, 2015). Herein, we report the synthesis and the crystal structure of the title xanthene derivative.





2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. The C1—O1 and C15—O2 bond lengths are 1.234 (4) and 1.202 (4) Å, respectively. The central dihydropyran ring shows an envelope conformation with atom C19 as the flap, while the bilateral cyclohexene and cyclohexane rings adopt a half boat conformation and a chair conformation, respectively. The nitrobenzene ring is twisted with respect to the C7–C10 and C20–C25 benzene rings, making dihedral angles of 63.1 (1) and 63.0 (1)°, respectively.

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4*C*···O1^{i}	0.82	1.89	2.714 (3)	180

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

3. Supramolecular features

In the crystal (Fig. 2), the molecules are linked by O—H···O hydrogen bonds (Table 1), generating supramolecular chains propagating along the *a*-axis direction.

4. Database survey

A search of the Cambridge Structural Database for 10a-hydroxy-3,4,5,6,7,8a,9,10a-octahydro-1*H*-xanthene-1,8(2*H*)-dione gave 16 hits. None of them are substituted at the 3,6-position with two phenyl groups. Several compounds substituted at the 9-position with an aryl group are similar to the title compound, for example, 9-(2,6-dichlorophenyl)-4a-hydroxy-3,3,6,6-tetramethyl-1,2,3,4,4a,5,6,7,8,9a-decahydroxanthene-1,8-dione (Bolte *et al.*, 2001), 9-(2,3-dichlorophenyl)-4a-hydroxy-3,3,6,6-tetramethyl-3,4,4a,6,7,9,9a,10-octahydroanthracene-1,8(2*H*,5*H*)-dione (Mohammadi Ziarani *et al.*, 2008) and 9-(2-chlorophenyl)-4a-hydroxy-3,4,4a,5,6,7,9,9a-octahydro-1*H*-xanthracene-1,8(2*H*)-dione (Liu *et al.*, 2014).

5. Synthesis and crystallization

The title compound was synthesized in accordance to our previous procedure (Wang *et al.*, 2015). 5-Phenylcyclohexane-1,3-dione (7.52 g, 40 mmol) and 3-nitrobenzaldehyde (20 mmol) were dissolved in the mixture of methanol (10 ml) and ethanol (10 ml) in the presence of trace L-proline (5 mmol) and stirred for 4 h. After completion of the reaction, the white solid products were filtered under reduced pressure and washed with ethanol (78% yield). m.p. 445.15–447.15 K. IR (KBr pellets, cm^{-1}): 3370 (O—H), 1648 (C=O), 1562 (C=C). MS (ESI) *m*/*z*: 510.2 [M + H^{+}]. ^{1}H NMR (DMSO-d_6, 400 MHz): δ 2.56–2.92 (*m*, 8H, 2a-H, 7a-H, 2b-H, 7b-H, 4a-H, 5a-H, 4b-H, 5b-H); 3.43 (*m*, 2H, 6-H, 3-H); 5.46 (*m*, 1H, 9-H); 7.14 (*m*, 2H, 11-H, 10-OH); 7.22–8.02 (*m*, 14H, PhH). Analysis calculated for $C_{31}H_{27}NO_6$: C 73.07, H 5.34, N 2.75%; found: C 72.92, H 5.30, N 2.65%. Single crystals of the title compound were obtained by slow evaporation from an ethanol solution at room temperature in the form of colorless blocks.

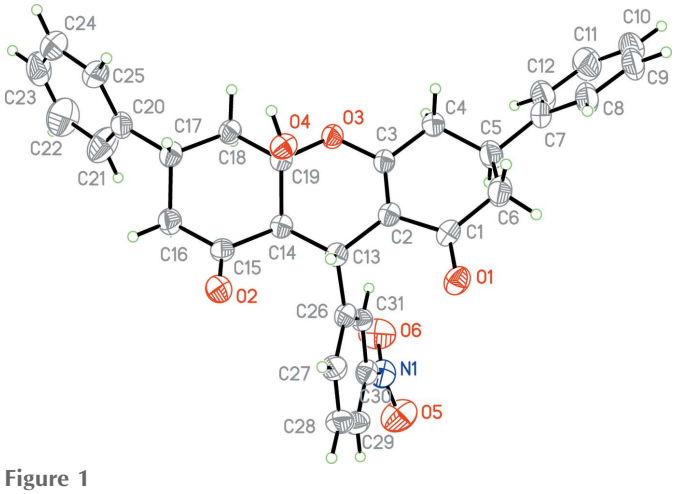


Figure 1
The molecular structure of the title compound, showing the atom labelling, with displacement ellipsoids drawn at the 50% probability level.

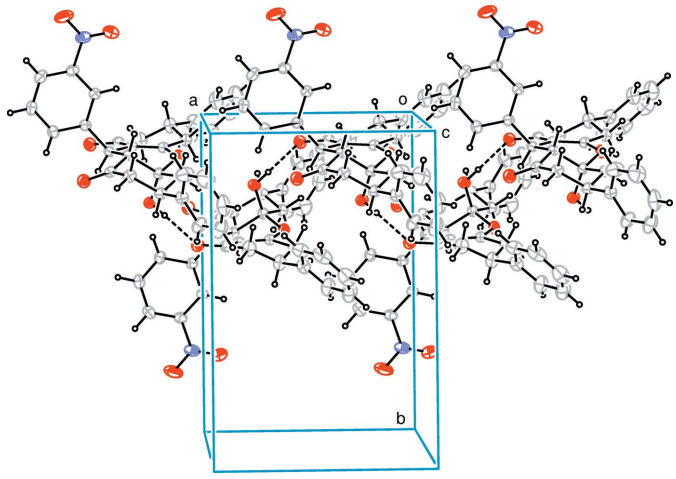


Figure 2
Packing diagram showing the hydrogen bonds as dashed lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{31}H_{27}NO_6$
M_r	509.53
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	291
a, b, c (Å)	8.973 (4), 13.520 (6), 21.251 (9)
V (Å^3)	2578 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.25 × 0.20 × 0.16
Data collection	
Diffractometer	Bruker SMART APEXII area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2000)
T_{min}, T_{max}	0.979, 0.986
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19805, 5040, 3978
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.125, 1.03
No. of reflections	5040
No. of parameters	344
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.43, −0.21

Computer programs: *APEX2* and *SAINT* (Bruker, 2000), *SHELXS2014* (Sheldrick, 2008) and *SHELXL2014* (Sheldrick, 2015).

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms of were fixed geometrically and treated as riding with C—H = 0.97 (methylene), 0.98 (methine), 0.93 (phenyl) and O—H = 0.82 Å, with U_{iso}(H) = 1.2U_{eq}(C,O).

Acknowledgements

This work was supported by the Jiangsu Innovation Program for Undergraduate and Graduate Students (No. KYZZ15-0300) and the Innovation Program for Undergraduate and Graduate Students from Jiangsu University of Science and Technology, China.

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supporting information

Acta Cryst. (2015). E**71**, 1510-1512 [https://doi.org/10.1107/S2056989015021246]

Crystal structure of 10a-hydroxy-9-(3-nitrophenyl)-3,6-diphenyl-3,4,5,6,7,8a,9,10a-octahydro-1*H*-xanthene-1,8(2*H*)-dione

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Computing details

Data collection: *APEX2* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS2014* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXL2014* (Sheldrick, 2015); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015).

10a-Hydroxy-9-(3-nitrophenyl)-3,6-diphenyl-3,4,5,6,7,8a,9,10a-octahydro-1*H*-xanthene-1,8(2*H*)-dione

Crystal data

$C_{31}H_{27}NO_6$
$M_r = 509.53$
Orthorhombic, $P2_12_12_1$
$a = 8.973$ (4) Å
$b = 13.520$ (6) Å
$c = 21.251$ (9) Å
$V = 2578$ (2) Å^3
$Z = 4$
$F(000) = 1072$
$D_x = 1.313$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7999 reflections
$\theta = 2.5$–25.5°
$\mu = 0.09$ mm^{-1}
$T = 291$ K
Block, colorless
0.25 × 0.20 × 0.16 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
$T_{min} = 0.979$, $T_{max} = 0.986$
19805 measured reflections
5040 independent reflections
3978 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.4°$
$h = -10\rightarrow11$
$k = -16\rightarrow16$
$l = -26\rightarrow26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.125$
$S = 1.03$
5040 reflections
344 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.3283P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.010$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Absolute structure: Flack *x* determined using 1503 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 1.3 (4)

Special details

Experimental. Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)
6.7298 (0.0116) x + 5.2391 (0.0295) y + 11.3904 (0.0347) z = 4.9647 (0.0106)
* -0.0122 (0.0031) C20 * 0.0030 (0.0039) C21 * 0.0057 (0.0040) C22 * -0.0052 (0.0037) C23 * -0.0042 (0.0035) C24 * 0.0128 (0.0031) C25
Rms deviation of fitted atoms = 0.0081
5.3891 (0.0148) x - 9.6057 (0.0212) y - 7.7932 (0.0389) z = 1.2144 (0.0092)
Angle to previous plane (with approximate esd) = 88.773 (14.5)
* 0.0060 (0.0029) C7 * -0.0023 (0.0029) C8 * -0.0024 (0.0031) C9 * 0.0034 (0.0036) C10 * 0.0004 (0.0038) C11 * -0.0052 (0.0034) C12
Rms deviation of fitted atoms = 0.0038
- 1.0457 (0.0125) x + 0.3544 (0.0170) y + 21.0986 (0.0100) z = 0.3439 (0.0082)
Angle to previous plane (with approximate esd) = 63.082 (14.2)
* 0.0003 (0.0022) C26 * -0.0038 (0.0023) C27 * 0.0040 (0.0025) C28 * -0.0005 (0.0025) C29 * -0.0030 (0.0022) C30 * 0.0030 (0.0020) C31
Rms deviation of fitted atoms = 0.0028

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3896 (4)	0.0908 (2)	−0.07887 (14)	0.0532 (7)
C2	0.3277 (3)	0.09893 (19)	−0.01590 (13)	0.0444 (6)
C3	0.1786 (3)	0.1037 (2)	−0.00994 (13)	0.0448 (6)
C4	0.0696 (3)	0.0842 (2)	−0.06132 (15)	0.0537 (7)
H4A	0.0206	0.1457	−0.0725	0.064*
H4B	−0.0061	0.0390	−0.0460	0.064*
C5	0.1411 (4)	0.0405 (3)	−0.11952 (16)	0.0699 (10)
H5A	0.1651	−0.0280	−0.1084	0.084*
C6	0.2850 (4)	0.0872 (3)	−0.13382 (17)	0.0749 (10)
H6A	0.2672	0.1541	−0.1484	0.090*
H6B	0.3323	0.0511	−0.1678	0.090*
C7	0.0334 (4)	0.0332 (3)	−0.17443 (15)	0.0657 (9)
C8	0.0491 (5)	0.0871 (3)	−0.22896 (16)	0.0795 (12)
H8A	0.1254	0.1333	−0.2324	0.095*
C9	−0.0483 (8)	0.0729 (4)	−0.27876 (19)	0.1001 (17)
H9A	−0.0365	0.1097	−0.3154	0.120*
C10	−0.1603 (7)	0.0061 (5)	−0.2747 (2)	0.109 (2)
H10A	−0.2248	−0.0033	−0.3084	0.131*
C11	−0.1778 (6)	−0.0470 (5)	−0.2208 (3)	0.1080 (17)
H11A	−0.2546	−0.0929	−0.2176	0.130*
C12	−0.0816 (5)	−0.0329 (4)	−0.1710 (2)	0.0873 (13)
H12A	−0.0952	−0.0692	−0.1342	0.105*
C13	0.4348 (3)	0.1112 (2)	0.03895 (13)	0.0448 (6)
H13A	0.4897	0.1731	0.0329	0.054*
C14	0.3463 (3)	0.1195 (2)	0.10028 (13)	0.0454 (6)

H14A	0.3183	0.0525	0.1133	0.054*
C15	0.4323 (4)	0.1669 (2)	0.15441 (14)	0.0502 (7)
C16	0.3445 (4)	0.1767 (3)	0.21452 (15)	0.0594 (8)
H16A	0.4040	0.2117	0.2455	0.071*
H16B	0.3230	0.1114	0.2310	0.071*
C17	0.1982 (4)	0.2325 (2)	0.20407 (14)	0.0531 (7)
H17A	0.2239	0.2996	0.1905	0.064*
C18	0.1095 (4)	0.1841 (2)	0.15049 (13)	0.0500 (7)
H18A	0.0795	0.1180	0.1629	0.060*
H18B	0.0202	0.2223	0.1422	0.060*
C19	0.2036 (3)	0.1785 (2)	0.09110 (13)	0.0477 (7)
C20	0.1030 (4)	0.2418 (3)	0.26275 (14)	0.0583 (8)
C21	0.0884 (7)	0.1681 (4)	0.3066 (2)	0.0987 (15)
H21A	0.1430	0.1101	0.3018	0.118*
C22	−0.0045 (7)	0.1777 (5)	0.3573 (2)	0.1152 (18)
H22A	−0.0120	0.1263	0.3862	0.138*
C23	−0.0851 (5)	0.2603 (6)	0.3660 (2)	0.1031 (18)
H23A	−0.1489	0.2661	0.4003	0.124*
C24	−0.0718 (6)	0.3356 (5)	0.3235 (2)	0.1023 (17)
H24A	−0.1272	0.3931	0.3289	0.123*
C25	0.0237 (5)	0.3272 (3)	0.27247 (18)	0.0790 (11)
H25A	0.0341	0.3798	0.2446	0.095*
C26	0.5470 (3)	0.0276 (2)	0.04296 (13)	0.0469 (7)
C27	0.6988 (3)	0.0467 (3)	0.04997 (15)	0.0556 (8)
H27A	0.7324	0.1117	0.0502	0.067*
C28	0.7995 (4)	−0.0294 (3)	0.05661 (17)	0.0660 (9)
H28A	0.9002	−0.0150	0.0617	0.079*
C29	0.7535 (4)	−0.1262 (3)	0.05574 (17)	0.0654 (9)
H29A	0.8214	−0.1777	0.0599	0.079*
C30	0.6046 (4)	−0.1444 (2)	0.04855 (14)	0.0538 (7)
C31	0.4998 (3)	−0.0703 (2)	0.04240 (14)	0.0484 (7)
H31A	0.3993	−0.0856	0.0379	0.058*
N1	0.5518 (4)	−0.2479 (2)	0.04813 (15)	0.0718 (8)
O1	0.5249 (2)	0.09640 (17)	−0.08882 (11)	0.0611 (6)
O2	0.5583 (3)	0.19545 (18)	0.14823 (11)	0.0635 (6)
O3	0.1096 (2)	0.12872 (16)	0.04468 (9)	0.0519 (5)
O4	0.2463 (2)	0.27061 (15)	0.06846 (10)	0.0537 (5)
H4C	0.1792	0.3106	0.0746	0.081*
O5	0.6420 (4)	−0.3133 (2)	0.03810 (18)	0.1123 (12)
O6	0.4215 (4)	−0.2637 (2)	0.0582 (2)	0.1056 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0563 (18)	0.0513 (17)	0.0522 (17)	−0.0012 (15)	0.0112 (15)	−0.0060 (14)
C2	0.0519 (17)	0.0372 (14)	0.0441 (14)	0.0000 (12)	0.0036 (13)	0.0003 (12)
C3	0.0515 (16)	0.0427 (15)	0.0402 (14)	−0.0003 (12)	0.0023 (12)	−0.0001 (12)
C4	0.0528 (17)	0.0608 (18)	0.0476 (16)	0.0010 (15)	−0.0005 (13)	−0.0023 (14)

C5	0.064 (2)	0.098 (3)	0.0475 (18)	−0.0060 (19)	−0.0007 (16)	0.0034 (18)
C6	0.075 (2)	0.098 (3)	0.0523 (19)	−0.001 (2)	0.0060 (17)	−0.0111 (19)
C7	0.072 (2)	0.085 (2)	0.0401 (16)	0.014 (2)	0.0003 (16)	−0.0048 (17)
C8	0.116 (3)	0.076 (2)	0.0467 (19)	0.015 (2)	−0.009 (2)	−0.0058 (17)
C9	0.152 (5)	0.103 (3)	0.046 (2)	0.049 (4)	−0.020 (3)	−0.011 (2)
C10	0.122 (4)	0.138 (5)	0.067 (3)	0.039 (4)	−0.037 (3)	−0.037 (3)
C11	0.087 (3)	0.148 (5)	0.088 (3)	−0.010 (3)	−0.019 (3)	−0.028 (3)
C12	0.076 (3)	0.126 (4)	0.060 (2)	−0.011 (3)	−0.008 (2)	0.000 (2)
C13	0.0469 (15)	0.0406 (14)	0.0468 (15)	−0.0042 (12)	0.0022 (12)	−0.0013 (12)
C14	0.0508 (16)	0.0393 (14)	0.0461 (15)	−0.0014 (13)	0.0015 (12)	0.0004 (12)
C15	0.0555 (19)	0.0452 (16)	0.0499 (16)	0.0099 (15)	−0.0032 (14)	0.0003 (13)
C16	0.068 (2)	0.0623 (19)	0.0475 (17)	0.0058 (17)	−0.0025 (15)	−0.0006 (15)
C17	0.0623 (19)	0.0505 (16)	0.0464 (16)	0.0015 (15)	0.0059 (14)	−0.0012 (13)
C18	0.0537 (16)	0.0511 (16)	0.0451 (15)	0.0001 (14)	0.0066 (14)	0.0009 (13)
C19	0.0478 (15)	0.0505 (16)	0.0448 (15)	0.0019 (13)	0.0008 (12)	−0.0022 (13)
C20	0.0622 (19)	0.068 (2)	0.0450 (16)	−0.0021 (18)	−0.0014 (15)	−0.0082 (15)
C21	0.136 (4)	0.082 (3)	0.078 (3)	0.008 (3)	0.043 (3)	0.011 (2)
C22	0.132 (5)	0.132 (5)	0.082 (3)	−0.014 (4)	0.038 (3)	0.015 (3)
C23	0.068 (3)	0.183 (6)	0.058 (3)	−0.013 (3)	0.014 (2)	−0.030 (3)
C24	0.085 (3)	0.144 (5)	0.078 (3)	0.033 (3)	0.006 (3)	−0.032 (3)
C25	0.081 (3)	0.096 (3)	0.060 (2)	0.025 (2)	0.0047 (19)	−0.009 (2)
C26	0.0509 (16)	0.0467 (15)	0.0430 (14)	−0.0013 (13)	0.0070 (13)	0.0006 (12)
C27	0.0506 (17)	0.0625 (19)	0.0538 (18)	−0.0055 (15)	0.0042 (14)	0.0030 (15)
C28	0.0467 (17)	0.083 (2)	0.068 (2)	0.0033 (17)	0.0045 (16)	0.0026 (18)
C29	0.063 (2)	0.069 (2)	0.064 (2)	0.0190 (18)	0.0052 (17)	0.0072 (16)
C30	0.067 (2)	0.0470 (16)	0.0469 (15)	0.0042 (14)	0.0035 (15)	0.0039 (13)
C31	0.0508 (16)	0.0501 (16)	0.0444 (14)	0.0012 (13)	0.0013 (13)	0.0014 (12)
N1	0.095 (2)	0.0502 (17)	0.0700 (18)	0.0082 (18)	0.0015 (18)	0.0109 (14)
O1	0.0579 (13)	0.0663 (14)	0.0591 (13)	−0.0065 (11)	0.0139 (10)	−0.0047 (10)
O2	0.0595 (14)	0.0683 (15)	0.0627 (14)	−0.0025 (12)	−0.0050 (11)	−0.0077 (11)
O3	0.0455 (10)	0.0657 (13)	0.0444 (10)	−0.0024 (10)	0.0029 (9)	−0.0066 (9)
O4	0.0587 (12)	0.0471 (11)	0.0554 (12)	0.0045 (10)	0.0076 (10)	0.0075 (9)
O5	0.139 (3)	0.0557 (16)	0.142 (3)	0.0285 (18)	0.020 (2)	0.0107 (18)
O6	0.097 (2)	0.0608 (16)	0.159 (3)	−0.0136 (16)	−0.007 (2)	0.0041 (18)

Geometric parameters (Å, º)

C1—O1	1.234 (4)	C16—H16A	0.9700
C1—C2	1.453 (4)	C16—H16B	0.9700
C1—C6	1.499 (5)	C17—C20	1.517 (4)
C2—C3	1.345 (4)	C17—C18	1.536 (4)
C2—C13	1.520 (4)	C17—H17A	0.9800
C3—O3	1.358 (3)	C18—C19	1.520 (4)
C3—C4	1.489 (4)	C18—H18A	0.9700
C4—C5	1.513 (5)	C18—H18B	0.9700
C4—H4A	0.9700	C19—O4	1.389 (4)
C4—H4B	0.9700	C19—O3	1.462 (4)
C5—C6	1.468 (5)	C20—C21	1.371 (5)

C5—C7	1.519 (5)	C20—C25	1.371 (5)
C5—H5A	0.9800	C21—C22	1.369 (7)
C6—H6A	0.9700	C21—H21A	0.9300
C6—H6B	0.9700	C22—C23	1.343 (8)
C7—C12	1.367 (6)	C22—H22A	0.9300
C7—C8	1.376 (5)	C23—C24	1.365 (9)
C8—C9	1.385 (6)	C23—H23A	0.9300
C8—H8A	0.9300	C24—C25	1.387 (6)
C9—C10	1.354 (8)	C24—H24A	0.9300
C9—H9A	0.9300	C25—H25A	0.9300
C10—C11	1.360 (8)	C26—C31	1.390 (4)
C10—H10A	0.9300	C26—C27	1.394 (4)
C11—C12	1.380 (6)	C27—C28	1.377 (5)
C11—H11A	0.9300	C27—H27A	0.9300
C12—H12A	0.9300	C28—C29	1.373 (6)
C13—C26	1.515 (4)	C28—H28A	0.9300
C13—C14	1.530 (4)	C29—C30	1.366 (5)
C13—H13A	0.9800	C29—H29A	0.9300
C14—C19	1.521 (4)	C30—C31	1.380 (4)
C14—C15	1.527 (4)	C30—N1	1.477 (5)
C14—H14A	0.9800	C31—H31A	0.9300
C15—O2	1.202 (4)	N1—O6	1.207 (4)
C15—C16	1.507 (5)	N1—O5	1.218 (4)
C16—C17	1.530 (5)	O4—H4C	0.8200

O1—C1—C2	121.9 (3)	C15—C16—H16B	109.3
O1—C1—C6	119.0 (3)	C17—C16—H16B	109.3
C2—C1—C6	118.7 (3)	H16A—C16—H16B	108.0
C3—C2—C1	118.1 (3)	C20—C17—C16	113.9 (3)
C3—C2—C13	123.5 (3)	C20—C17—C18	110.7 (3)
C1—C2—C13	118.2 (3)	C16—C17—C18	110.0 (3)
C2—C3—O3	123.1 (3)	C20—C17—H17A	107.3
C2—C3—C4	125.1 (3)	C16—C17—H17A	107.3
O3—C3—C4	111.8 (2)	C18—C17—H17A	107.3
C3—C4—C5	112.9 (3)	C19—C18—C17	110.4 (2)
C3—C4—H4A	109.0	C19—C18—H18A	109.6
C5—C4—H4A	109.0	C17—C18—H18A	109.6
C3—C4—H4B	109.0	C19—C18—H18B	109.6
C5—C4—H4B	109.0	C17—C18—H18B	109.6
H4A—C4—H4B	107.8	H18A—C18—H18B	108.1
C6—C5—C4	112.0 (3)	O4—C19—O3	109.8 (2)
C6—C5—C7	115.4 (3)	O4—C19—C18	113.4 (2)
C4—C5—C7	112.5 (3)	O3—C19—C18	105.2 (2)
C6—C5—H5A	105.3	O4—C19—C14	106.4 (2)
C4—C5—H5A	105.3	O3—C19—C14	109.3 (2)
C7—C5—H5A	105.3	C18—C19—C14	112.8 (2)
C5—C6—C1	113.8 (3)	C21—C20—C25	117.4 (3)
C5—C6—H6A	108.8	C21—C20—C17	123.5 (3)

C1—C6—H6A	108.8	C25—C20—C17	119.1 (3)
C5—C6—H6B	108.8	C22—C21—C20	121.6 (5)
C1—C6—H6B	108.8	C22—C21—H21A	119.2
H6A—C6—H6B	107.7	C20—C21—H21A	119.2
C12—C7—C8	118.0 (4)	C23—C22—C21	121.0 (5)
C12—C7—C5	118.8 (3)	C23—C22—H22A	119.5
C8—C7—C5	123.2 (4)	C21—C22—H22A	119.5
C7—C8—C9	120.4 (5)	C22—C23—C24	118.8 (4)
C7—C8—H8A	119.8	C22—C23—H23A	120.6
C9—C8—H8A	119.8	C24—C23—H23A	120.6
C10—C9—C8	120.8 (5)	C23—C24—C25	120.7 (5)
C10—C9—H9A	119.6	C23—C24—H24A	119.7
C8—C9—H9A	119.6	C25—C24—H24A	119.7
C9—C10—C11	119.4 (5)	C20—C25—C24	120.4 (5)
C9—C10—H10A	120.3	C20—C25—H25A	119.8
C11—C10—H10A	120.3	C24—C25—H25A	119.8
C10—C11—C12	120.1 (6)	C31—C26—C27	118.3 (3)
C10—C11—H11A	120.0	C31—C26—C13	120.5 (3)
C12—C11—H11A	120.0	C27—C26—C13	121.2 (3)
C7—C12—C11	121.4 (5)	C28—C27—C26	121.0 (3)
C7—C12—H12A	119.3	C28—C27—H27A	119.5
C11—C12—H12A	119.3	C26—C27—H27A	119.5
C26—C13—C2	112.5 (2)	C29—C28—C27	120.9 (3)
C26—C13—C14	110.6 (2)	C29—C28—H28A	119.6
C2—C13—C14	109.4 (2)	C27—C28—H28A	119.6
C26—C13—H13A	108.1	C30—C29—C28	117.9 (3)
C2—C13—H13A	108.1	C30—C29—H29A	121.1
C14—C13—H13A	108.1	C28—C29—H29A	121.1
C19—C14—C15	107.6 (2)	C29—C30—C31	123.0 (3)
C19—C14—C13	111.5 (2)	C29—C30—N1	119.0 (3)
C15—C14—C13	114.2 (2)	C31—C30—N1	117.9 (3)
C19—C14—H14A	107.8	C30—C31—C26	118.9 (3)
C15—C14—H14A	107.8	C30—C31—H31A	120.5
C13—C14—H14A	107.8	C26—C31—H31A	120.5
O2—C15—C16	123.8 (3)	O6—N1—O5	123.1 (4)
O2—C15—C14	121.9 (3)	O6—N1—C30	118.5 (3)
C16—C15—C14	114.3 (3)	O5—N1—C30	118.4 (4)
C15—C16—C17	111.7 (3)	C3—O3—C19	115.4 (2)
C15—C16—H16A	109.3	C19—O4—H4C	109.5
C17—C16—H16A	109.3		

O1—C1—C2—C3	172.3 (3)	C16—C17—C18—C19	−55.4 (3)
C6—C1—C2—C3	−0.5 (4)	C17—C18—C19—O4	−61.8 (3)
O1—C1—C2—C13	−2.1 (4)	C17—C18—C19—O3	178.2 (2)
C6—C1—C2—C13	−175.0 (3)	C17—C18—C19—C14	59.2 (3)
C1—C2—C3—O3	−168.0 (2)	C15—C14—C19—O4	68.2 (3)
C13—C2—C3—O3	6.1 (4)	C13—C14—C19—O4	−57.8 (3)
C1—C2—C3—C4	11.1 (4)	C15—C14—C19—O3	−173.4 (2)

C13—C2—C3—C4	−174.8 (3)	C13—C14—C19—O3	60.6 (3)
C2—C3—C4—C5	10.0 (4)	C15—C14—C19—C18	−56.7 (3)
O3—C3—C4—C5	−170.8 (3)	C13—C14—C19—C18	177.3 (2)
C3—C4—C5—C6	−40.7 (4)	C16—C17—C20—C21	−38.2 (5)
C3—C4—C5—C7	−172.6 (3)	C18—C17—C20—C21	86.3 (5)
C4—C5—C6—C1	51.0 (5)	C16—C17—C20—C25	143.3 (3)
C7—C5—C6—C1	−178.6 (3)	C18—C17—C20—C25	−92.1 (4)
O1—C1—C6—C5	155.9 (4)	C25—C20—C21—C22	1.8 (8)
C2—C1—C6—C5	−31.1 (5)	C17—C20—C21—C22	−176.7 (4)
C6—C5—C7—C12	161.1 (4)	C20—C21—C22—C23	−0.1 (9)
C4—C5—C7—C12	−68.7 (5)	C21—C22—C23—C24	−0.7 (9)
C6—C5—C7—C8	−16.1 (6)	C22—C23—C24—C25	−0.2 (8)
C4—C5—C7—C8	114.1 (4)	C21—C20—C25—C24	−2.7 (6)
C12—C7—C8—C9	−0.9 (6)	C17—C20—C25—C24	175.9 (4)
C5—C7—C8—C9	176.3 (4)	C23—C24—C25—C20	2.0 (7)
C7—C8—C9—C10	0.1 (6)	C2—C13—C26—C31	−51.4 (3)
C8—C9—C10—C11	0.4 (7)	C14—C13—C26—C31	71.3 (3)
C9—C10—C11—C12	−0.2 (8)	C2—C13—C26—C27	131.3 (3)
C8—C7—C12—C11	1.2 (7)	C14—C13—C26—C27	−106.0 (3)
C5—C7—C12—C11	−176.2 (4)	C31—C26—C27—C28	−0.4 (5)
C10—C11—C12—C7	−0.7 (8)	C13—C26—C27—C28	176.9 (3)
C3—C2—C13—C26	128.5 (3)	C26—C27—C28—C29	0.8 (5)
C1—C2—C13—C26	−57.3 (3)	C27—C28—C29—C30	−0.5 (5)
C3—C2—C13—C14	5.2 (4)	C28—C29—C30—C31	−0.2 (5)
C1—C2—C13—C14	179.3 (2)	C28—C29—C30—N1	−179.3 (3)
C26—C13—C14—C19	−162.0 (2)	C29—C30—C31—C26	0.6 (5)
C2—C13—C14—C19	−37.5 (3)	N1—C30—C31—C26	179.7 (3)
C26—C13—C14—C15	75.8 (3)	C27—C26—C31—C30	−0.2 (4)
C2—C13—C14—C15	−159.8 (2)	C13—C26—C31—C30	−177.5 (3)
C19—C14—C15—O2	−123.8 (3)	C29—C30—N1—O6	161.0 (4)
C13—C14—C15—O2	0.6 (4)	C31—C30—N1—O6	−18.2 (5)
C19—C14—C15—C16	54.7 (3)	C29—C30—N1—O5	−18.4 (5)
C13—C14—C15—C16	179.0 (2)	C31—C30—N1—O5	162.4 (3)
O2—C15—C16—C17	123.6 (3)	C2—C3—O3—C19	17.6 (4)
C14—C15—C16—C17	−54.8 (4)	C4—C3—O3—C19	−161.6 (2)
C15—C16—C17—C20	178.2 (3)	O4—C19—O3—C3	66.2 (3)
C15—C16—C17—C18	53.3 (4)	C18—C19—O3—C3	−171.5 (2)
C20—C17—C18—C19	177.8 (3)	C14—C19—O3—C3	−50.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4*C*···O1^{i}	0.82	1.89	2.714 (3)	180

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