Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Hopeahainol C monohydrate

**Hoong-Kun Fun,[a]*‡ Kanokorn Sudto,[b] Hui-Ming Ge,[c] Ren-Xiang Tan,[c] Supa Hannongbua[b] and Suchada Chantrapromma[d]§**

[a]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]Department of Chemistry, Faculty of Science, Kasetsart University, Jatujak, Bangkok 10900, Thailand, [c]Institute of Functional Biomolecules, State Key Laboratory of Pharmaceutical Biotechnology, Nanjing University, Nanjing 210093, People's Republic of China, and [d]Crystal Materials Research Unit, Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat-Yai, Songkhla 90112, Thailand
Correspondence e-mail: hkfun@usm.my

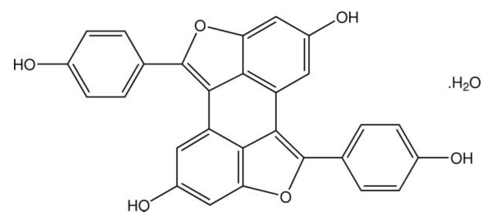
Received 28 April 2011; accepted 5 May 2011

Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.068; $wR$ factor = 0.161; data-to-parameter ratio = 13.3.

In the structure of the title compound, $C_{28}H_{16}O_6\cdot H_2O$ [systematic name 3,11-bis(4-hydroxyphenyl)-4,12-dioxapentacyclo[8.6.1.1$^{2,5}$.0$^{13,17}$.0$^{9,18}$]octadeca-1(16),2,5(18),6,8,10,-13(17),14-octaene-7,15-diol monohydrate], the hopeahainol C molecule lies about an inversion center with the solvent water molecule located on a crystallographic twofold axis. Hopeahainol C is an oligostillbenoid compound and was isolated from the bark of *Shorea roxburghii* G. Don. The five central fused rings are essentially planar with an r.m.s. deviation of 0.0173 (3) Å. The 4-hydroxyphenyl ring is twisted with respect to this plane, with the dihedral angle between the phenyl ring and the fused-ring system being 41.70 (10)°. The crystal features intermolecular O—H···O hydrogen bonds. These interactions link the hopeahainol C molecules into chains along the $b$ axis. Water molecules are located interstitially between the hopeahainol C molecules linked by O(water)—H···O(hydroxy) and O(hydroxy)—H···O(water) hydrogen bonds. $\pi$–$\pi$ interactions are also observed with centroid–centroid distances of 3.6056 (17) and 3.5622 (17) Å. Short O···O contacts [2.703 (2)–2.720 (3) Å] are also present in the crystal.

## Related literature

For bond-length data, see: Allen *et al.* (1987). For background to oligostillbenoids and their activities, see: Cai *et al.* (2003); Donnelly *et al.* (2004); Ge *et al.* (2009); Jang & Pezzuto (1999); Stivala *et al.* (2001). For details of Dipterocarpaceae plants, see: Gorham (1995); Hakim (2002); Sotheeswaran & Pasuphaty (1993); Symington (1974). For the stability of the temperature controller used in the data collection, see Cosier & Glazer, (1986).




## Experimental

*Crystal data*

$C_{28}H_{16}O_6\cdot H_2O$
$M_r$ = 466.42
Monoclinic, $C2/c$
$a$ = 21.225 (4) Å
$b$ = 3.8500 (7) Å
$c$ = 25.353 (5) Å
$\beta$ = 108.933 (4)°
$V$ = 1959.7 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 100 K
0.25 × 0.15 × 0.05 mm

*Data collection*

Bruker APEX DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.972, $T_{max}$ = 0.994
7974 measured reflections
2171 independent reflections
1463 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.082

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.161
$S$ = 1.07
2171 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1$W$—H1$W$1···O2$^{i}$ | 0.92 (3) | 1.83 (3) | 2.720 (2) | 163 (3) |
| O3—H3$A$···O1$W^{ii}$ | 0.82 | 1.89 | 2.703 (2) | 169 |
| O2—H2$A$···O3$^{iii}$ | 0.82 | 2.00 | 2.716 (3) | 145 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

This project was supported by the Thailand Research Fund (grant Nos. DBG5280018 and RTA5380010). The authors are also very grateful to Associate Professor Srunya Vajrodaya, Head of Department of Botany, Faculty of Science, Kasetsart University, Thailand, for identification of the plant specimens. The authors also thank the Malaysian government and Universiti Sains Malaysia for the Research University Grant No. 1001/PFIZIK/811160.

‡ Thomson Reuters ResearcherID: A-3561-2009.
§ Additional correspondence author, e-mail: suchada.c@psu.ac.th. Thomson Reuters ResearcherID: A-5085-2009.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5138).

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cai, Y. J., Fang, J. G., Ma, L. P., Yang, L. & Liu, Z. L. (2003). *Biochim. Biophys. Acta*, **1637**, 31–38.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Donnelly, L. E., Newton, R., Kennedy, G. E., Fenwick, P. S., Leung, R. H., Ito, K., Russell, R. E. & Barnes, P. J. (2004). *Am. J. Physiol. Lung Cell Mol. Physiol*. **287**, L774–L783.

Ge, H. M., Yang, W. H., Zang, J. & Tan, R. X. (2009). *J. Agric. Food. Chem*. **57**, 5756–5761.

Gorham, J. (1995). *The Biochemistry of the Stilbenoids*. London: Chapman & Hall.

Hakim, E. H. (2002). *Bull. Ind. Soc. Nat. Prod. Chem. (Indonesia)*, **2**, 1.

Jang, M. & Pezzuto, J. M. (1999). *Drugs Exp. Clin. Res*. **25**, 65–77.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sotheeswaran, S. & Pasuphaty, V. (1993). *Phytochemistry*, **32**, 1083–1092.

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

Stivala, L. A., Savio, M., Carafoli, F., Perucca, P., Bianchi, L., Maga, G., Forti, L., Pagnoni, U. M., Albini, A., Prosperi, E. & Vannini, V. (2001). *J. Biol. Chem*. **276**, 22586–22594.

Symington, C. F. (1974). *Foresters Manual of Dipterocarps*. Penebit University Malaya, Kuala Lumpur, Malaysia.

## supplementary materials

*Acta Cryst.* (2011). E**67**, o1392-o1393 [ **doi:10.1107/S1600536811017053** ]

## Hopeahainol C monohydrate

**H.-K. Fun, K. Sudto, H.-M. Ge, R.-X. Tan, S. Hannongbua and S. Chantrapromma**

### Comment

The genus *Shorea* is the largest genus of the family Dipterocarpaceae and is mostly distributed in Southeast Asia (Symington, 1974). The Dipterocarpaceous plant has already proved to be a rich source of oligostilbene compounds that are derived from stilbene and resveratrol (3,5,4'-trihydroxystilbene) (Gorham, 1995; Hakim, 2002; Sotheeswaran & Pasuphaty, 1993). It also has been known that resveratrol possesses various biological activities including antioxidant (Cai *et al*., 2003), anti-cancer, chemo-preventive (Jang & Pezzuto, 1999; Stivala *et al*., 2001) and anti-inflammatory properties (Donnelly *et al*., 2004). During the course of our research on searching for novel bioactive compounds from Thai dipeterocarpaceous plants, the title compound (I), known as hopeahainol C, was obtained from *Shorea roxburghii* G. Don. Hopeahainol C is an oligostilbenoid, which is a highly unsaturated resveratrol dimer, and it possesses potent antioxidant activity (Ge *et al*., 2009). Herein we report its crystal structure.

The molecule of the title oligostilbenoid (I) (Fig. 1), $C_{28}H_{16}O_6.H_2O$, is a symmetrical dimer. Its asymmetric unit contains one half-molecule. The complete molecule of hopeahainol C is generated by a crystallographic center of symmetry 1/2-x, 3/2-y, -z whereas the other hydrogen atom of the water molecule is generated by a two-fold rotation axis -x, y, 1/2-z. The five central fused rings are essentially planar with the *r.m.s*. 0.0173 (3) Å for the eighteen non-hydrogen atoms. The 4-hydroxyphenyl ring is twisted which respect to the five central fused rings with the dihedral angle between the phenyl and the five central fused rings being 41.65 (10)°. The dihedral angle between the phenyl and the attached dihydrofuran (O1/C7–C9/C14) rings is 40.50 (15)°. The two hydroxy groups of the half molecule are co-planar with the attached benzene ring with the torsion angles O3–C4–C5–C6 = -178.1 (2)° and C10–C11–C12–O2 = -179.5 (2)°. The bond distances are of normal values (Allen *et al*., 1987).

In the crystal packing (Fig. 2), the molecules of hopeahainol C are linked into chains along the *b* axis by O(hydroxy)—H···O(hydroxy) hydrogen bonds which form between the two hydroxy groups (Table 1). The water molecules are located in the interstitials of hopeahainol C molecules and are linked to the molecules of hopehainol C by two types of hydrogen bond *i.e*. O(water)—H···O(hydroxy) and O(hydroxy)—H···O(water) hydrogens bond (Table 1). The crystal is consolidated by these O—H···O hydrogen bonds. $\pi$–$\pi$ interaction with $Cg_1\cdots Cg_3$ distance = 3.6055 (17) Å (symmetry code: x, 1+y, z) and $Cg_2\cdots Cg_3$ distance = 3.5622 (17) Å (symmetry code: 1/2-x, 5/2-y, -z) were observed where *Cg*1, *Cg*2 and *Cg*3 are the centroids of the O1/C7–C9/C14, C8–C10/C8A–C10A and C9–C14 rings, respectively. In addition O···O short contacts [2.703 (2)-2.720 (3) Å] are also presented in the crystal.

### Experimental

The dried powdered bark of *Shorea roxburghii* G. Don. (1 kg) which was collected during June-August 2010 from Sam Sung District, Khon Kaen province in the northeastern part of Thailand, was macerated in $C_2H_5OH$ (2.5 L) for 7 days. The slurry was filtered and the ethanolic extract obtained was dried with a rotary evaporator under reduced pressure at 313 K. The dried extract (98 g) was ground, dissolved in $CH_3OH$, mixed with silica gel (118 g), and then dried in hot air oven at 333

K for one day. The sample was separated by using a wet column chromatographic technique. The column was packed with 1 kg of silica gel (200-300 mesh) and was eluted with gradient mixtures of $CHCl_3$ and $CH_3OH$ (100:0 to 0:100), to give 8 major fractions (A–H). Fraction G was further isolated using sephadex column chromatography, eluted with 100% $CH_3OH$. Only the blue methanolic extract could be selectively collected and was pre-concentrated and heated for 5-10 minutes at 313-333 K before being left for 2-3 days at 298 K to allow crystallization of hopeahainol C. Colorless needle-shaped single crystals of the hopeahainol C suitable for *X*-ray structure determination were obtained from $CH_3OH$ by slow evaporation at room temperature after a few days.

### Refinement

The water H atom was located in a difference map and refined isotropically. The remaining H atoms were placed in calculated positions with d(O—H) = 0.82 Å and d(C—H) = 0.93 Å for aromatic. The $U_{iso}$ values were constrained to be $1.5U_{eq}$ of the carrier atom for hydroxy and $1.2U_{eq}$ for the remaining H atoms. The highest residual electron density peak is located at 0.66 Å from C9 and the deepest hole is located at 0.90 Å from C4.

### Figures

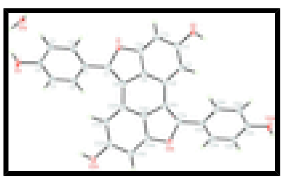


Fig. 1. The molecular structure of the title compound, with 60% probability displacement ellipsoids and the atom-numbering scheme. Atoms with suffix A were generated by the symmetry code 1/2-x, 3/2-y, -z.

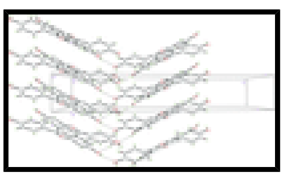


Fig. 2. The crystal packing of the title compound viewed down the *a* axis, showing chains running along the *b* axis. Hydrogen bonds are shown as dashed lines.

### 3,11-bis(4-hydroxyphenyl)-4,12- dioxapentacyclo[8.6.1.1$^{2,5}$.0$^{13,17}$.0$^{9,18}$]octadeca-1(16),2,5(18),6,8,10,13 (17),14-octaene-7,15-diol monohydrate

*Crystal data*

| | |
|---|---|
| $C_{28}H_{16}O_6 \cdot H_2O$ | $F(000) = 968$ |
| $M_r = 466.42$ | $D_x = 1.581$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -C 2yc | Cell parameters from 2171 reflections |
| $a = 21.225$ (4) Å | $\theta = 2.0$–$24.8°$ |
| $b = 3.8500$ (7) Å | $\mu = 0.11$ mm$^{-1}$ |
| $c = 25.353$ (5) Å | $T = 100$ K |
| $\beta = 108.933$ (4)° | Needle, colorless |
| $V = 1959.7$ (6) Å$^3$ | 0.25 × 0.15 × 0.05 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker APEX DUO CCD area-detector diffractometer | 2171 independent reflections |
| Radiation source: sealed tube | 1463 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.082$ |
| φ and ω scans | $\theta_{max} = 27.5°$, $\theta_{min} = 2.0°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2009) | $h = -27 \rightarrow 27$ |
| $T_{min} = 0.972$, $T_{max} = 0.994$ | $k = -4 \rightarrow 4$ |
| 7974 measured reflections | $l = -32 \rightarrow 32$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.068$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.161$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.07$ | $w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 1.9771P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 2171 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 163 parameters | $\Delta\rho_{max} = 0.27$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.35$ e Å$^{-3}$ |

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1W | 0.0000 | 0.5114 (9) | 0.2500 | 0.0199 (7) |
| H1W1 | 0.0200 (15) | 0.649 (9) | 0.2803 (13) | 0.036 (10)* |
| O1 | 0.10067 (8) | 0.6569 (5) | 0.01488 (7) | 0.0151 (5) |

| | | | | |
|---|---|---|---|---|
| O2 | 0.08243 (8) | 0.1345 (5) | −0.16160 (7) | 0.0170 (5) |
| H2A | 0.1076 | 0.0687 | −0.1782 | 0.026* |
| O3 | 0.11424 (8) | 1.1827 (5) | 0.25471 (7) | 0.0193 (5) |
| H3A | 0.0782 | 1.2799 | 0.2487 | 0.029* |
| C1 | 0.14502 (11) | 0.9053 (7) | 0.10634 (10) | 0.0136 (6) |
| C2 | 0.08471 (11) | 1.0602 (8) | 0.10499 (10) | 0.0156 (6) |
| H2B | 0.0521 | 1.1031 | 0.0709 | 0.019* |
| C3 | 0.07334 (12) | 1.1499 (7) | 0.15414 (10) | 0.0155 (6) |
| H3B | 0.0331 | 1.2504 | 0.1530 | 0.019* |
| C4 | 0.12224 (12) | 1.0890 (8) | 0.20487 (10) | 0.0156 (6) |
| C5 | 0.18124 (12) | 0.9313 (8) | 0.20726 (10) | 0.0165 (6) |
| H5A | 0.2133 | 0.8855 | 0.2415 | 0.020* |
| C6 | 0.19264 (12) | 0.8408 (8) | 0.15796 (10) | 0.0152 (6) |
| H6A | 0.2327 | 0.7357 | 0.1595 | 0.018* |
| C7 | 0.15626 (11) | 0.8033 (7) | 0.05476 (10) | 0.0136 (6) |
| C8 | 0.20988 (11) | 0.8046 (8) | 0.03621 (10) | 0.0130 (6) |
| C9 | 0.18721 (11) | 0.6449 (7) | −0.01786 (10) | 0.0124 (6) |
| C10 | 0.22099 (11) | 0.5734 (7) | −0.05605 (10) | 0.0119 (6) |
| C11 | 0.18493 (12) | 0.4004 (7) | −0.10467 (10) | 0.0150 (6) |
| H11A | 0.2051 | 0.3459 | −0.1312 | 0.018* |
| C12 | 0.11835 (12) | 0.3081 (7) | −0.11382 (10) | 0.0137 (6) |
| C13 | 0.08421 (11) | 0.3852 (7) | −0.07681 (10) | 0.0140 (6) |
| H13A | 0.0397 | 0.3262 | −0.0838 | 0.017* |
| C14 | 0.12110 (11) | 0.5554 (7) | −0.02905 (10) | 0.0129 (6) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1W | 0.0212 (12) | 0.026 (2) | 0.0148 (13) | 0.000 | 0.0088 (11) | 0.000 |
| O1 | 0.0162 (8) | 0.0197 (12) | 0.0120 (8) | −0.0010 (8) | 0.0081 (7) | −0.0009 (9) |
| O2 | 0.0175 (8) | 0.0233 (13) | 0.0119 (8) | −0.0030 (8) | 0.0070 (7) | −0.0049 (9) |
| O3 | 0.0252 (9) | 0.0244 (13) | 0.0129 (9) | 0.0047 (9) | 0.0128 (7) | 0.0005 (9) |
| C1 | 0.0164 (11) | 0.0117 (16) | 0.0152 (12) | −0.0029 (11) | 0.0086 (9) | −0.0007 (12) |
| C2 | 0.0161 (11) | 0.0175 (17) | 0.0154 (12) | −0.0018 (11) | 0.0079 (9) | 0.0004 (12) |
| C3 | 0.0158 (11) | 0.0165 (17) | 0.0180 (12) | −0.0008 (11) | 0.0106 (9) | −0.0006 (13) |
| C4 | 0.0217 (12) | 0.0167 (17) | 0.0129 (12) | −0.0037 (11) | 0.0117 (10) | −0.0019 (12) |
| C5 | 0.0189 (11) | 0.0174 (17) | 0.0139 (12) | −0.0006 (11) | 0.0064 (9) | 0.0015 (12) |
| C6 | 0.0165 (11) | 0.0155 (16) | 0.0171 (12) | 0.0000 (11) | 0.0102 (9) | 0.0002 (12) |
| C7 | 0.0157 (11) | 0.0130 (16) | 0.0120 (11) | −0.0012 (11) | 0.0044 (9) | −0.0005 (12) |
| C8 | 0.0170 (11) | 0.0128 (16) | 0.0109 (11) | 0.0002 (11) | 0.0069 (9) | 0.0009 (12) |
| C9 | 0.0166 (11) | 0.0099 (15) | 0.0122 (11) | 0.0013 (11) | 0.0068 (9) | 0.0019 (12) |
| C10 | 0.0155 (11) | 0.0102 (16) | 0.0122 (11) | 0.0019 (10) | 0.0073 (9) | 0.0035 (12) |
| C11 | 0.0198 (11) | 0.0156 (17) | 0.0128 (12) | 0.0003 (11) | 0.0097 (9) | 0.0008 (12) |
| C12 | 0.0202 (11) | 0.0100 (16) | 0.0116 (11) | −0.0006 (11) | 0.0060 (9) | 0.0013 (12) |
| C13 | 0.0140 (10) | 0.0140 (17) | 0.0152 (12) | −0.0010 (11) | 0.0065 (9) | 0.0015 (12) |
| C14 | 0.0178 (11) | 0.0114 (16) | 0.0130 (12) | 0.0010 (11) | 0.0099 (9) | 0.0017 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1W—H1W1 | 0.92 (3) | C5—C6 | 1.392 (3) |
| O1—C14 | 1.377 (3) | C5—H5A | 0.9300 |
| O1—C7 | 1.399 (3) | C6—H6A | 0.9300 |
| O2—C12 | 1.377 (3) | C7—C8 | 1.365 (3) |
| O2—H2A | 0.8200 | C8—C9 | 1.436 (3) |
| O3—C4 | 1.377 (3) | C8—C10$^{i}$ | 1.465 (3) |
| O3—H3A | 0.8200 | C9—C14 | 1.382 (3) |
| C1—C6 | 1.392 (3) | C9—C10 | 1.406 (3) |
| C1—C2 | 1.403 (3) | C10—C11 | 1.392 (3) |
| C1—C7 | 1.457 (3) | C10—C8$^{i}$ | 1.465 (3) |
| C2—C3 | 1.388 (3) | C11—C12 | 1.402 (3) |
| C2—H2B | 0.9300 | C11—H11A | 0.9300 |
| C3—C4 | 1.386 (4) | C12—C13 | 1.391 (3) |
| C3—H3B | 0.9300 | C13—C14 | 1.376 (4) |
| C4—C5 | 1.375 (3) | C13—H13A | 0.9300 |
| | | | |
| C14—O1—C7 | 106.61 (17) | O1—C7—C1 | 114.32 (19) |
| C12—O2—H2A | 109.5 | C7—C8—C9 | 105.6 (2) |
| C4—O3—H3A | 109.5 | C7—C8—C10$^{i}$ | 137.4 (2) |
| C6—C1—C2 | 118.5 (2) | C9—C8—C10$^{i}$ | 116.94 (19) |
| C6—C1—C7 | 121.0 (2) | C14—C9—C10 | 121.3 (2) |
| C2—C1—C7 | 120.4 (2) | C14—C9—C8 | 107.8 (2) |
| C3—C2—C1 | 120.5 (2) | C10—C9—C8 | 130.8 (2) |
| C3—C2—H2B | 119.8 | C11—C10—C9 | 116.5 (2) |
| C1—C2—H2B | 119.8 | C11—C10—C8$^{i}$ | 131.2 (2) |
| C4—C3—C2 | 119.7 (2) | C9—C10—C8$^{i}$ | 112.2 (2) |
| C4—C3—H3B | 120.2 | C10—C11—C12 | 120.2 (2) |
| C2—C3—H3B | 120.2 | C10—C11—H11A | 119.9 |
| C5—C4—O3 | 117.2 (2) | C12—C11—H11A | 119.9 |
| C5—C4—C3 | 120.8 (2) | O2—C12—C13 | 115.8 (2) |
| O3—C4—C3 | 122.0 (2) | O2—C12—C11 | 120.6 (2) |
| C4—C5—C6 | 119.5 (2) | C13—C12—C11 | 123.5 (2) |
| C4—C5—H5A | 120.3 | C14—C13—C12 | 115.0 (2) |
| C6—C5—H5A | 120.3 | C14—C13—H13A | 122.5 |
| C1—C6—C5 | 121.0 (2) | C12—C13—H13A | 122.5 |
| C1—C6—H6A | 119.5 | C13—C14—O1 | 127.5 (2) |
| C5—C6—H6A | 119.5 | C13—C14—C9 | 123.3 (2) |
| C8—C7—O1 | 110.7 (2) | O1—C14—C9 | 109.2 (2) |
| C8—C7—C1 | 134.9 (2) | | |
| | | | |
| C6—C1—C2—C3 | 0.7 (4) | C10$^{i}$—C8—C9—C14 | −178.9 (2) |
| C7—C1—C2—C3 | 178.5 (3) | C7—C8—C9—C10 | −179.2 (3) |
| C1—C2—C3—C4 | 0.7 (4) | C10$^{i}$—C8—C9—C10 | 2.0 (5) |
| C2—C3—C4—C5 | −1.9 (4) | C14—C9—C10—C11 | −1.7 (4) |
| C2—C3—C4—O3 | 178.0 (3) | C8—C9—C10—C11 | 177.4 (3) |
| O3—C4—C5—C6 | −178.1 (2) | C14—C9—C10—C8$^{i}$ | 179.0 (2) |

| | | | |
|---|---|---|---|
| C3—C4—C5—C6 | 1.8 (4) | C8—C9—C10—C8$^{i}$ | −1.9 (5) |
| C2—C1—C6—C5 | −0.8 (4) | C9—C10—C11—C12 | 0.4 (4) |
| C7—C1—C6—C5 | −178.5 (3) | C8$^{i}$—C10—C11—C12 | 179.5 (3) |
| C4—C5—C6—C1 | −0.5 (4) | C10—C11—C12—O2 | −179.5 (2) |
| C14—O1—C7—C8 | 1.7 (3) | C10—C11—C12—C13 | 1.2 (4) |
| C14—O1—C7—C1 | −176.0 (2) | O2—C12—C13—C14 | 179.3 (2) |
| C6—C1—C7—C8 | −39.1 (5) | C11—C12—C13—C14 | −1.3 (4) |
| C2—C1—C7—C8 | 143.2 (3) | C12—C13—C14—O1 | −178.7 (3) |
| C6—C1—C7—O1 | 138.0 (3) | C12—C13—C14—C9 | −0.1 (4) |
| C2—C1—C7—O1 | −39.8 (4) | C7—O1—C14—C13 | 177.0 (3) |
| O1—C7—C8—C9 | −1.0 (3) | C7—O1—C14—C9 | −1.7 (3) |
| C1—C7—C8—C9 | 176.1 (3) | C10—C9—C14—C13 | 1.6 (4) |
| O1—C7—C8—C10$^{i}$ | 177.4 (3) | C8—C9—C14—C13 | −177.7 (3) |
| C1—C7—C8—C10$^{i}$ | −5.5 (6) | C10—C9—C14—O1 | −179.6 (2) |
| C7—C8—C9—C14 | −0.1 (3) | C8—C9—C14—O1 | 1.1 (3) |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1W—H1W1···O2$^{ii}$ | 0.92 (3) | 1.83 (3) | 2.720 (2) | 163 (3) |
| O3—H3A···O1W$^{iii}$ | 0.82 | 1.89 | 2.703 (2) | 169 |
| O2—H2A···O3$^{iv}$ | 0.82 | 2.00 | 2.716 (3) | 145 |

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

**Fig. 1**

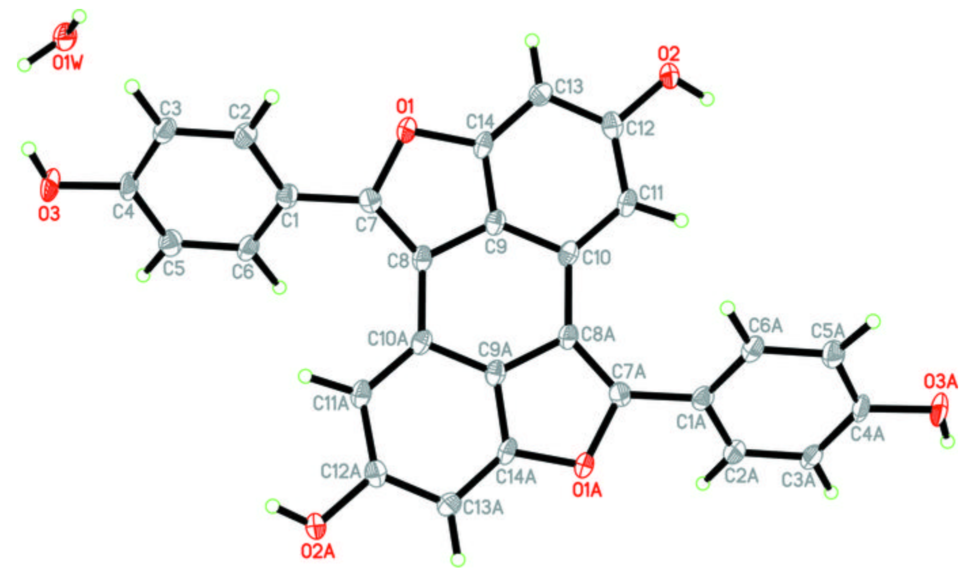

O1W
C3
C2
O1
C14
C13
O2
C12
O3
C4
C1
C7
C11
C8
C9
C10
C5
C6
C10A
C9A
C8A
C6A
C5A
C7A
O3A
C11A
C1A
C4A
C12A
C14A
C2A
C3A
O1A
C13A
O2A

Fig. 2