Crystal structure and Hirshfeld analysis of (1aS,3aR,4aS,5aR)-15-acetoxylinden-7(11),8-trieno-12,8-lactone

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The structure of the title compound, C_{17}H_{20}O_{4} [systematic name: (1aS,3aR,4aS,5aR)-15-(acetoxy)linden-7(11),8-trieno-12,8-lactone or (4aR,S,5aR,6aS,6bR)-5-(acetoxy)methyl]-4a,5,5a,6,6a,6b-hexahydro-3,6b-dimethylcyclopenta[2,3]-indeno[5,6-b]furan-2(4H)-one, ent-chloranthalactone C], a natural product isolated from the whole plant *Chloranthus japonicus* Sieb., is a typical lindenane-type sesquiterpenoid. The molecule comprises a bicyclo[3.1.0]hexane ring (A/B system) bearing an acetoxymethyl (C-4) group, a bicyclo[4.3.0]nonane ring (B/C system) containing a double bond (C-8/9) and a chiral quaternary carbon (C-10), and a 7(11)-en-12,8-olide structural moiety on the cyclohexan-8-ene (C ring). In the tetracyclic skeleton, the 1,3-cyclopropane ring has a β-configuration, and atoms H-5 and H_{3-14} have α- and β-orientations, respectively. In the crystal, the molecules are assembled into a two-dimensional network by weak O···H/H···O interactions. Hirshfeld surface analysis illustrates that the greatest contributions are from H···O (55.2%), O···H/H···O (34.6%) and C···H/H···C (8.9%) contacts.

1. Chemical context

Lindenanolides are precursors for various sesquiterpene dimer derivatives (Uchida *et al.*, 1980; Wang *et al.*, 2009; Shi *et al.*, 2016). Inspired by the clinical application of artemisinin, these compounds have become a products library for screening antimalarial drugs (Dondorp *et al.*, 2010; Zhou *et al.*, 2017). The roots of *Chloranthus japonicus* (called Yinxiancao) were reported to exhibit antifungal and anti-inflammatory activities, and have been used as traditional Chinese medicine to treat malaria (Kawabata & Mizutani, 1989). Chlorantha-lactone C was characterized as an α,β,γ,δ-unsaturated γ-lactone and was converted into desacetyl enol lactone hydrate and ketoalcohol under moderate alkaline conditions (Uchida *et al.*, 1980). Because of the unique stereostructure in lindenane, these lactone derivatives have been studied extensively and serve as precursors for screening cytotoxicity against mouse lymphosarcoma, liver cancer and human cervical cancer cells, the expression of cell adhesion molecules and the mode of antiplasmodial agents (Uchida *et al.*, 1980; Zhang *et al.*, 2012; Zhou *et al.*, 2017). Based on the antiwiggler activity, we are currently searching for a biological pesticide preparation to inhibit flyblow breeding in vegetable production (Shi *et al.*, 2016) and report here the structure of the title compound.

2. Structural commentary

The molecular structure of the title compound is shown in Scheme 1 and Fig. 1. This compound consists of a novel...
C8—C7 are the same at 155.5 (4)°, respectively. The torsion angles C5—C6—C11—C12 and C2—C3—C4—O2, which were 179.9 (3)° and 177.6 (4)°, respectively, indicate the geometric stability of the B/C and C/D ring junctions. In addition, the main A/B/C/D skeleton and the acetoxymethyl system (atoms C15–C17/O3/O4) are not coplanar, the torsion angles C15—O3—C16—C17 and C15—O3—C16—O4 being −175.9 (3)° and 2.8 (6)°, respectively.

| D—H · · · A | D—H | H···A | D···A | D—H · · · A |
|-------------|------|-------|-------|------------|
| C8—H8A···O1i | 0.97 | 2.81  | 3.481 (5) | 127        |
| C11—H11···O3ii | 0.98 | 2.54  | 3.497 (5) | 167        |
| C13—H13C···O1iii | 0.96 | 2.60  | 3.499 (5) | 157        |
| C13—H13B···O4iv | 0.96 | 2.61  | 3.530 (6) | 160        |
| C14—H14A···O2v | 0.96 | 2.76  | 3.530 (5) | 138        |
| C17—H17C···O3vi | 0.96 | 2.86  | 3.478 (5) | 124        |

Symmetry codes: (i) x, y, z; (ii) x + 1, y, z; (iii) x + 1/2, −y + 1/2, −z + 1; (iv) x, y + 1, z; (v) −x + 2, y −1/2, −z + 1/2.

polycyclic framework embedded with a sterically congested cyclopentane ring (B), an unusual trans-5/6 ring junction and an angular methyl group. The chiral quaternary C atom at the 10-position is located on the same side of the B ring plane as the cyclopropane ring and the 4-acetoxymethyl and 5-hydrogen are positioned on the other side. The positions of the substituents can be described as having a β-configuration for the cyclopropane ring at the 1,3-positions, axial for the H atom at the 5-position and bisectional for the methyl H atom at the chiral quaternary C atom in the 10-position. Two cyclic olefinic bonds are located between atoms C2 and C3, and between atoms C4 and C5, and are attached to the cyclohexane (C) and cyclopentanolactone (D) rings, respectively. The torsion angles C9—C10—C11—C12 and C12—C10—C11—C6 of 115.2 (4)° and −115.2 (4)°, respectively, describe the geometric metamerism of the junction between cyclopropane ring A and cyclopentane ring B. The difference in configuration of the oxygen-containing groups can be confirmed by the torsion angles C7—C9—C10—O3 and C1—O1—C1—O2—C4, which were 179.9 (3)° and −179.0 (4)°, respectively. The torsion angles C5—C6—C11—C12 and C2—C3—C8—C7 are the same at 155.5 (4)°, indicating the conformational stability of the A/B and C/D ring junctions. Also, the C2—C3—C4—C5 and C8—C3—C4—O2 torsion angles are 177.1 (4)° and 177.2 (3)°, respectively, and the O2—C1—C2—C4 and C14—C2—C3—C4 torsion angles are 179.9 (3)° and −178.9 (4)°, respectively, and describe the geometric characteristics of the C and D rings. In the title molecule, the central six-membered lindenane sesquiterpenoid ring has a half-chair conformation, with puckering parameters (Cremer & Pople, 1975; Luger & Bülow, 1983) of Q1 = 0.3387 (11) Å, θ = 49.11 (19)° and ψ = 167.3 (2)°. Furthermore, the C9—C7—C8—C3 and C5—C4—O2—C1 torsion angles [−178.6 (3)° and −177.6 (4)°, respectively] indicate the geometric stability of the B/C and C/D ring junctions. In addition, the main A/B/C/D skeleton and the acetoxymethyl system (atoms C15–C17/O3/O4) are not coplanar, the torsion angles C15—O3—C16—C17 and C15—O3—C16—O4 being −175.9 (3)° and 2.8 (6)°, respectively.

3. Supramolecular features

In the crystal of the title compound, the molecules are linked via multiple C—H···O weak hydrogen bonds, generating two-dimensional (2D) layers propagating along the c-axis direction (Fig. 2 and Table 1). Details of the hydrogen-bonding interactions and the symmetry codes are given in Table 1.

4. Hirshfeld surface analysis

Hirshfeld surface analysis was performed and the associated fingerprint plots, providing a 2D view of the intermolecular interactions within the molecular crystals, were generated using CrystalExplorer (Version 21.5; Spackman et al., 2021), with a standard resolution of the three-dimensional (3D) d norm surfaces plotted over a fixed colour scale of −0.1253 (red) to yellow band.

![Figure 1](image_url)

**Figure 1**
The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

![Figure 2](image_url)

**Figure 2**
The packing of molecules in the crystal structure of the title compound, viewed along the c direction (C—H···O hydrogen bonds are shown as green dashed lines).
1.4046 (blue) arbitrary units (Fig. 3). The intense red spots symbolize short contacts and negative $d_{\text{norm}}$ values on the surface are related to the presence of C—H···O hydrogen bonds in the crystal structure. This result corresponds to the results obtained from the solid crystalline structure with the formation of hydrogen bonds. Weak C···H/C/H/C contacts are shown by dim red spots (Fig. 4). The 2D fingerprint plots for the H···H, H···O/O···H, and H···C/C···H contacts are shown in Fig. 5. H···H interactions play an integral role in the overall crystal packing, contributing 55.2%, and are located in the middle region of the fingerprint plot. The most significant H···O/O···H contacts contribute 34.6% to the Hirshfeld surface and the proportion of weak H···C/C···H contacts is 8.9%.

5. Database survey
A search of the Cambridge Structural Database (CSD, Version 5.43, last update November 2021; Groom et al., 2016) for the same carbon ring skeleton as the title compound yielded only one molecule, 5-[(tert-butyldimethylsilyl)oxy]-3,6b-dimethyl-4a,5,5a,6,6a,6b-hexahydrocyclopropane[2,3]indeno[5,6-b]furan-2(4H)-one (CCDC reference 804060; Qian & Zhao, 2011), which has a (tert-butyldimethylsilyl)oxy group attached to ring A of the carbon skeleton.

6. Isolation and crystallization
The title sesquiterpenoid was isolated as a colourless solid from the EtOAc soluble fraction of *C. japonicus* by chromatography over silica gel, and eluted with a mixture of ethyl acetate and hexane (1:20 to 5:1 v/v gradient) to yield the title compound. Crystals were obtained after recrystallization from
acetone or chloroform–methanol (6:1 v/v) at room temperature by slow evaporation over a period of a few days. 1H NMR (500 MHz, chloroform-d): δ 6.22 (1H, s, H-9), 4.20 (2H, d, J = 6.1 Hz, H-11), 2.63 (1H, d, J = 13.0 Hz, H2-21 (2H, m), 2.09 (3H, s, COCH3), 1.87 (3H, br s, H-13), 1.73 (1H, tt, J = 10.1, 4.9 Hz), 1.53 (1H, td, J = 8.1, 3.8 Hz), 1.30 (1H, ddd, J = 11.9, 8.0, 3.7 Hz), 0.91 (1H, d, J = 3.8, 2.1 Hz), 0.89 (3H, s, H-15), 0.83 (1H, td, J = 8.4, 6.0 Hz). 13C NMR (125 MHz, chloroform-d): δ 171.34 (OCOCH3 or C14), 173.11 (OCOCH3 or C12), 149.69 (C-8), 148.41 (C-7), 122.47 (C-11), 120.13 (C-9), 66.23 (C-15), 60.45 (C-5), 43.11 (C-4), 42.15 (C-10), 27.47 (C-1), 22.87 (C-6), 22.48 (C-3), 21.25 (OCOCH3 or C-14), 21.21 (OCOCH3 or C14), 17.15 (C-2), 8.83 (C-13).

7. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were positioned geometrically (C–H = 0.96–0.98 Å) and refined as riding, with Uiso(H) = 1.2Ueq(C) for CH hydrogens or 1.5Ueq(C) for methyl H atoms.

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Crystal structure and Hirshfeld analysis of (1aS,3aR,4aS,5aR)-15-acetoxy-linden-7(11),8-trieno-12,8-lactone

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Computing details

Data collection: SMART (Bruker, 2002); cell refinement: SMART (Bruker, 2002); data reduction: SAINT (Bruker, 2002); program(s) used to solve structure: SHELXT2014 (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2014 (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: publCIF (Westrip, 2010).

(4aR,5S,5aR,6aS,6bR)-5-(Acetoxymethyl)-4a,5,5a,6,6a,6b-hexahydro-3,6b-dimethylcyclopropa[2,3]indeno[5,6-b]furan-2(4H)-one

Crystal data

C_{17}H_{20}O_{4}  
Mr = 288.33
Orthorhombic, P2_{1}2_{1}2_{1}

a = 6.7641 (3) Å  
b = 6.9254 (3) Å  
c = 31.4538 (14) Å

V = 1473.42 (11) Å³  
Z = 4

F(000) = 616

Data collection

Bruker SMART CCD diffractometer  
phi and omega scans  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

2576 independent reflections  
1857 reflections with I > 2σ(I)

Θmax = 25.0°, Θmin = 2.6°  
h = −7→8  
k = −8→6  
l = −32→37

Refinement

Refinement on F²

Least-squares matrix: full  
R[F² > 2σ(F²)] = 0.051  
wR(F²) = 0.117  
S = 1.05

2576 reflections  
193 parameters  
0 restraints

Hydrogen site location: inferred from neighbouring sites  
H-atoms parameters constrained

w = 1/[σ²(Fo²) + (0.0466P)² + 0.380P]  
where P = (Fo² + 2Fc²)/3  
(Δ/σ)max < 0.001  
Δρmax = 0.30 e Å⁻³  
Δρmin = −0.21 e Å⁻³

Absolute structure: Flack x determined using 574 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons et al., 2013)

Absolute structure parameter: 0.10 (8)
**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 (Å²)**

|   | x     | y     | z     | U_iso*/U_eq |
|---|-------|-------|-------|-------------|
| O1| 0.1658 (5) | 0.5144 (4) | 0.54793 (8) | 0.0745 (9) |
| O2| 0.1329 (4)  | 0.5303 (4)  | 0.47683 (7)  | 0.0537 (7)  |
| O3| 0.9151 (4)  | 0.1520 (4)  | 0.31668 (8)  | 0.0566 (8)  |
| O4| 0.9315 (6)  | −0.1600 (5) | 0.32844 (14) | 0.1261 (18) |
| C1| 0.2446 (6)  | 0.5140 (6)  | 0.51366 (12) | 0.0536 (10) |
| C2| 0.4539 (6)  | 0.4960 (5)  | 0.50214 (10) | 0.0480 (9)  |
| C3| 0.4648 (5)  | 0.4991 (5)  | 0.45952 (10) | 0.0418 (8)  |
| C4| 0.2650 (5)  | 0.5215 (5)  | 0.44282 (11) | 0.0442 (9)  |
| C5| 0.2143 (5)  | 0.5386 (5)  | 0.40250 (11) | 0.0447 (10) |
| H5| 0.0829      | 0.5495      | 0.3942      | 0.054*      |
| C6| 0.3810 (5)  | 0.5396 (5)  | 0.37059 (10) | 0.0389 (9)  |
| C7| 0.5465 (5)  | 0.4057 (5)  | 0.38746 (10) | 0.0387 (9)  |
| H7| 0.4802      | 0.2839      | 0.3944      | 0.046*      |
| C8| 0.6345 (5)  | 0.4765 (6)  | 0.42937 (10) | 0.0426 (9)  |
| C9| 0.6749 (5)  | 0.3603 (5)  | 0.34836 (10) | 0.0414 (9)  |
| H9| 0.7586      | 0.4713      | 0.3414      | 0.050*      |
| C10| 0.5169 (6) | 0.3322 (6) | 0.31409 (11) | 0.0503 (10) |
| H10| 0.4988   | 0.2025   | 0.3023   | 0.060*   |
| C11| 0.3340 (6) | 0.4435 (6) | 0.32784 (11) | 0.0506 (11) |
| H11| 0.2047   | 0.3818   | 0.3243   | 0.061*   |
| C12| 0.4461 (6) | 0.5003 (7) | 0.28864 (10) | 0.0613 (11) |
| H12A| 0.3853 | 0.4739 | 0.2613 | 0.074* |
| H12B| 0.5235 | 0.6181 | 0.2897 | 0.074* |
| C13| 0.4477 (6) | 0.7496 (5) | 0.36559 (12) | 0.0523 (11) |
| H13A| 0.3469 | 0.8217 | 0.3511 | 0.078* |
| H13B| 0.5679 | 0.7539 | 0.3494 | 0.078* |
| H13C| 0.4701 | 0.8050 | 0.3932 | 0.078* |
| C14| 0.6127 (7) | 0.4764 (6) | 0.53450 (12) | 0.0650 (12) |
| H14A| 0.6047 | 0.3512 | 0.5475 | 0.098* |
| H14B| 0.5968 | 0.5743 | 0.5558 | 0.098* |
| H14C| 0.7393 | 0.4911 | 0.5211 | 0.098* |
| C15| 0.8003 (6) | 0.1832 (5) | 0.35511 (11) | 0.0488 (10) |
| H15A| 0.8877 | 0.2019 | 0.3792 | 0.059* |
| H15B| 0.7171 | 0.0720 | 0.3608 | 0.059* |
| C16| 0.9745 (5) | −0.0230 (6) | 0.30723 (13) | 0.0550 (10) |
| C17| 1.1017 (6) | −0.0305 (7) | 0.26893 (12) | 0.0660 (12) |
| H17A| 1.2179 | 0.0462 | 0.2735 | 0.099* |
| H17B  | 1.0301 | 0.0192 | 0.2450 | 0.099* |
|-------|--------|--------|--------|--------|
| H17C  | 1.1394 | −0.1619| 0.2635 | 0.099* |

### Atomic displacement parameters (\(\AA^2\))

|        | \(U_1^{11}\) | \(U_2^{12}\) | \(U_3^{13}\) | \(U_4^{14}\) | \(U_5^{15}\) |
|--------|---------------|---------------|---------------|---------------|---------------|
| O1     | 0.106 (2)     | 0.074 (2)     | 0.0437 (16)   | 0.017 (2)     | 0.0283 (16)   |
| O2     | 0.0592 (16)   | 0.0591 (17)   | 0.0427 (15)   | 0.0035 (15)   | 0.0182 (13)   |
| O3     | 0.076 (2)     | 0.0422 (16)   | 0.0520 (16)   | 0.0031 (15)   | 0.0267 (16)   |
| O4     | 0.137 (4)     | 0.058 (2)     | 0.184 (4)     | 0.013 (2)     | 0.101 (3)     |
| C1     | 0.080 (3)     | 0.040 (2)     | 0.040 (2)     | 0.013 (2)     | 0.014 (3)     |
| C2     | 0.069 (3)     | 0.036 (2)     | 0.039 (2)     | 0.002 (2)     | 0.0037 (19)   |
| C3     | 0.054 (2)     | 0.0344 (19)   | 0.0366 (19)   | 0.000 (2)     | 0.0037 (17)   |
| C4     | 0.049 (2)     | 0.043 (2)     | 0.040 (2)     | 0.000 (2)     | 0.0113 (18)   |
| C5     | 0.038 (2)     | 0.052 (2)     | 0.044 (2)     | −0.003 (2)    | 0.0027 (17)   |
| C6     | 0.0395 (19)   | 0.045 (2)     | 0.0324 (18)   | −0.0053 (18)  | 0.0005 (16)   |
| C7     | 0.042 (2)     | 0.040 (2)     | 0.0340 (19)   | −0.0054 (18)  | 0.0046 (17)   |
| C8     | 0.044 (2)     | 0.047 (2)     | 0.0366 (18)   | −0.003 (2)    | −0.0007 (16)  |
| C9     | 0.045 (2)     | 0.041 (2)     | 0.039 (2)     | −0.0055 (18)  | 0.0052 (18)   |
| C10    | 0.054 (2)     | 0.058 (3)     | 0.039 (2)     | −0.005 (2)    | 0.005 (2)     |
| C11    | 0.044 (2)     | 0.068 (3)     | 0.040 (2)     | −0.011 (2)    | 0.0019 (18)   |
| C12    | 0.061 (2)     | 0.091 (3)     | 0.0322 (19)   | −0.002 (3)    | −0.0002 (18)  |
| C13    | 0.057 (3)     | 0.048 (2)     | 0.052 (2)     | −0.006 (2)    | 0.000 (2)     |
| C14    | 0.093 (3)     | 0.058 (3)     | 0.044 (2)     | 0.004 (3)     | −0.006 (2)    |
| C15    | 0.062 (2)     | 0.046 (2)     | 0.039 (2)     | −0.001 (2)    | 0.015 (2)     |
| C16    | 0.047 (2)     | 0.048 (3)     | 0.070 (3)     | −0.006 (2)    | 0.013 (2)     |
| C17    | 0.063 (3)     | 0.071 (3)     | 0.064 (3)     | 0.006 (3)     | 0.014 (2)     |

### Geometric parameters (\(\AA\), °)

|        | O1—C1 | 1.202 (4) | C9—C15 | 1.507 (5) |
|--------|-------|-----------|--------|-----------|
|        | O2—C1 | 1.388 (4) | C9—C10 | 1.530 (5) |
|        | O2—C4 | 1.396 (4) | C9—H9  | 0.9800    |
|        | O3—C16| 1.311 (5) | C10—C12| 1.492 (6) |
|        | O3—C15| 1.453 (4) | C10—C11| 1.520 (5) |
|        | O4—C16| 1.196 (5) | C10—H10| 0.9800    |
|        | C1—C2 | 1.467 (6) | C11—C12| 1.500 (5) |
|        | C2—C3 | 1.343 (4) | C11—H11| 0.9800    |
|        | C2—C4 | 1.486 (5) | C12—H12A| 0.9700   |
|        | C3—C4 | 1.458 (5) | C12—H12B| 0.9700   |
|        | C3—C8 | 1.497 (5) | C13—H13A| 0.9600   |
|        | C4—C5 | 1.319 (5) | C13—H13B| 0.9600   |
|        | C5—C6 | 1.509 (5) | C13—H13C| 0.9600   |
|        | C5—H5 | 0.9300   | C14—H14A| 0.9600   |
|        | C6—C13| 1.531 (5) | C14—H14B| 0.9600   |
|        | C6—C11| 1.534 (5) | C14—H14C| 0.9600   |
|        | C6—C7 | 1.548 (5) | C15—H15A| 0.9700   |
|        | C7—C8 | 1.528 (4) | C15—H15B| 0.9700   |
C7—C9  1.538 (5)  C16—C17  1.481 (5)
C7—H7  0.9800  C17—H17A  0.9600
C8—H8A  0.9700  C17—H17B  0.9600
C8—H8B  0.9700  C17—H17C  0.9600
C1—O2—C4  106.7 (3)  C11—C10—C9  107.7 (3)
C16—O3—C15  119.3 (3)  C12—C10—H10  118.1
O1—C1—O2  120.4 (4)  C11—C10—H10  118.1
O1—C1—C2  130.5 (4)  C9—C10—H10  118.1
O2—C1—C2  109.0 (3)  C12—C11—C10  59.2 (3)
C3—C2—C1  107.4 (3)  C12—C11—C6  120.1 (3)
C3—C2—C14  130.2 (4)  C10—C11—C6  107.5 (3)
C1—C2—C14  122.4 (3)  C12—C11—H11  118.2
C2—C3—C4  108.1 (3)  C10—C11—H11  118.2
C2—C3—C8  132.3 (3)  C6—C11—H11  118.2
C4—C3—C8  119.6 (3)  C10—C12—C11  61.1 (3)
C5—C4—O2  124.5 (3)  C10—C12—H12A  117.7
C5—C4—C3  126.6 (3)  C11—C12—H12A  117.7
O2—C4—C3  108.8 (3)  C10—C12—H12B  117.7
C4—C5—C6  116.5 (3)  C11—C12—H12B  117.7
C4—C5—H5  121.8  H12A—C12—H12B  114.8
C5—C6—C13  107.0 (3)  C6—C13—C14  109.5
C5—C6—C7  115.2 (3)  C6—C13—H13A  109.5
C13—C6—C7  112.5 (3)  C6—C13—H13B  109.5
C5—C6—C11  108.0 (3)  H13A—C13—H13B  109.5
C13—C6—C11  113.1 (3)  H13A—C13—H13C  109.5
C11—C6—C13  101.0 (3)  H13B—C13—H13C  109.5
C8—C7—C9  122.4 (3)  C2—C14—H14A  109.5
C8—C7—C6  112.7 (3)  C2—C14—H14B  109.5
C9—C7—C6  104.9 (3)  H14A—C14—H14B  109.5
C8—C7—H7  105.2  H14A—C14—H14C  109.5
C9—C7—H7  105.2  H14B—C14—H14C  109.5
C6—C7—H7  105.2  O3—C15—C9  107.7 (3)
C3—C8—C7  106.3 (3)  O3—C15—C10  110.2
C3—C8—H8A  110.5  C9—C15—H15A  110.2
C7—C8—H8A  110.5  O3—C15—H15B  110.2
C3—C8—H8B  110.5  C9—C15—H15B  110.2
C7—C8—H8B  110.5  H15A—C15—H15B  108.5
H8A—C8—H8B  108.7  O4—C16—O3  122.2 (4)
C15—C9—C10  112.9 (3)  O4—C16—C17  124.5 (4)
C15—C9—C7  111.8 (3)  O3—C16—C17  113.3 (4)
C10—C9—C7  101.2 (3)  C16—C17—H17A  109.5
C15—C9—H9  110.2  C16—C17—H17B  109.5
C10—C9—H9  110.2  H17A—C17—H17B  109.5
C7—C9—H9  110.2  C16—C17—H17C  109.5
C12—C10—C11  59.7 (3)  H17A—C17—H17C  109.5
C12—C10—C9  120.2 (4)  H17B—C17—H17C  109.5
C4—O2—C1—O1 −178.9 (4) C4—C3—C8—C7 −21.5 (5)
C4—O2—C1—C2 0.5 (4) C9—C7—C8—C3 −178.6 (3)
O1—C1—C2—C3 178.5 (4) C6—C7—C8—C3 55.0 (4)
O2—C1—C2—C3 −0.8 (5) C8—C7—C9—C15 69.2 (4)
O1—C1—C2—C14 −0.9 (7) C6—C7—C9—C15 −161.0 (3)
O2—C1—C2—C14 179.8 (3) C8—C7—C9—C10 −170.4 (3)
C1—C2—C3—C4 0.7 (5) C6—C7—C9—C10 −40.5 (3)
C14—C2—C3—C4 −179.9 (4) C15—C9—C10—C12 −151.0 (3)
C1—C2—C3—C8 −176.5 (4) C7—C9—C10—C12 89.3 (4)
C14—C2—C3—C8 2.9 (7) C15—C9—C10—C11 144.3 (3)
C1—O2—C4—C3 0.0 (4) C7—C9—C10—C11 24.6 (4)
C1—O2—C4—C5 −0.9 (7) C9—C10—C11—C12 115.2 (4)
C2—C3—C4—C5 177.1 (4) C12—C10—C11—C12 −115.2 (4)
C8—C3—C4—C5 −5.3 (6) C9—C10—C11—C6 0.0 (4)
C2—C3—C4—O2 −0.4 (4) C5—C6—C11—C12 155.4 (4)
C8—C3—C4—O2 177.2 (3) C13—C6—C11—C12 32.3 (5)
O2—C4—C5—C6 175.6 (3) C6—C11—C12—C10 −140.6 (3)
C3—C4—C5—C6 −1.5 (6) C5—C6—C11—C10 14.6 (4)
C4—C5—C6—C7 33.5 (4) C9—C10—C11—C6 93.8 (4)
C5—C6—C7—C8 −62.8 (4) C6—C11—C12—C10 93.4 (4)
C13—C6—C7—C8 55.4 (4) C16—O3—C15—C9 −153.7 (3)
C11—C6—C7—C8 175.8 (3) C10—C9—C15—O3 66.5 (4)
C5—C6—C7—C9 161.8 (3) C7—C9—C15—O3 179.9 (3)
C13—C6—C7—C9 −80.0 (3) C15—O3—C16—O4 2.8 (6)
C11—C6—C7—C9 40.5 (3) C15—O3—C16—C17 −175.9 (3)
C2—C3—C8—C7 155.5 (4)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|-------|---------|
| C8—H8· ·O1ⅰ | 0.97 | 2.81 | 3.481 (5) | 127 |
| C11—H11···O3ⅱ | 0.98 | 2.54 | 3.497 (5) | 167 |
| C13—H13C···O1ⅲ | 0.96 | 2.60 | 3.499 (5) | 157 |
| C13—H13B···O4ⅳ | 0.96 | 2.61 | 3.530 (6) | 160 |
| C14—H14A···O2ⅹ | 0.96 | 2.76 | 3.530 (5) | 138 |
| C17—H17C···O3ⅹⅰ | 0.96 | 2.86 | 3.478 (5) | 124 |

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