

# 9-(4-Formylphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione

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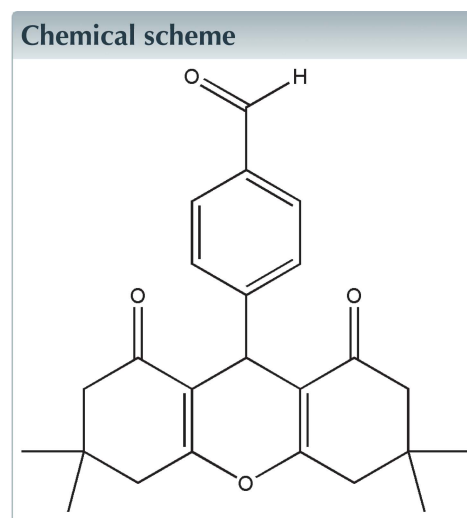
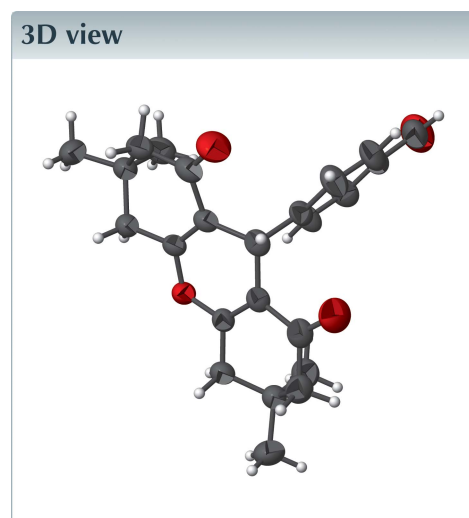
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Structural data: full structural data are available from [iucrdata.iucr.org](http://iucrdata.iucr.org)

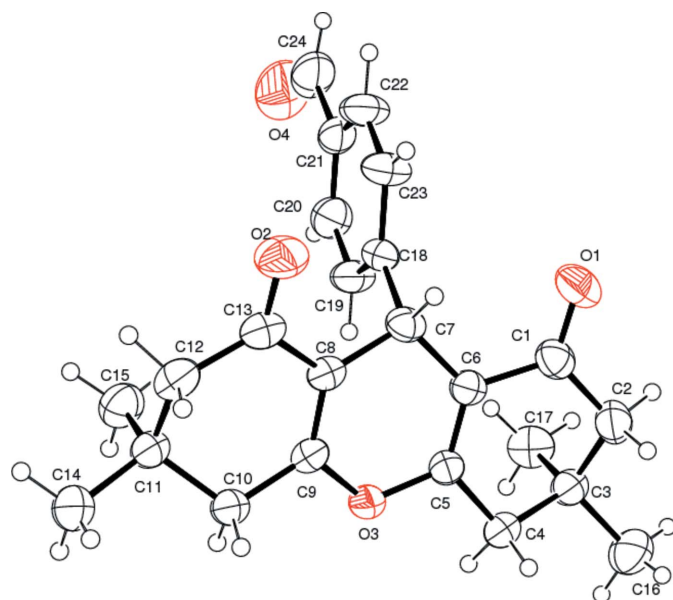
In the title compound, C<sub>24</sub>H<sub>26</sub>O<sub>4</sub>, the central 4*H*-pyran ring adopts a flattened boat conformation, with the mean and maximum deviations of the ring being 0.0582 (6) and 0.1012 (3) Å, respectively. The two cyclohexenone rings on opposite sides of the pyran ring each adopt an envelope conformation; the C atom bearing a dimethyl substituent is the flap atom in each case. The mean planes of the pyran ring and the substituent benzene ring subtend a dihedral angle of 86.45 (2)°. In the crystal, molecules are linked into inversion dimers *via* pairs of C–H···O hydrogen bonds.



## Structure description

Xanthene is the parent compound of a number of naturally occurring substances and some synthetic dyes. Xanthene derivatives (Reddy *et al.*, 2009; Mehdi *et al.*, 2011; Rizwana Begum *et al.*, 2014; Sughanya & Sureshbabu, 2012; Sureshbabu & Sughanya, 2013) are used as dyes (Hilderbrand & Weissleder, 2007), possess biological properties such as antibacterial, antiviral and anti-inflammatory (Dimmock *et al.*, 1988) activities and are used in medicine. Ehretianone, a quinonoid xanthene, has been reported to possess anti-snake venom activity (Selvanayagam *et al.*, 1996; Lambert *et al.*, 1997; Poupelin *et al.*, 1978).

In the title molecule (Fig. 1), the central pyran ring *B* (O3/C5–C9) is almost planar with a mean deviation of 0.0582 (6) Å from the mean plane and a maximum deviation of 0.1012 (3) Å for C7. Atoms O3 and C7 are displaced out of this mean plane, which means that the ring may also be described as having a highly flattened boat conformation. The cyclohexenone rings *A* (C1–C6) and *C* (C8–C13) both adopt envelope conformations, with atoms C3 and C11 being the respective flap atoms deviating from the ring plane by



**Figure 1**  
The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

0.3256 (4) and 0.3211 (2) Å, respectively. Rings *A*, *B* and *C* show total puckering amplitudes  $Q(T)$  of 0.4611 (4), 0.1558 (2) and 0.4565 (2) Å, respectively, and the puckering parameters are  $\varphi = 116.1(2)^\circ$  and  $\theta = 54.59(13)^\circ$  for *A*,  $\varphi = 185.54(8)^\circ$  and  $\theta = 77.49(2)^\circ$  for *B*, and  $\varphi = -2.12(2)^\circ$  and  $\theta = 119.64(2)^\circ$  for *C*. The benzene substituent (C18–C23) and the pyran ring form a dihedral angle of  $86.45(2)^\circ$ .

In the crystal, the molecules are linked into inversion dimers *via* pairs of C–H···O hydrogen bonds (Table 1).

### Synthesis and crystallization

A mixture of terephaldehyde (0.402 g, 3 mmol) and 5,5-dimethylcyclohexane-1,3-dione (0.84 g, 6 mmol) was dissolved in 25 ml of ethanol. To this solution about 15 drops of concentrated hydrochloric acid were added and the content was refluxed for 30 minutes. The reaction was monitored by TLC. After completion of the reaction, the reaction mixture was poured into crushed ice and stirred well. The formed precipitate was filtered and dried. Colourless single crystals suitable for X-ray diffraction were obtained from an ethanol solution at room temperature (m.p. 481 K, yield 92%).

### Refinement

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

### Acknowledgements

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C22–H22···O2 <sup>i</sup>	0.93	2.46	3.380 (3)	171

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

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	C <sub>24</sub> H <sub>26</sub> O <sub>4</sub>
<i>M<sub>r</sub></i>	378.45
Crystal system, space group	Orthorhombic, <i>Pbcn</i>
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	15.1185 (14), 11.0273 (9), 24.141 (2)
<i>V</i> (Å <sup>3</sup> )	4024.8 (6)
<i>Z</i>	8
Radiation type	Mo <i>K</i> α
$\mu$ (mm <sup>-1</sup> )	0.08
Crystal size (mm)	0.20 × 0.20 × 0.15
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2004)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.686, 0.745
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	69441, 3540, 1947
<i>R<sub>int</sub></i>	0.099
(sin θ/λ) <sub>max</sub> (Å <sup>-1</sup> )	0.595
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.041, 0.140, 1.12
No. of reflections	3540
No. of parameters	258
H-atom treatment	H-atom parameters constrained
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	0.19, -0.16

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SIR92* (Altomare *et al.*, 1993), *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

### References

- Altomare, A., Casciarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
- Bruker (2004). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dimmock, J. R., Raghavan, S. K. & Bigam, G. E. (1988). *Eur. J. Med. Chem.* **23**, 111–117.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Hilderbrand, S. A. & Weissleder, R. (2007). *Tetrahedron Lett.* **48**, 4383–4385.
- Lambert, R. W., Martin, J. A., Merrett, J. H., Parkes, K. E. B. & Thomas, G. J. (1997). *PCT Int. Appl.* WO 9706178.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Mehdi, S. H., Sulaiman, O., Ghalib, R. M., Yeap, C. S. & Fun, H.-K. (2011). *Acta Cryst.* **E67**, o1719–o1720.
- Poupelin, J. P., Saint-Ruf, G., Foussard-Blanpin, O., Narcisse, G., Uchida- Ernouf, G. & Lacroix, R. (1978). *Eur. J. Med. Chem.* **13**, 67–71.
- Reddy, B. P., Vijayakumar, V., Narasimhamurthy, T., Suresh, J. & Lakshman, P. L. N. (2009). *Acta Cryst.* **E65**, o916.
- Rizwana Begum, S., Hema, R., Sumathi, G., Valliappan, R. & Srinivasan, N. (2014). *Acta Cryst.* **E70**, o276–o277.

Selvanayagam, Z. E., Gnanavendhan, S. G., Balakrishna, K., Rao, R. B., Sivaraman, J., Subramanian, K., Puri, R. & Puri, R. K. (1996). *J. Nat. Prod.* **59**, 664–667.

Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Sughanya, V. & Sureshbabu, N. (2012). *Acta Cryst.* **E68**, o1060.

Sureshbabu, N. & Sughanya, V. (2013). *Acta Cryst.* **E69**, o281.

## full crystallographic data

*IUCrData* (2018). 3, x180105 [https://doi.org/10.1107/S2414314618001050]

## 9-(4-Formylphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione

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### 9-(4-Formylphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione

#### Crystal data

C<sub>24</sub>H<sub>26</sub>O<sub>4</sub>

*M<sub>r</sub>* = 378.45

Orthorhombic, *Pbcn*

*a* = 15.1185 (14) Å

*b* = 11.0273 (9) Å

*c* = 24.141 (2) Å

*V* = 4024.8 (6) Å<sup>3</sup>

*Z* = 8

*F*(000) = 1616

*D<sub>x</sub>* = 1.249 Mg m<sup>-3</sup>

Melting point: 481 K

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 7686 reflections

θ = 2.3–22.5°

μ = 0.08 mm<sup>-1</sup>

*T* = 296 K

Block, colourless

0.20 × 0.20 × 0.15 mm

#### Data collection

Bruker Kappa APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω and φ scan

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2004)

*T<sub>min</sub>* = 0.686, *T<sub>max</sub>* = 0.745

69441 measured reflections

3540 independent reflections

1947 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.099

θ<sub>max</sub> = 25.0°, θ<sub>min</sub> = 2.2°

*h* = -17→17

*k* = -13→13

*l* = -28→28

#### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.041

*wR*(*F*<sup>2</sup>) = 0.140

*S* = 1.12

3540 reflections

258 parameters

0 restraints

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0505*P*)<sup>2</sup> + 1.1747*P*]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.19 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.15 e Å<sup>-3</sup>

Extinction correction: SHELXL97 (Sheldrick,  
2008), *F<sub>c</sub>*\* = *kF<sub>c</sub>*[1 + 0.001*xF<sub>c</sub>*<sup>2</sup>λ<sup>3</sup>/sin(2θ)]<sup>-1/4</sup>

Extinction coefficient: 0.0032 (4)

#### 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.69613 (17)	0.3599 (2)	0.63271 (11)	0.0520 (7)
C2	0.74775 (18)	0.4377 (2)	0.67209 (11)	0.0595 (7)
H2A	0.7802	0.4977	0.6509	0.071*
H2B	0.7907	0.3873	0.6911	0.071*
C3	0.69178 (17)	0.5031 (2)	0.71530 (11)	0.0522 (7)
C4	0.61917 (17)	0.5724 (2)	0.68500 (11)	0.0500 (7)
H4A	0.5770	0.6033	0.7118	0.060*
H4B	0.6451	0.6412	0.6659	0.060*
C5	0.57195 (16)	0.4947 (2)	0.64411 (10)	0.0422 (6)
C6	0.60483 (16)	0.3958 (2)	0.61981 (9)	0.0419 (6)
C7	0.55205 (16)	0.3201 (2)	0.57950 (10)	0.0452 (6)
H7	0.5883	0.3076	0.5463	0.054*
C8	0.47054 (16)	0.3896 (2)	0.56266 (9)	0.0420 (6)
C9	0.44183 (16)	0.4876 (2)	0.59004 (10)	0.0422 (6)
C10	0.35954 (17)	0.5563 (2)	0.57806 (10)	0.0482 (7)
H10A	0.3740	0.6264	0.5555	0.058*
H10B	0.3346	0.5854	0.6126	0.058*
C11	0.29050 (17)	0.4800 (2)	0.54792 (11)	0.0505 (7)
C12	0.33608 (19)	0.4167 (2)	0.49966 (11)	0.0558 (7)
H12A	0.2952	0.3581	0.4841	0.067*
H12B	0.3481	0.4766	0.4712	0.067*
C13	0.42085 (18)	0.3525 (2)	0.51294 (11)	0.0508 (7)
C14	0.2168 (2)	0.5632 (3)	0.52696 (14)	0.0774 (10)
H14A	0.1896	0.6036	0.5578	0.116*
H14B	0.1732	0.5158	0.5078	0.116*
H14C	0.2412	0.6223	0.5021	0.116*
C15	0.2508 (2)	0.3866 (2)	0.58760 (12)	0.0689 (8)
H15A	0.2963	0.3325	0.6001	0.103*
H15B	0.2057	0.3411	0.5688	0.103*
H15C	0.2253	0.4275	0.6189	0.103*
C16	0.7482 (2)	0.5926 (3)	0.74823 (13)	0.0811 (10)
H16A	0.7797	0.6445	0.7231	0.122*
H16B	0.7897	0.5487	0.7707	0.122*
H16C	0.7107	0.6407	0.7716	0.122*
C17	0.6509 (2)	0.4119 (2)	0.75569 (11)	0.0673 (8)
H17A	0.6168	0.4545	0.7830	0.101*
H17B	0.6971	0.3669	0.7736	0.101*
H17C	0.6131	0.3572	0.7357	0.101*
C18	0.52809 (16)	0.1958 (2)	0.60290 (10)	0.0452 (6)
C19	0.48770 (18)	0.1855 (2)	0.65403 (11)	0.0565 (7)
H19	0.4753	0.2550	0.6744	0.068*
C20	0.46557 (18)	0.0737 (2)	0.67525 (12)	0.0630 (8)
H20	0.4381	0.0682	0.7096	0.076*
C21	0.48412 (19)	-0.0305 (2)	0.64562 (13)	0.0601 (8)
C22	0.5248 (2)	-0.0208 (2)	0.59482 (14)	0.0698 (9)

H22	0.5380	-0.0904	0.5747	0.084*
C23	0.54615 (19)	0.0911 (2)	0.57368 (12)	0.0627 (8)
H23	0.5732	0.0964	0.5392	0.075*
C24	0.4585 (2)	-0.1513 (3)	0.66713 (19)	0.0884 (12)
H24	0.4779	-0.2188	0.6475	0.106*
O1	0.72885 (13)	0.27064 (18)	0.61019 (8)	0.0747 (6)
O2	0.45149 (14)	0.27458 (18)	0.48226 (8)	0.0731 (6)
O3	0.48830 (11)	0.53829 (13)	0.63329 (6)	0.0466 (5)
O4	0.4148 (2)	-0.1695 (2)	0.70769 (14)	0.1232 (11)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0489 (18)	0.0509 (15)	0.0562 (17)	0.0036 (14)	0.0095 (14)	0.0040 (14)
C2	0.0442 (16)	0.0618 (16)	0.0724 (19)	0.0003 (14)	0.0018 (15)	0.0046 (15)
C3	0.0509 (17)	0.0447 (14)	0.0610 (17)	-0.0012 (13)	-0.0088 (14)	-0.0013 (13)
C4	0.0480 (16)	0.0417 (14)	0.0602 (17)	-0.0042 (12)	-0.0051 (14)	-0.0040 (12)
C5	0.0407 (15)	0.0373 (13)	0.0486 (15)	-0.0012 (12)	-0.0001 (12)	0.0022 (12)
C6	0.0433 (16)	0.0388 (13)	0.0434 (15)	-0.0004 (12)	0.0065 (12)	0.0017 (11)
C7	0.0489 (16)	0.0431 (14)	0.0435 (15)	0.0029 (12)	0.0106 (12)	-0.0049 (11)
C8	0.0475 (16)	0.0372 (13)	0.0413 (14)	-0.0049 (12)	0.0022 (12)	-0.0031 (11)
C9	0.0475 (16)	0.0368 (13)	0.0422 (14)	-0.0059 (12)	-0.0032 (12)	-0.0015 (12)
C10	0.0500 (16)	0.0403 (13)	0.0543 (16)	0.0008 (12)	-0.0071 (13)	-0.0029 (12)
C11	0.0507 (16)	0.0407 (14)	0.0601 (17)	-0.0037 (13)	-0.0083 (14)	-0.0019 (13)
C12	0.0674 (19)	0.0478 (15)	0.0522 (16)	-0.0106 (14)	-0.0101 (15)	0.0011 (13)
C13	0.0629 (19)	0.0409 (14)	0.0485 (16)	-0.0068 (13)	0.0056 (14)	-0.0032 (13)
C14	0.067 (2)	0.0635 (18)	0.102 (2)	0.0065 (16)	-0.0347 (19)	-0.0103 (17)
C15	0.0623 (19)	0.0621 (17)	0.082 (2)	-0.0124 (16)	0.0097 (17)	-0.0029 (16)
C16	0.075 (2)	0.074 (2)	0.095 (2)	-0.0056 (18)	-0.030 (2)	-0.0115 (18)
C17	0.081 (2)	0.0639 (18)	0.0564 (18)	0.0017 (16)	-0.0012 (16)	0.0035 (15)
C18	0.0449 (15)	0.0374 (14)	0.0533 (16)	0.0044 (12)	0.0041 (13)	-0.0063 (12)
C19	0.0671 (19)	0.0387 (14)	0.0638 (18)	-0.0022 (13)	0.0168 (15)	-0.0074 (13)
C20	0.0598 (19)	0.0561 (18)	0.0730 (19)	-0.0077 (14)	0.0117 (16)	0.0043 (15)
C21	0.0541 (18)	0.0404 (15)	0.086 (2)	-0.0019 (13)	-0.0124 (16)	0.0031 (15)
C22	0.086 (2)	0.0380 (16)	0.086 (2)	0.0100 (15)	-0.0033 (19)	-0.0132 (16)
C23	0.080 (2)	0.0437 (15)	0.0648 (19)	0.0097 (15)	0.0117 (16)	-0.0118 (14)
C24	0.077 (3)	0.0518 (19)	0.136 (4)	-0.0064 (18)	-0.024 (2)	0.016 (2)
O1	0.0634 (14)	0.0746 (13)	0.0862 (15)	0.0203 (11)	0.0075 (11)	-0.0170 (11)
O2	0.0878 (16)	0.0701 (13)	0.0615 (13)	0.0051 (11)	-0.0014 (11)	-0.0249 (11)
O3	0.0460 (11)	0.0413 (9)	0.0525 (10)	0.0030 (8)	-0.0058 (9)	-0.0094 (8)
O4	0.116 (2)	0.0848 (18)	0.169 (3)	-0.0282 (16)	0.004 (2)	0.0485 (19)

*Geometric parameters (Å, °)*

C1—O1	1.228 (3)	C12—H12A	0.9700
C1—C6	1.470 (3)	C12—H12B	0.9700
C1—C2	1.500 (4)	C13—O2	1.225 (3)
C2—C3	1.524 (4)	C14—H14A	0.9600

C2—H2A	0.9700	C14—H14B	0.9600
C2—H2B	0.9700	C14—H14C	0.9600
C3—C4	1.525 (3)	C15—H15A	0.9600
C3—C16	1.527 (4)	C15—H15B	0.9600
C3—C17	1.531 (4)	C15—H15C	0.9600
C4—C5	1.489 (3)	C16—H16A	0.9600
C4—H4A	0.9700	C16—H16B	0.9600
C4—H4B	0.9700	C16—H16C	0.9600
C5—C6	1.334 (3)	C17—H17A	0.9600
C5—O3	1.378 (3)	C17—H17B	0.9600
C6—C7	1.510 (3)	C17—H17C	0.9600
C7—C8	1.507 (3)	C18—C23	1.380 (3)
C7—C18	1.526 (3)	C18—C19	1.382 (3)
C7—H7	0.9800	C19—C20	1.376 (3)
C8—C9	1.339 (3)	C19—H19	0.9300
C8—C13	1.474 (3)	C20—C21	1.382 (4)
C9—O3	1.377 (3)	C20—H20	0.9300
C9—C10	1.485 (3)	C21—C22	1.376 (4)
C10—C11	1.525 (3)	C21—C24	1.482 (4)
C10—H10A	0.9700	C22—C23	1.374 (4)
C10—H10B	0.9700	C22—H22	0.9300
C11—C12	1.523 (4)	C23—H23	0.9300
C11—C15	1.530 (3)	C24—O4	1.198 (4)
C11—C14	1.530 (3)	C24—H24	0.9300
C12—C13	1.499 (4)		
O1—C1—C6	120.0 (2)	C11—C12—H12A	108.3
O1—C1—C2	122.0 (2)	C13—C12—H12B	108.3
C6—C1—C2	117.9 (2)	C11—C12—H12B	108.3
C1—C2—C3	114.6 (2)	H12A—C12—H12B	107.4
C1—C2—H2A	108.6	O2—C13—C8	119.6 (2)
C3—C2—H2A	108.6	O2—C13—C12	121.7 (2)
C1—C2—H2B	108.6	C8—C13—C12	118.6 (2)
C3—C2—H2B	108.6	C11—C14—H14A	109.5
H2A—C2—H2B	107.6	C11—C14—H14B	109.5
C2—C3—C4	108.0 (2)	H14A—C14—H14B	109.5
C2—C3—C16	110.6 (2)	C11—C14—H14C	109.5
C4—C3—C16	109.2 (2)	H14A—C14—H14C	109.5
C2—C3—C17	110.4 (2)	H14B—C14—H14C	109.5
C4—C3—C17	110.1 (2)	C11—C15—H15A	109.5
C16—C3—C17	108.6 (2)	C11—C15—H15B	109.5
C5—C4—C3	112.0 (2)	H15A—C15—H15B	109.5
C5—C4—H4A	109.2	C11—C15—H15C	109.5
C3—C4—H4A	109.2	H15A—C15—H15C	109.5
C5—C4—H4B	109.2	H15B—C15—H15C	109.5
C3—C4—H4B	109.2	C3—C16—H16A	109.5
H4A—C4—H4B	107.9	C3—C16—H16B	109.5
C6—C5—O3	122.9 (2)	H16A—C16—H16B	109.5

C6—C5—C4	125.7 (2)	C3—C16—H16C	109.5
O3—C5—C4	111.41 (19)	H16A—C16—H16C	109.5
C5—C6—C1	118.5 (2)	H16B—C16—H16C	109.5
C5—C6—C7	122.6 (2)	C3—C17—H17A	109.5
C1—C6—C7	118.9 (2)	C3—C17—H17B	109.5
C8—C7—C6	108.96 (18)	H17A—C17—H17B	109.5
C8—C7—C18	111.3 (2)	C3—C17—H17C	109.5
C6—C7—C18	112.54 (19)	H17A—C17—H17C	109.5
C8—C7—H7	108.0	H17B—C17—H17C	109.5
C6—C7—H7	108.0	C23—C18—C19	118.3 (2)
C18—C7—H7	108.0	C23—C18—C7	121.0 (2)
C9—C8—C13	117.5 (2)	C19—C18—C7	120.6 (2)
C9—C8—C7	122.8 (2)	C20—C19—C18	120.9 (2)
C13—C8—C7	119.7 (2)	C20—C19—H19	119.5
C8—C9—O3	122.4 (2)	C18—C19—H19	119.5
C8—C9—C10	125.9 (2)	C19—C20—C21	120.2 (3)
O3—C9—C10	111.60 (19)	C19—C20—H20	119.9
C9—C10—C11	112.63 (19)	C21—C20—H20	119.9
C9—C10—H10A	109.1	C22—C21—C20	119.2 (2)
C11—C10—H10A	109.1	C22—C21—C24	120.0 (3)
C9—C10—H10B	109.1	C20—C21—C24	120.9 (3)
C11—C10—H10B	109.1	C23—C22—C21	120.4 (3)
H10A—C10—H10B	107.8	C23—C22—H22	119.8
C12—C11—C10	107.9 (2)	C21—C22—H22	119.8
C12—C11—C15	110.4 (2)	C22—C23—C18	121.0 (3)
C10—C11—C15	110.0 (2)	C22—C23—H23	119.5
C12—C11—C14	110.6 (2)	C18—C23—H23	119.5
C10—C11—C14	109.00 (19)	O4—C24—C21	125.5 (4)
C15—C11—C14	109.0 (2)	O4—C24—H24	117.2
C13—C12—C11	116.1 (2)	C21—C24—H24	117.2
C13—C12—H12A	108.3	C9—O3—C5	118.07 (18)
O1—C1—C2—C3	-152.7 (2)	C9—C10—C11—C12	-48.4 (3)
C6—C1—C2—C3	29.6 (3)	C9—C10—C11—C15	72.0 (3)
C1—C2—C3—C4	-53.1 (3)	C9—C10—C11—C14	-168.6 (2)
C1—C2—C3—C16	-172.4 (2)	C10—C11—C12—C13	48.9 (3)
C1—C2—C3—C17	67.4 (3)	C15—C11—C12—C13	-71.4 (3)
C2—C3—C4—C5	49.3 (3)	C14—C11—C12—C13	168.0 (2)
C16—C3—C4—C5	169.6 (2)	C9—C8—C13—O2	169.7 (2)
C17—C3—C4—C5	-71.4 (3)	C7—C8—C13—O2	-8.3 (3)
C3—C4—C5—C6	-24.5 (3)	C9—C8—C13—C12	-6.9 (3)
C3—C4—C5—O3	156.2 (2)	C7—C8—C13—C12	175.1 (2)
O3—C5—C6—C1	177.7 (2)	C11—C12—C13—O2	161.3 (2)
C4—C5—C6—C1	-1.5 (4)	C11—C12—C13—C8	-22.2 (3)
O3—C5—C6—C7	-2.0 (4)	C8—C7—C18—C23	109.2 (3)
C4—C5—C6—C7	178.8 (2)	C6—C7—C18—C23	-128.2 (3)
O1—C1—C6—C5	-178.6 (2)	C8—C7—C18—C19	-70.8 (3)
C2—C1—C6—C5	-0.8 (3)	C6—C7—C18—C19	51.8 (3)



O1—C1—C6—C7	1.1 (3)	C23—C18—C19—C20	-0.3 (4)
C2—C1—C6—C7	178.9 (2)	C7—C18—C19—C20	179.7 (3)
C5—C6—C7—C8	13.1 (3)	C18—C19—C20—C21	0.4 (4)
C1—C6—C7—C8	-166.6 (2)	C19—C20—C21—C22	0.0 (4)
C5—C6—C7—C18	-110.8 (2)	C19—C20—C21—C24	-178.3 (3)
C1—C6—C7—C18	69.5 (3)	C20—C21—C22—C23	-0.5 (5)
C6—C7—C8—C9	-14.6 (3)	C24—C21—C22—C23	177.8 (3)
C18—C7—C8—C9	110.1 (3)	C21—C22—C23—C18	0.6 (5)
C6—C7—C8—C13	163.2 (2)	C19—C18—C23—C22	-0.2 (4)
C18—C7—C8—C13	-72.1 (3)	C7—C18—C23—C22	179.8 (3)
C13—C8—C9—O3	-172.8 (2)	C22—C21—C24—O4	-171.7 (4)
C7—C8—C9—O3	5.1 (4)	C20—C21—C24—O4	6.5 (5)
C13—C8—C9—C10	6.0 (4)	C8—C9—O3—C5	7.8 (3)
C7—C8—C9—C10	-176.1 (2)	C10—C9—O3—C5	-171.09 (19)
C8—C9—C10—C11	23.6 (3)	C6—C5—O3—C9	-9.4 (3)
O3—C9—C10—C11	-157.5 (2)	C4—C5—O3—C9	169.92 (19)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C22—H22...O2 <sup>i</sup>	0.93	2.46	3.380 (3)	171

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