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# Crystal structure of (1R,3S,8R,11R)-11-acetyl-3,7,7-trimethyl-10-oxatricyclo[6.4.0.0$^{1,3}$]dodecan-9-one

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The title compound, $C_{16}H_{24}O_3$, is built up from three fused rings, a six-membered, a seven-membered and a three-membered ring. The absolute configuration of the title compound was determined as (1R,3S,8R,11R) based on the synthetic pathway. The six-membered ring has an half-chair conformation whereas the seven-membered ring displays a boat conformation. In the cyrstal, C—H···O hydrogen bonds build up a two-dimensional network parallel to (0 0 1). The crystal studied was an inversion twin with a minor twin component of 34%.

## 1. Related literature

For biological activities of terpenic lactones, see: Hall *et al.* (1987); Ohnishi *et al.* (1997); Ghosh & Karin (2002); Bremner & Heinrich (2002); Francois *et al.* (1996); Rabe *et al.* (2002); Calera *et al.* (1995). For the synthesis, see: Bimoussa *et al.* (2014). For the ring puckering parameters, see: Boessenkool & Boyens (1980). For the absolute configuration, see: Parsons *et al.* (2013); Hooft *et al.* (2008). For the refinement of twined crystals, see: Cooper *et al.* (2002).
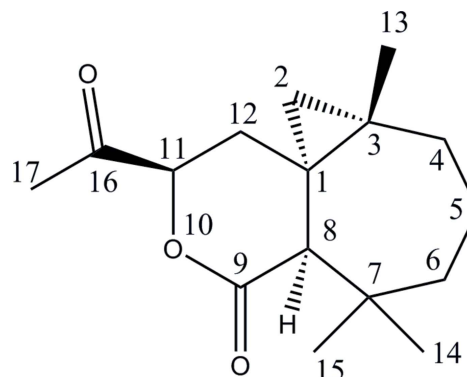
## 2. Experimental

### 2.1. Crystal data

$C_{16}H_{24}O_3$
$M_r = 264.35$
Monoclinic, $P2_1$
$a = 6.4443$ (6) Å
$b = 8.4437$ (7) Å
$c = 13.7083$ (12) Å
$\beta = 98.654$ (9)°

$V = 737.43$ (11) Å$^3$
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.08$ mm$^{-1}$
$T = 180$ K
$0.52 \times 0.45 \times 0.25$ mm

### 2.2. Data collection

Agilent Xcalibur (Eos, Gemini ultra) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014). $T_{min} = 0.717$, $T_{max} = 1.000$

7878 measured reflections
7878 independent reflections
7173 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.141$
$S = 1.05$
7878 reflections
177 parameters

1 restraint
H-atom parameters constrained
$\Delta\rho_{max} = 0.31$ e Å$^{-3}$
$\Delta\rho_{min} = -0.34$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C11—H11A···O2[i] | 0.99 | 2.40 | 3.184 (6) | 136 |
| C16—H16C···O2[ii] | 0.98 | 2.37 | 3.262 (8) | 152 |

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL2013*.

# data reports

## References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Abingdon, England.

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.

Bimoussa, A., Auhmani, A., Ait Itto, M. Y., Daran, J.-C. & Auhmani, A. (2014). *Acta Cryst.* E**70**, o480.

Boessenkool, I. K. & Boeyens, J. C. A. (1980). *J. Cryst. Mol. Struct.* **10**, 11–18.

Bremner, P. & Heinrich, M. (2002). *J. Pharm. Pharmacol.* **54**, 453–472.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Calera, M. R., Soto, F., Sanchez, P., Bye, R., Hernandez-Bautista, B. B., Anaya, A. L., Lotina-Hennsen, B. & Mata, R. (1995). *Phytochemistry*, **40**, 419–425.

Cooper, R. I., Gould, R. O., Parsons, S. & Watkin, D. J. (2002). *J. Appl. Cryst.* **35**, 168–174.

Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.

François, G., Passreiter, C. M., Woerdenbag, H. J. & Van Looveren, M. (1996). *Planta Med.* **62**, 126–129.

Ghosh, S. & Karin, M. (2002). *Cell*, **109**, S81–S96.

Hall, I. H., Grippo, A. A., Lee, K. H., Chaney, S. G. & Holbrook, D. J. (1987). *Pharm. Res.* **4**, 509–514.

Hooft, R. W. W., Straver, L. H. & Spek, A. L. (2008). *J. Appl. Cryst.* **41**, 96–103.

Ohnishi, M., Yoshimi, N., Kawamori, T., Ino, N., Hirose, Y., Tanaka, T., Yamahara, J., Miyata, H. & Mori, H. (1997). *Jpn. J. Cancer Res.* **88**, 111–119.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Rabe, T., Mullholland, D. & van Staden, J. (2002). *J. Ethnopharmacol.* **80**, 91–94.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

# supporting information

## Crystal structure of (1*R*,3*S*,8*R*,11*R*)-11-acetyl-3,7,7-trimethyl-10-oxatricyclo-[6.4.0.0$^{1,3}$]dodecan-9-one

**Abdoullah Bismoussa, My Youssef Ait Itto, Jean-Claude Daran, Abdelwahed Auhmani and Aziz Auhmani**

### S1. Chemical context

\ In recent years many terpenic lactones were shown to exhibit broad spectrum of biological activities such anticancer activity (Hall *et al.*, 1987, Ohnishi *et al.*, 1997), anti-inflammatory activity (Ghosh & Karin, 2002; Bremner & Heinrich, 2002), anti-malarial activity (Francois *et al.*, 1996), antiviral activity, antibacterial activity (Rabe *et al.*, 2002) and anti-fungal activity (Calera *et al.*, 1995).

Their structural diversity and potential biological activities have made further interest for the drug discovery research. Thus, In order to prepare new lactones using natural products, we have prepared (1R,3S,8R,11R)-11-acetyl-3,7,7-tri-methyl-10-oxatricyclo[6.4.0.0$^{1,3}$]dodecan-\ 9-one from *β*-himachalène (sesquiterpenic hydrocarbon). The title compound was prepared by an oxidative cleavage of (1S,3S,8R,9S,10R)-9,10-Epoxy-3,7,7,10-tetramethyltricyclo[6.4.0.0$^{1,3}$]\ do-decane (Bimoussa *et al.*, 2014) using periodic acid as oxydant.

### S2. Structural commentary

The compound is built up from three fused rings, a six membered and a seven membered rings; these last ring is fused with a three membered ring (Fig. 1). The six-membered-ring has an half chair conformation with puckering parameters: Q = 0.469 (3) Å, $\theta$ = 39.3 (4)° and $\varphi$ = 213.6 (6)°, whereas the seven-membered ring displays a boat conformation with puckering amplitudes: Q2 = 1.130 (4) and Q3 =0.044 (4) (Boessenkool & Boyens, 1980).

The absolute configuration (1S,3R,8S,10S) is deduced from the chemical pathway. The refinement of the Flack's parameter (-0.0 (10)) (Parsons *et al.*, 2013) as well as the Hooft's parameter (Hooft *et al.*, 2008) do not allow to define reliably the absolute configuration.

### S3. Supramolecular features

There are weak C—H···O hydrogen bonds building a two dimensional network parallel to the (0 0 1) plane (Fig. 2).

### S4. Database survey

A search of the Cambridge Structural Database gave no hits with related structures.

### S5. Synthesis and crystallization

\ In 100 ml flask containing (0.120g, (0.515 mmol) of (1*S*,3*S*,8*R*,9*S*,10*R*)-9,10-Epoxy-3,7,7,10-\ tetramethyltricyclo-[6.4.0.0$^{1,3}$]dodecane in 6ml of CCl$_4$, 6 ml of acetonitrile and 6ml of watter was added 0.427 g (2.06 mmol) of periodic acid (*NaIO$_4$*) and 4.70 mg (0.0179 mmol) of RuCl$_3$,3H$_2$O ( 3.5%). The reaction mixture was stirred at 0°C for 20 min and for 24h at room temperature. The reaction mixture was extracted with dichloromethane (3x 20ml) and the organic layer were dried over Na$_2$SO$_4$ and concentrated under reduced pressure. The crude product was purified by chromatography on

silica gel (230–400 mesh) with Hexane/ethyl acetate (85:15) as eluent to give the title compound (1R,3S,8R,11R)-11-acetyl-3,7,7-trimethyl-10-oxatricyclo[6.4.0.0$^{1,3}$]dodecan-\ 9-one in 46% yield. X-ray quality crystals were obtained by slow evaporation from a petroleum ether solution of the title compound.

**S6. Refinement details**

The crystal is twinned and has been refined as a 2-component twin with the following matrix using ROTAX (Parsons 1 Gould; Cooper *et al.*, 2002): 180.0 degree rotation about 1. 0. 0. direct lattice direction: [ 1.000 0.000 0.000] [ 0.000 -1.000 0.000] [ -0.640 0.000 -1.000] BASF = 0.34
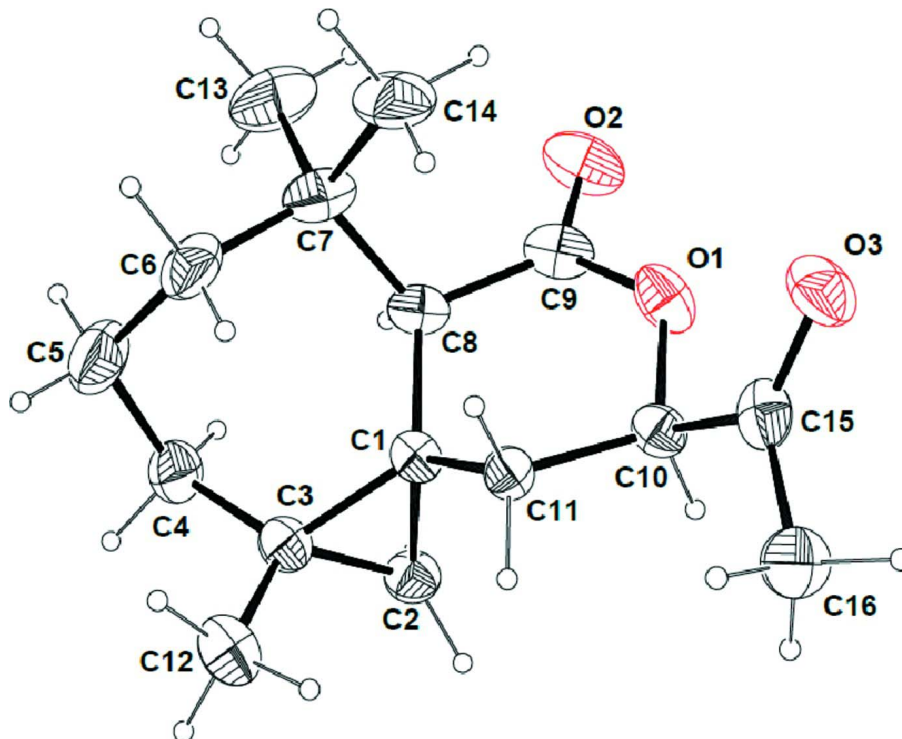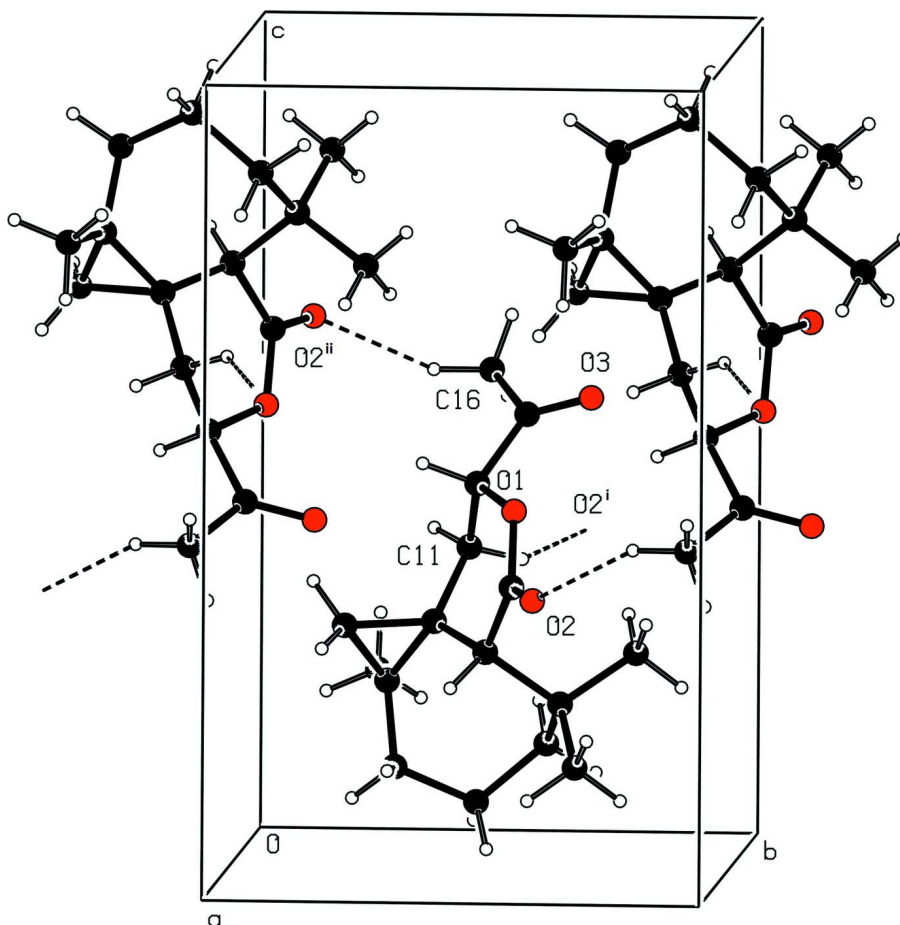


**Figure 1**

Molecular view of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circle of arbitrary radii.

**Figure 2**

Packing view showing the C—H···O hydrogen bonds building a two-dimensional network. H bonds are shown as dashed lines. [Symmetry codes: (i) $x + 1$, $y$, $z$; (ii) $-x + 1$, $y + 1/2$, $-z + 1$].

**(1*R*,3*S*,8*R*,11*R*)-11-Acetyl-3,7,7-trimethyl-10-oxatricyclo[6.4.0.0¹,³]dodecan-9-one**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{24}O_3$ | $F(000) = 288$ |
| $M_r = 264.35$ | $D_x = 1.191$ Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 6.4443$ (6) Å | Cell parameters from 2697 reflections |
| $b = 8.4437$ (7) Å | $\theta = 4.0–28.4°$ |
| $c = 13.7083$ (12) Å | $\mu = 0.08$ mm$^{-1}$ |
| $\beta = 98.654$ (9)° | $T = 180$ K |
| $V = 737.43$ (11) Å$^3$ | Box, colourless |
| $Z = 2$ | $0.52 \times 0.45 \times 0.25$ mm |

*Data collection*

| | |
|---|---|
| Agilent Xcalibur (Eos, Gemini ultra) diffractometer | Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014). |
| Radiation source: Enhance (Mo) X-ray Source | $T_{min} = 0.717$, $T_{max} = 1.000$ |
| Graphite monochromator | 7878 measured reflections |
| Detector resolution: 16.1978 pixels mm$^{-1}$ | 7878 independent reflections |
| $\omega$ scans | 7173 reflections with $I > 2\sigma(I)$ |

$R_{int} = 0.041$

$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$

$h = -8 \rightarrow 8$

$k = -10 \rightarrow 10$

$l = -17 \rightarrow 16$

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from |
| Least-squares matrix: full | neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.061$ | H-atom parameters constrained |
| $wR(F^2) = 0.141$ | $w = 1/[\sigma^2(F_o^2) + (0.0238P)^2 + 0.9242P]$ |
| $S = 1.05$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 7878 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 177 parameters | $\Delta\rho_{max} = 0.31$ e Å$^{-3}$ |
| 1 restraint | $\Delta\rho_{min} = -0.34$ e Å$^{-3}$ |

*Special details*

**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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.2887 (8) | 0.3804 (6) | 0.2813 (4) | 0.0237 (11) |
| C2 | 0.3418 (9) | 0.2072 (6) | 0.2798 (4) | 0.0297 (12) |
| H2A | 0.2894 | 0.1378 | 0.3289 | 0.036* |
| H2B | 0.4811 | 0.1776 | 0.2634 | 0.036* |
| C3 | 0.1834 (8) | 0.2745 (7) | 0.1985 (4) | 0.0274 (12) |
| C4 | 0.2531 (10) | 0.2989 (7) | 0.0992 (4) | 0.0359 (14) |
| H4A | 0.2061 | 0.2079 | 0.0559 | 0.043* |
| H4B | 0.4084 | 0.3023 | 0.1078 | 0.043* |
| C5 | 0.1648 (11) | 0.4526 (8) | 0.0492 (4) | 0.0482 (17) |
| H5A | 0.0218 | 0.4315 | 0.0140 | 0.058* |
| H5B | 0.2534 | 0.4840 | −0.0006 | 0.058* |
| C6 | 0.1541 (10) | 0.5920 (7) | 0.1208 (4) | 0.0402 (15) |
| H6A | 0.1000 | 0.6852 | 0.0811 | 0.048* |
| H6B | 0.0487 | 0.5650 | 0.1637 | 0.048* |
| C7 | 0.3540 (9) | 0.6425 (7) | 0.1873 (4) | 0.0378 (14) |
| C8 | 0.4454 (8) | 0.5023 (6) | 0.2584 (4) | 0.0278 (13) |
| H8 | 0.5466 | 0.4443 | 0.2228 | 0.033* |
| C9 | 0.5737 (8) | 0.5692 (7) | 0.3498 (4) | 0.0319 (13) |
| C10 | 0.3256 (8) | 0.4700 (7) | 0.4541 (4) | 0.0266 (12) |
| H10 | 0.3797 | 0.3666 | 0.4830 | 0.032* |
| C11 | 0.1686 (8) | 0.4366 (6) | 0.3621 (3) | 0.0234 (11) |
| H11A | 0.0888 | 0.5340 | 0.3408 | 0.028* |
| H11B | 0.0680 | 0.3541 | 0.3762 | 0.028* |
| C12 | −0.0452 (8) | 0.2292 (7) | 0.1912 (4) | 0.0371 (15) |
| H12A | −0.0664 | 0.1231 | 0.1623 | 0.056* |
| H12B | −0.1315 | 0.3057 | 0.1493 | 0.056* |
| H12C | −0.0860 | 0.2292 | 0.2572 | 0.056* |

| | | | | |
|---|---|---|---|---|
| C13 | 0.5222 (11) | 0.6924 (9) | 0.1249 (5) | 0.061 (2) |
| H13A | 0.6470 | 0.7301 | 0.1684 | 0.091* |
| H13B | 0.4669 | 0.7775 | 0.0797 | 0.091* |
| H13C | 0.5597 | 0.6014 | 0.0868 | 0.091* |
| C14 | 0.2968 (11) | 0.7879 (7) | 0.2460 (5) | 0.0483 (17) |
| H14A | 0.2497 | 0.8743 | 0.2002 | 0.072* |
| H14B | 0.4204 | 0.8221 | 0.2917 | 0.072* |
| H14C | 0.1839 | 0.7596 | 0.2834 | 0.072* |
| C15 | 0.2174 (8) | 0.5569 (7) | 0.5301 (4) | 0.0301 (12) |
| C16 | 0.0480 (9) | 0.4660 (7) | 0.5691 (4) | 0.0372 (14) |
| H16A | −0.0855 | 0.4816 | 0.5254 | 0.056* |
| H16B | 0.0349 | 0.5037 | 0.6355 | 0.056* |
| H16C | 0.0836 | 0.3531 | 0.5718 | 0.056* |
| O1 | 0.5017 (6) | 0.5651 (5) | 0.4367 (3) | 0.0370 (10) |
| O2 | 0.7419 (6) | 0.6310 (6) | 0.3489 (3) | 0.0476 (11) |
| O3 | 0.2640 (7) | 0.6912 (5) | 0.5555 (3) | 0.0415 (10) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.021 (3) | 0.023 (3) | 0.027 (3) | 0.002 (2) | 0.003 (2) | −0.003 (2) |
| C2 | 0.034 (3) | 0.023 (3) | 0.033 (3) | 0.003 (2) | 0.006 (3) | −0.001 (3) |
| C3 | 0.029 (3) | 0.025 (3) | 0.029 (3) | 0.002 (2) | 0.005 (2) | −0.005 (2) |
| C4 | 0.044 (3) | 0.037 (4) | 0.027 (3) | 0.000 (3) | 0.007 (3) | −0.005 (3) |
| C5 | 0.069 (5) | 0.048 (4) | 0.028 (3) | 0.006 (4) | 0.009 (3) | 0.005 (3) |
| C6 | 0.053 (4) | 0.032 (3) | 0.034 (3) | 0.012 (3) | 0.002 (3) | 0.011 (3) |
| C7 | 0.043 (3) | 0.027 (3) | 0.047 (3) | 0.004 (3) | 0.019 (3) | 0.006 (3) |
| C8 | 0.026 (3) | 0.024 (3) | 0.037 (3) | 0.000 (2) | 0.014 (3) | −0.003 (2) |
| C9 | 0.018 (2) | 0.027 (3) | 0.052 (3) | 0.004 (2) | 0.010 (3) | −0.002 (3) |
| C10 | 0.026 (3) | 0.023 (3) | 0.030 (3) | 0.001 (2) | 0.003 (2) | −0.003 (2) |
| C11 | 0.022 (2) | 0.027 (3) | 0.022 (2) | −0.002 (2) | 0.007 (2) | −0.002 (2) |
| C12 | 0.031 (3) | 0.039 (4) | 0.040 (3) | 0.001 (3) | 0.002 (3) | −0.009 (3) |
| C13 | 0.067 (5) | 0.056 (5) | 0.067 (4) | −0.008 (4) | 0.032 (4) | 0.019 (4) |
| C14 | 0.059 (4) | 0.021 (3) | 0.064 (4) | 0.006 (3) | 0.008 (4) | 0.004 (3) |
| C15 | 0.031 (3) | 0.030 (3) | 0.027 (3) | 0.013 (3) | −0.003 (2) | −0.001 (3) |
| C16 | 0.041 (3) | 0.036 (3) | 0.038 (3) | 0.001 (3) | 0.018 (3) | −0.006 (3) |
| O1 | 0.0256 (19) | 0.043 (2) | 0.041 (2) | −0.004 (2) | 0.0022 (18) | −0.013 (2) |
| O2 | 0.023 (2) | 0.044 (3) | 0.077 (3) | −0.008 (2) | 0.010 (2) | −0.011 (2) |
| O3 | 0.050 (3) | 0.030 (2) | 0.043 (2) | 0.001 (2) | 0.003 (2) | −0.012 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.503 (7) | C9—O2 | 1.205 (6) |
| C1—C8 | 1.507 (7) | C9—O1 | 1.343 (6) |
| C1—C11 | 1.520 (7) | C10—O1 | 1.438 (6) |
| C1—C3 | 1.523 (7) | C10—C11 | 1.519 (7) |
| C2—C3 | 1.505 (7) | C10—C15 | 1.527 (7) |
| C2—H2A | 0.9900 | C10—H10 | 1.0000 |

| | | | |
|---|---|---|---|
| C2—H2B | 0.9900 | C11—H11A | 0.9900 |
| C3—C12 | 1.511 (7) | C11—H11B | 0.9900 |
| C3—C4 | 1.511 (7) | C12—H12A | 0.9800 |
| C4—C5 | 1.537 (9) | C12—H12B | 0.9800 |
| C4—H4A | 0.9900 | C12—H12C | 0.9800 |
| C4—H4B | 0.9900 | C13—H13A | 0.9800 |
| C5—C6 | 1.541 (8) | C13—H13B | 0.9800 |
| C5—H5A | 0.9900 | C13—H13C | 0.9800 |
| C5—H5B | 0.9900 | C14—H14A | 0.9800 |
| C6—C7 | 1.523 (8) | C14—H14B | 0.9800 |
| C6—H6A | 0.9900 | C14—H14C | 0.9800 |
| C6—H6B | 0.9900 | C15—O3 | 1.211 (7) |
| C7—C13 | 1.537 (8) | C15—C16 | 1.498 (8) |
| C7—C14 | 1.542 (8) | C16—H16A | 0.9800 |
| C7—C8 | 1.590 (7) | C16—H16B | 0.9800 |
| C8—C9 | 1.503 (7) | C16—H16C | 0.9800 |
| C8—H8 | 1.0000 | | |
| | | | |
| C2—C1—C8 | 120.0 (5) | C1—C8—H8 | 105.9 |
| C2—C1—C11 | 117.1 (5) | C7—C8—H8 | 105.9 |
| C8—C1—C11 | 111.7 (4) | O2—C9—O1 | 116.9 (5) |
| C2—C1—C3 | 59.6 (3) | O2—C9—C8 | 122.5 (5) |
| C8—C1—C3 | 118.9 (5) | O1—C9—C8 | 120.6 (4) |
| C11—C1—C3 | 120.5 (4) | O1—C10—C11 | 114.2 (4) |
| C1—C2—C3 | 60.8 (4) | O1—C10—C15 | 107.3 (4) |
| C1—C2—H2A | 117.7 | C11—C10—C15 | 109.9 (4) |
| C3—C2—H2A | 117.7 | O1—C10—H10 | 108.4 |
| C1—C2—H2B | 117.7 | C11—C10—H10 | 108.4 |
| C3—C2—H2B | 117.7 | C15—C10—H10 | 108.4 |
| H2A—C2—H2B | 114.8 | C10—C11—C1 | 108.3 (4) |
| C2—C3—C12 | 120.0 (5) | C10—C11—H11A | 110.0 |
| C2—C3—C4 | 117.3 (5) | C1—C11—H11A | 110.0 |
| C12—C3—C4 | 113.2 (5) | C10—C11—H11B | 110.0 |
| C2—C3—C1 | 59.5 (3) | C1—C11—H11B | 110.0 |
| C12—C3—C1 | 121.3 (4) | H11A—C11—H11B | 108.4 |
| C4—C3—C1 | 115.6 (5) | C3—C12—H12A | 109.5 |
| C3—C4—C5 | 112.1 (5) | C3—C12—H12B | 109.5 |
| C3—C4—H4A | 109.2 | H12A—C12—H12B | 109.5 |
| C5—C4—H4A | 109.2 | C3—C12—H12C | 109.5 |
| C3—C4—H4B | 109.2 | H12A—C12—H12C | 109.5 |
| C5—C4—H4B | 109.2 | H12B—C12—H12C | 109.5 |
| H4A—C4—H4B | 107.9 | C7—C13—H13A | 109.5 |
| C4—C5—C6 | 114.2 (4) | C7—C13—H13B | 109.5 |
| C4—C5—H5A | 108.7 | H13A—C13—H13B | 109.5 |
| C6—C5—H5A | 108.7 | C7—C13—H13C | 109.5 |
| C4—C5—H5B | 108.7 | H13A—C13—H13C | 109.5 |
| C6—C5—H5B | 108.7 | H13B—C13—H13C | 109.5 |
| H5A—C5—H5B | 107.6 | C7—C14—H14A | 109.5 |

| | | | |
|---|---|---|---|
| C7—C6—C5 | 118.7 (5) | C7—C14—H14B | 109.5 |
| C7—C6—H6A | 107.6 | H14A—C14—H14B | 109.5 |
| C5—C6—H6A | 107.6 | C7—C14—H14C | 109.5 |
| C7—C6—H6B | 107.6 | H14A—C14—H14C | 109.5 |
| C5—C6—H6B | 107.6 | H14B—C14—H14C | 109.5 |
| H6A—C6—H6B | 107.1 | O3—C15—C16 | 122.7 (5) |
| C6—C7—C13 | 110.3 (5) | O3—C15—C10 | 121.8 (5) |
| C6—C7—C14 | 106.7 (5) | C16—C15—C10 | 115.4 (5) |
| C13—C7—C14 | 108.4 (5) | C15—C16—H16A | 109.5 |
| C6—C7—C8 | 111.1 (5) | C15—C16—H16B | 109.5 |
| C13—C7—C8 | 108.5 (5) | H16A—C16—H16B | 109.5 |
| C14—C7—C8 | 111.6 (5) | C15—C16—H16C | 109.5 |
| C9—C8—C1 | 112.6 (4) | H16A—C16—H16C | 109.5 |
| C9—C8—C7 | 109.7 (5) | H16B—C16—H16C | 109.5 |
| C1—C8—C7 | 116.0 (4) | C9—O1—C10 | 123.2 (4) |
| C9—C8—H8 | 105.9 | | |

*Hydrogen-bond geometry (Å, º)*

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C11—H11$A$⋯O2[i] | 0.99 | 2.40 | 3.184 (6) | 136 |
| C16—H16$C$⋯O2[ii] | 0.98 | 2.37 | 3.262 (8) | 152 |

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