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ISSN 2414-3146

# 5,8-Dihydroxy-2,2-dimethyl-12-(3-methylbut-2-enyl)pyrano[3,2-*b*]xanthen-6-one (brasixanthone B)

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Received 12 October 2022

Accepted 19 December 2022

Edited by S. Parkin, University of Kentucky, USA

Keywords: crystal structure; brasixanthone B; *Calophyllum gracilentum*; xanthone; pyrano ring; 3-methylbut-2-enyl side chain.

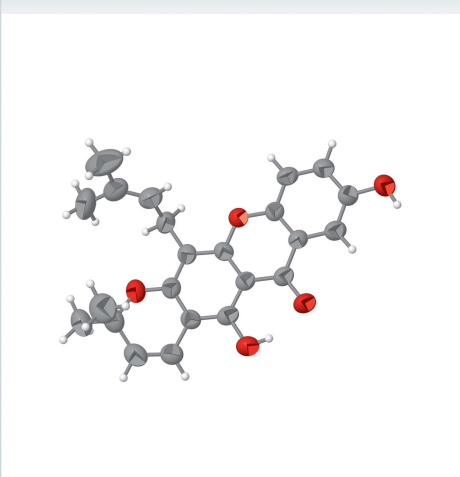
CCDC reference: 2227328

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

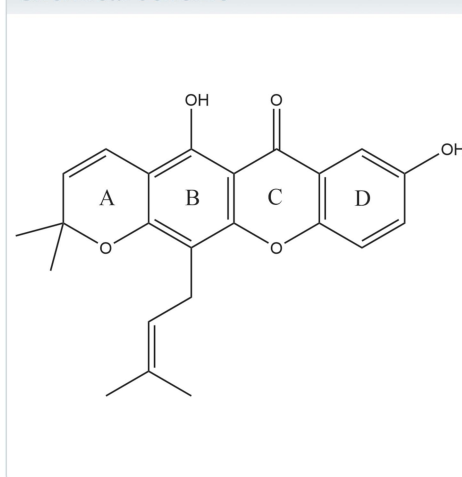
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The title compound (trivial name brasixanthone B), C<sub>23</sub>H<sub>22</sub>O<sub>5</sub>, isolated from *Calophyllum gracilentum*, is characterized by a xanthone skeleton of three fused six-membered rings plus an additional fused pyrano ring and one 3-methylbut-2-enyl side chain. The core xanthone moiety is almost planar, with a maximum deviation 0.057 (4) Å from the mean plane. In the molecule, an intramolecular O—H···O hydrogen bond forms an *S*(6) ring motif. The crystal structure features intermolecular O—H···O and C—H···O interactions.

## 3D view



## Chemical scheme



## Structure description

*Calophyllum*, frequently referred as 'bintangor' or 'penaga' in Malay is a part of the *Calophyllaceae* family (Crane *et al.*, 2005; Filho *et al.*, 2009). 80 different species have been identified in Malaysia (Corner, 1952), but studies on only 45 of them have been reported so far (Wang *et al.*, 2019). The ethnobotanical uses of *Calophyllum* in traditional medicine has been utilising several *Calophyllum* species for many thousands of years. This genus is well known for its medicinal uses and has been traditionally used for the treatment of potentially chronic diseases such as peptic ulcers, malaria, tumors, infections, high blood pressure, rheumatic disorders, eye infections, hemorrhoids, inflammation, malaria, and certain venereal diseases (Dweck & Meadows, 2002; Thiagarajan *et al.*, 2017; Zamakshshari *et al.*, 2019; Gupta & Gupta, 2020). For the biological activity of *Calophyllum* species, see: Guilet *et al.* (2001); Mah *et al.* (2012); Reyes-Chilpa *et al.* (2004);



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

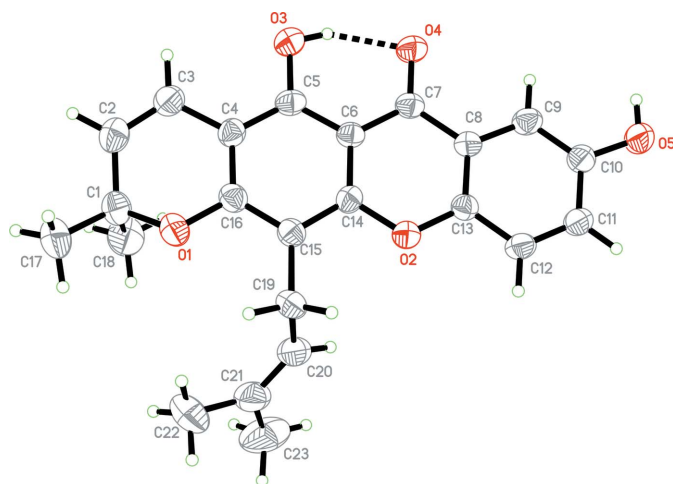
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O3-H1O3\cdots O4$	0.82	1.77	2.520 (3)	152
$O5-H1O5\cdots O4^i$	0.82	1.89	2.679 (4)	160
$C11-H11A\cdots O3^{iii}$	0.93	2.58	3.352 (5)	141
$C12-H12A\cdots O5^{iii}$	0.93	2.50	3.352 (5)	152

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

Aminudin *et al.* (2015); Lim *et al.* (2017, 2019); Zamakshshari *et al.* (2019); Karunakaran *et al.*, (2022) and Gupta & Gupta (2020). Novel xanthones and coumarins are being identified from *Calophyllum* species on a regular basis (Aminudin *et al.*, 2015; Li *et al.*, 2016). The X-ray crystallographic structure for the title compound, brasixanthone B, isolated from *Calophyllum gracilentum* is reported herein. Related structures have been reported by Ito *et al.* (2002) and Mah *et al.* (2012).

The orientation of the 3-methylbut-2-enyl side chain attached to ring B can be defined by the torsion angles  $C14-C15-C19-C20$  and  $C16-C15-C19-C20$ , which have values of  $-85.2$  (5) and  $94.2$  (4)°, respectively, suggesting a synclinal conformation (Ee *et al.*, 2010). Ring A, a 3,6-dihydro-2H-pyran, forms a screw-boat conformation (Cremer & Pople, 1975), with puckering parameters  $Q = 0.352$  (4) Å,  $\theta = 65.4$  (7)° and  $\varphi = 38.2$  (7)°. The core xanthone moiety (rings B, C and D) is almost planar, with maximum deviation of 0.057 (4) Å from the mean plane for C16. The dihedral angles between xanthone rings are:  $2.29$  (19)° for B and C,  $2.94$  (19)° for B and D, and  $0.75$  (19)° between C and D. There are two methyl groups attached to atom C1 in ring A with C—C distances of 1.488 (6) and 1.483 (6) Å.

In the title compound (Fig. 1), an intramolecular hydrogen bond,  $O3-H1O3\cdots O4$ , forms an *S*(6) ring motif. In the crystal, the molecules are linked by intermolecular hydrogen bonds  $O5-H1O5\cdots O4$ ,  $C11-H11A\cdots O3$  and  $C12-H12A\cdots O5$  (Table 1), forming extended layers lying parallel



**Figure 1**  
The molecular structure of brasixanthone B showing the atomic labeling. Displacement ellipsoids are drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

to (101) (Fig. 2). Inversion-related  $(1-x, 1-y, 2-x)$  molecules are stacked by  $\pi-\pi$  interactions with an interplanar spacing of 3.319 (4) Å between corresponding xanthone rings.

### Synthesis and crystallization

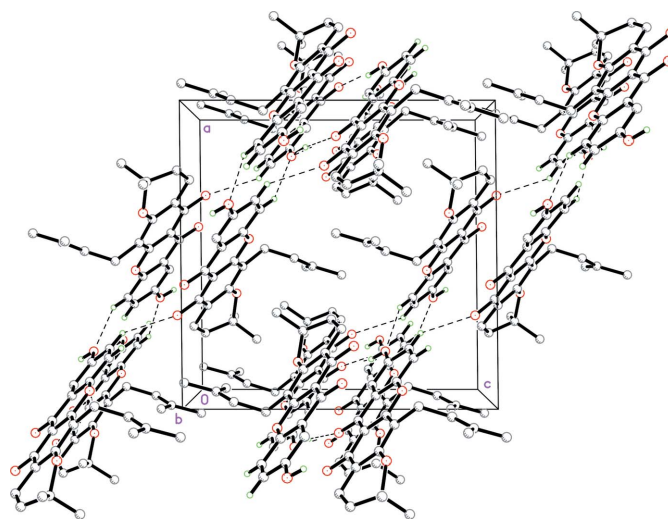
The stem bark (1.2 kg) of *calophyllum gracilentum* was ground and extracted with *n*-hexane, chloroform, ethyl acetate and methanol. Fractionation of the hexane extract by gravity column chromatography over (Merck Kieselgel No. 1.09385.1000) silica gel with elution of *n*-hexane: ethyl acetate and ethyl acetate: methanol in a step-wise gradual increment in polarity. This produced 28 fractions, which were combined and pooled together as 10 sub-fractions based on the TLC profile. Fraction 5 was subjected to further isolation by column chromatography over Sephadex LH20 eluted with methanol and several more purification steps using radial chromatography over silica (Merck Kieselgel No. 1.07749.1000), eluting with an *n*-hexane:ethyl acetate (8:2) mixture. Yellow needle-like crystals were obtained. The melting point was found to be 500–502 K (Ee *et al.*, 2011).

### Refinement

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

### Acknowledgements

The authors would like to the Sarawak Research Development Council (SRDC) (RDCRG/CAT/2019/11) and Universiti Teknologi MARA (UiTM) for financial assistance and support given throughout the research conducted on the genus *Calophyllum*. The Sarawak Biodiversity Centre (SBC) is also acknowledged for providing a research permit (SBC-2022-RDP-40-VJYM).



**Figure 2**  
The crystal packing of brasixanthone B viewed along the *b*-axis direction. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonds are omitted for clarity.

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	C <sub>23</sub> H <sub>22</sub> O <sub>5</sub>
<i>M<sub>r</sub></i>	378.40
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>n</i>
Temperature (K)	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	13.071 (3), 10.458 (3), 13.358 (3)
$\beta$ (°)	90.576 (19)
<i>V</i> (Å <sup>3</sup> )	1825.9 (8)
<i>Z</i>	4
Radiation type	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.10
Crystal size (mm)	0.31 × 0.31 × 0.31
Data collection	
Diffractometer	Bruker APEX Duo CCD area detector
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2012)
<i>T</i> <sub>min</sub> , <i>T</i> <sub>max</sub>	0.773, 0.944
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	39924, 2381, 1409
<i>R</i> <sub>int</sub>	0.117
$\theta_{\max}$ (°)	22.5
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.538
Refinement	
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.063, 0.198, 1.08
No. of reflections	2381
No. of parameters	257
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.27, -0.24

Computer programs: *APEX2* and *SAINTE* (Bruker, 2012), *SHELXT* (Sheldrick, 2015a), *SHELXL2014/7* (Sheldrick, 2015b), *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2020).

## Funding information

Funding for this research was provided by: Sarawak Research Development Council (SRDC) (grant No. RDCRG/CAT/2019/11).

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## full crystallographic data

*IUCrData* (2023). **8**, x221198 [https://doi.org/10.1107/S2414314622011981]

## 5,8-Dihydroxy-2,2-dimethyl-12-(3-methylbut-2-enyl)pyrano[3,2-*b*]xanthen-6-one (brasixanthone B)

Nurr Maria Ulfa Binti Seruji, Mohd Mustaqim Rosli, Vivien Yi Mian Jong, Thiruventhan Karunakaran, Mas Atikah Lizazman, Anita Marlina and Yanti Yana Binti Halid

### 5,8-Dihydroxy-2,2-dimethyl-12-(3-methylbut-2-enyl)pyrano[3,2-*b*]xanthen-6-one

#### Crystal data

$C_{23}H_{22}O_5$

$M_r = 378.40$

Monoclinic,  $P2_1/n$

$a = 13.071$  (3) Å

$b = 10.458$  (3) Å

$c = 13.358$  (3) Å

$\beta = 90.576$  (19)°

$V = 1825.9$  (8) Å<sup>3</sup>

$Z = 4$

$F(000) = 800$

$D_x = 1.377$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2238 reflections

$\theta = 2.2$ – $26.0$ °

$\mu = 0.10$  mm<sup>-1</sup>

$T = 296$  K

Block, orange

$0.31 \times 0.31 \times 0.31$  mm

#### Data collection

Bruker APEX Duo CCD area detector  
diffractometer

Radiation source: fine-focus sealed tube

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2012)

$T_{\min} = 0.773$ ,  $T_{\max} = 0.944$

39924 measured reflections

2381 independent reflections

1409 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.117$

$\theta_{\max} = 22.5$ °,  $\theta_{\min} = 2.2$ °

$h = -14 \rightarrow 14$

$k = -11 \rightarrow 11$

$l = -14 \rightarrow 14$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.063$

$wR(F^2) = 0.198$

$S = 1.08$

2381 reflections

257 parameters

0 restraints

Hydrogen site location: mixed

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1114P)^2 + 0.0299P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.27$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

#### 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.** The O-bound H atoms were located in a difference map, but fixed during refinement, with distance set to 0.82 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . The remaining H atoms were placed in calculated positions with  $d(\text{C-H}) = 0.93$  Å for aromatic, 0.97 Å for  $\text{CH}_2$  and 0.96 Å for  $\text{CH}_3$  atoms. The  $U_{\text{iso}}(\text{H})$  values were constrained to be  $1.5U_{\text{eq}}$  of the carrier atom for methyl H atoms and  $1.2U_{\text{eq}}$  for the remaining H atoms.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.6393 (2)	0.7811 (2)	0.86169 (18)	0.0705 (8)
O2	0.44525 (18)	0.4259 (2)	0.80181 (17)	0.0591 (7)
O3	0.70808 (19)	0.4135 (2)	1.04401 (18)	0.0706 (8)
H1O3	0.6883	0.3391	1.0424	0.106*
O4	0.60038 (19)	0.2194 (2)	1.00573 (19)	0.0700 (8)
O5	0.3386 (2)	−0.0672 (2)	0.8438 (2)	0.0850 (9)
H1O5	0.3681	−0.1001	0.8918	0.127*
C1	0.7421 (3)	0.8284 (4)	0.8784 (3)	0.0756 (12)
C2	0.7815 (3)	0.7784 (4)	0.9731 (3)	0.0755 (12)
H2A	0.8273	0.8272	1.0107	0.091*
C3	0.7539 (3)	0.6671 (4)	1.0056 (3)	0.0694 (11)
H3A	0.7841	0.6333	1.0632	0.083*
C4	0.6778 (3)	0.5970 (4)	0.9535 (3)	0.0554 (10)
C5	0.6545 (3)	0.4736 (4)	0.9740 (3)	0.0560 (10)
C6	0.5765 (3)	0.4110 (3)	0.9236 (3)	0.0496 (9)
C7	0.5540 (3)	0.2827 (4)	0.9415 (3)	0.0536 (10)
C8	0.4738 (3)	0.2279 (4)	0.8831 (3)	0.0515 (9)
C9	0.4461 (3)	0.1026 (4)	0.8922 (3)	0.0629 (11)
H9A	0.4805	0.0505	0.9378	0.076*
C10	0.3699 (3)	0.0538 (4)	0.8363 (3)	0.0624 (10)
C11	0.3208 (3)	0.1285 (4)	0.7681 (3)	0.0634 (11)
H11A	0.2690	0.0940	0.7282	0.076*
C12	0.3464 (3)	0.2513 (4)	0.7580 (3)	0.0604 (10)
H12A	0.3119	0.3026	0.7119	0.072*
C13	0.4232 (3)	0.3012 (4)	0.8152 (3)	0.0517 (9)
C14	0.5218 (3)	0.4796 (4)	0.8538 (3)	0.0515 (9)
C15	0.5407 (3)	0.6040 (4)	0.8320 (3)	0.0533 (10)
C16	0.6206 (3)	0.6586 (4)	0.8813 (3)	0.0573 (10)
C17	0.7282 (4)	0.9696 (4)	0.8798 (4)	0.0996 (15)
H17A	0.7940	1.0104	0.8830	0.149*
H17B	0.6891	0.9933	0.9373	0.149*
H17C	0.6928	0.9960	0.8200	0.149*
C18	0.8048 (4)	0.7856 (5)	0.7928 (4)	0.1052 (16)
H18A	0.8721	0.8217	0.7985	0.158*
H18B	0.7734	0.8133	0.7313	0.158*
H18C	0.8096	0.6940	0.7932	0.158*
C19	0.4787 (3)	0.6755 (4)	0.7582 (3)	0.0663 (11)
H19A	0.4081	0.6478	0.7629	0.080*
H19B	0.4811	0.7657	0.7751	0.080*
C20	0.5124 (3)	0.6594 (4)	0.6548 (3)	0.0754 (12)

H20A	0.5130	0.5761	0.6304	0.091*
C21	0.5412 (4)	0.7472 (6)	0.5937 (4)	0.0971 (16)
C22	0.5441 (5)	0.8841 (6)	0.6180 (5)	0.150 (3)
H22A	0.6108	0.9175	0.6039	0.225*
H22B	0.5297	0.8957	0.6878	0.225*
H22C	0.4938	0.9285	0.5784	0.225*
C23	0.5722 (4)	0.7131 (7)	0.4911 (4)	0.150 (3)
H23A	0.5340	0.7634	0.4437	0.225*
H23B	0.5589	0.6240	0.4796	0.225*
H23C	0.6439	0.7296	0.4834	0.225*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.074 (2)	0.0613 (19)	0.0765 (19)	−0.0085 (14)	0.0017 (15)	0.0073 (14)
O2	0.0612 (16)	0.0555 (17)	0.0604 (16)	−0.0020 (13)	−0.0084 (13)	0.0054 (13)
O3	0.0690 (19)	0.0756 (19)	0.0669 (18)	−0.0041 (14)	−0.0158 (15)	0.0045 (14)
O4	0.0730 (19)	0.0682 (18)	0.0683 (18)	0.0026 (14)	−0.0207 (15)	0.0108 (14)
O5	0.099 (2)	0.0598 (19)	0.095 (2)	−0.0146 (15)	−0.0337 (17)	0.0113 (15)
C1	0.070 (3)	0.074 (3)	0.083 (3)	−0.017 (2)	0.005 (3)	0.006 (3)
C2	0.067 (3)	0.079 (3)	0.081 (3)	−0.011 (2)	0.000 (2)	−0.008 (3)
C3	0.068 (3)	0.075 (3)	0.065 (3)	−0.004 (2)	−0.004 (2)	−0.002 (2)
C4	0.049 (2)	0.062 (3)	0.055 (2)	−0.005 (2)	0.0009 (19)	−0.001 (2)
C5	0.050 (2)	0.067 (3)	0.052 (2)	0.009 (2)	−0.002 (2)	0.004 (2)
C6	0.046 (2)	0.054 (2)	0.049 (2)	0.0064 (18)	0.0004 (18)	0.0034 (19)
C7	0.051 (2)	0.059 (3)	0.051 (2)	0.0056 (19)	−0.001 (2)	0.006 (2)
C8	0.053 (2)	0.053 (2)	0.048 (2)	0.0031 (19)	−0.0023 (19)	0.0026 (19)
C9	0.067 (3)	0.059 (3)	0.063 (3)	0.007 (2)	−0.013 (2)	0.002 (2)
C10	0.068 (3)	0.051 (3)	0.067 (3)	0.001 (2)	−0.011 (2)	0.003 (2)
C11	0.066 (3)	0.062 (3)	0.062 (3)	−0.001 (2)	−0.013 (2)	0.001 (2)
C12	0.061 (3)	0.064 (3)	0.055 (2)	0.006 (2)	−0.011 (2)	0.004 (2)
C13	0.055 (2)	0.052 (3)	0.048 (2)	−0.0012 (19)	0.0014 (19)	0.0014 (19)
C14	0.047 (2)	0.055 (3)	0.053 (2)	−0.0028 (18)	−0.0011 (19)	−0.0026 (19)
C15	0.054 (2)	0.053 (2)	0.052 (2)	0.0002 (19)	0.0054 (19)	0.0014 (19)
C16	0.061 (3)	0.056 (3)	0.055 (2)	−0.003 (2)	0.006 (2)	0.003 (2)
C17	0.105 (4)	0.076 (3)	0.118 (4)	−0.027 (3)	−0.002 (3)	0.008 (3)
C18	0.104 (4)	0.123 (4)	0.089 (3)	−0.019 (3)	0.030 (3)	−0.004 (3)
C19	0.070 (3)	0.059 (3)	0.070 (3)	−0.002 (2)	−0.004 (2)	0.015 (2)
C20	0.075 (3)	0.085 (3)	0.066 (3)	0.002 (2)	−0.008 (2)	0.008 (3)
C21	0.086 (3)	0.128 (5)	0.078 (4)	−0.023 (3)	−0.012 (3)	0.034 (3)
C22	0.181 (6)	0.117 (5)	0.151 (6)	−0.050 (4)	−0.026 (5)	0.068 (4)
C23	0.122 (5)	0.263 (8)	0.065 (4)	−0.019 (5)	0.005 (3)	0.032 (4)

*Geometric parameters (Å, °)*

O1—C16	1.330 (4)	C11—C12	1.335 (5)
O1—C1	1.446 (4)	C11—H11A	0.9300
O2—C14	1.336 (4)	C12—C13	1.360 (5)

O2—C13	1.348 (4)	C12—H12A	0.9300
O3—C5	1.321 (4)	C14—C15	1.357 (5)
O3—H1O3	0.8200	C15—C16	1.356 (5)
O4—C7	1.238 (4)	C15—C19	1.474 (5)
O5—C10	1.334 (4)	C17—H17A	0.9600
O5—H1O5	0.8199	C17—H17B	0.9600
C1—C2	1.458 (6)	C17—H17C	0.9600
C1—C18	1.483 (6)	C18—H18A	0.9600
C1—C17	1.488 (6)	C18—H18B	0.9600
C2—C3	1.295 (5)	C18—H18C	0.9600
C2—H2A	0.9300	C19—C20	1.464 (5)
C3—C4	1.414 (5)	C19—H19A	0.9700
C3—H3A	0.9300	C19—H19B	0.9700
C4—C5	1.354 (5)	C20—C21	1.287 (6)
C4—C16	1.374 (5)	C20—H20A	0.9300
C5—C6	1.382 (5)	C21—C22	1.468 (8)
C6—C14	1.371 (5)	C21—C23	1.477 (7)
C6—C7	1.395 (5)	C22—H22A	0.9600
C7—C8	1.421 (5)	C22—H22B	0.9600
C8—C13	1.355 (5)	C22—H22C	0.9600
C8—C9	1.365 (5)	C23—H23A	0.9600
C9—C10	1.340 (5)	C23—H23B	0.9600
C9—H9A	0.9300	C23—H23C	0.9600
C10—C11	1.357 (5)		
C16—O1—C1	118.1 (3)	O2—C14—C15	115.4 (3)
C14—O2—C13	119.8 (3)	O2—C14—C6	121.1 (3)
C5—O3—H1O3	105.6	C15—C14—C6	123.5 (4)
C10—O5—H1O5	108.3	C16—C15—C14	116.2 (3)
O1—C1—C2	109.3 (3)	C16—C15—C19	121.8 (4)
O1—C1—C18	107.3 (4)	C14—C15—C19	122.0 (4)
C2—C1—C18	111.5 (4)	O1—C16—C15	116.9 (3)
O1—C1—C17	103.2 (4)	O1—C16—C4	119.4 (4)
C2—C1—C17	112.7 (4)	C15—C16—C4	123.6 (4)
C18—C1—C17	112.2 (4)	C1—C17—H17A	109.5
C3—C2—C1	121.1 (4)	C1—C17—H17B	109.5
C3—C2—H2A	119.5	H17A—C17—H17B	109.5
C1—C2—H2A	119.5	C1—C17—H17C	109.5
C2—C3—C4	119.9 (4)	H17A—C17—H17C	109.5
C2—C3—H3A	120.1	H17B—C17—H17C	109.5
C4—C3—H3A	120.1	C1—C18—H18A	109.5
C5—C4—C16	117.9 (4)	C1—C18—H18B	109.5
C5—C4—C3	123.6 (4)	H18A—C18—H18B	109.5
C16—C4—C3	118.5 (4)	C1—C18—H18C	109.5
O3—C5—C4	118.6 (4)	H18A—C18—H18C	109.5
O3—C5—C6	120.2 (4)	H18B—C18—H18C	109.5
C4—C5—C6	121.3 (3)	C20—C19—C15	113.8 (3)
C14—C6—C5	117.4 (4)	C20—C19—H19A	108.8

C14—C6—C7	120.7 (4)	C15—C19—H19A	108.8
C5—C6—C7	121.9 (3)	C20—C19—H19B	108.8
O4—C7—C6	122.0 (4)	C15—C19—H19B	108.8
O4—C7—C8	121.2 (3)	H19A—C19—H19B	107.7
C6—C7—C8	116.7 (3)	C21—C20—C19	127.5 (5)
C13—C8—C9	118.3 (3)	C21—C20—H20A	116.3
C13—C8—C7	119.4 (4)	C19—C20—H20A	116.3
C9—C8—C7	122.2 (3)	C20—C21—C22	124.2 (5)
C10—C9—C8	120.8 (4)	C20—C21—C23	120.0 (6)
C10—C9—H9A	119.6	C22—C21—C23	115.7 (5)
C8—C9—H9A	119.6	C21—C22—H22A	109.5
O5—C10—C9	123.1 (4)	C21—C22—H22B	109.5
O5—C10—C11	117.0 (4)	H22A—C22—H22B	109.5
C9—C10—C11	119.9 (4)	C21—C22—H22C	109.5
C12—C11—C10	120.3 (4)	H22A—C22—H22C	109.5
C12—C11—H11A	119.8	H22B—C22—H22C	109.5
C10—C11—H11A	119.8	C21—C23—H23A	109.5
C11—C12—C13	119.8 (4)	C21—C23—H23B	109.5
C11—C12—H12A	120.1	H23A—C23—H23B	109.5
C13—C12—H12A	120.1	C21—C23—H23C	109.5
O2—C13—C8	122.2 (3)	H23A—C23—H23C	109.5
O2—C13—C12	117.0 (3)	H23B—C23—H23C	109.5
C8—C13—C12	120.8 (4)		
C16—O1—C1—C2	-43.1 (5)	C14—O2—C13—C12	-178.1 (3)
C16—O1—C1—C18	78.0 (4)	C9—C8—C13—O2	179.6 (3)
C16—O1—C1—C17	-163.3 (3)	C7—C8—C13—O2	-1.0 (5)
O1—C1—C2—C3	31.7 (6)	C9—C8—C13—C12	0.3 (5)
C18—C1—C2—C3	-86.9 (5)	C7—C8—C13—C12	179.7 (3)
C17—C1—C2—C3	145.9 (4)	C11—C12—C13—O2	-179.8 (3)
C1—C2—C3—C4	-5.5 (6)	C11—C12—C13—C8	-0.4 (5)
C2—C3—C4—C5	170.6 (4)	C13—O2—C14—C15	177.4 (3)
C2—C3—C4—C16	-12.8 (6)	C13—O2—C14—C6	-2.3 (5)
C16—C4—C5—O3	-179.3 (3)	C5—C6—C14—O2	180.0 (3)
C3—C4—C5—O3	-2.6 (5)	C7—C6—C14—O2	0.5 (5)
C16—C4—C5—C6	0.7 (5)	C5—C6—C14—C15	0.3 (5)
C3—C4—C5—C6	177.3 (3)	C7—C6—C14—C15	-179.1 (3)
O3—C5—C6—C14	178.2 (3)	O2—C14—C15—C16	-177.6 (3)
C4—C5—C6—C14	-1.7 (5)	C6—C14—C15—C16	2.1 (5)
O3—C5—C6—C7	-2.4 (5)	O2—C14—C15—C19	1.8 (5)
C4—C5—C6—C7	177.7 (3)	C6—C14—C15—C19	-178.5 (3)
C14—C6—C7—O4	-178.1 (3)	C1—O1—C16—C15	-155.5 (3)
C5—C6—C7—O4	2.4 (5)	C1—O1—C16—C4	28.4 (5)
C14—C6—C7—C8	1.0 (5)	C14—C15—C16—O1	-179.2 (3)
C5—C6—C7—C8	-178.4 (3)	C19—C15—C16—O1	1.4 (5)
O4—C7—C8—C13	178.4 (3)	C14—C15—C16—C4	-3.3 (5)
C6—C7—C8—C13	-0.8 (5)	C19—C15—C16—C4	177.3 (3)
O4—C7—C8—C9	-2.2 (5)	C5—C4—C16—O1	177.8 (3)



C6—C7—C8—C9	178.6 (3)	C3—C4—C16—O1	1.0 (5)
C13—C8—C9—C10	-0.8 (5)	C5—C4—C16—C15	2.0 (6)
C7—C8—C9—C10	179.8 (3)	C3—C4—C16—C15	-174.8 (3)
C8—C9—C10—O5	-178.6 (3)	C16—C15—C19—C20	94.2 (4)
C8—C9—C10—C11	1.4 (6)	C14—C15—C19—C20	-85.2 (5)
O5—C10—C11—C12	178.5 (4)	C15—C19—C20—C21	-121.4 (5)
C9—C10—C11—C12	-1.5 (6)	C19—C20—C21—C22	-0.8 (8)
C10—C11—C12—C13	1.0 (6)	C19—C20—C21—C23	-179.6 (4)
C14—O2—C13—C8	2.5 (5)		

*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>
O3—H1O3...O4	0.82	1.77	2.520 (3)	152
O5—H1O5...O4 <sup>i</sup>	0.82	1.89	2.679 (4)	160
C11—H11A...O3 <sup>ii</sup>	0.93	2.58	3.352 (5)	141
C12—H12A...O5 <sup>iii</sup>	0.93	2.50	3.352 (5)	152

Symmetry codes: (i)  $-x+1, -y, -z+2$ ; (ii)  $x-3/2, -y-1/2, z-3/2$ ; (iii)  $-x+1/2, y+1/2, -z+3/2$ .