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# (3*S*,3a*S*,5a*S*,7*S*,8*S*,10a*S*,10b*R*)-7,8-Dihydroxy-3-isopropyl-5a,8-dimethyl-2,3,4,5,5a,6,7,8,10a,10b-decahydrocyclohepta[e]indene-3a(1*H*)-carboxylic acid

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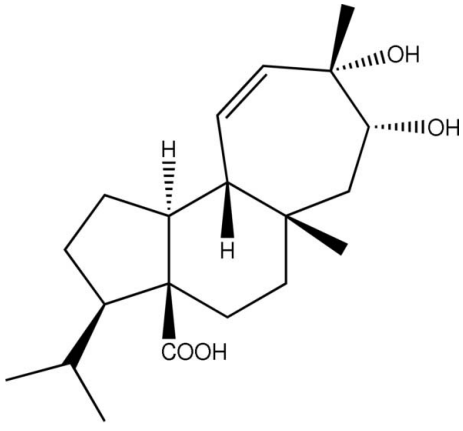
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.040; $wR$ factor = 0.117; data-to-parameter ratio = 8.5.

The molecule of the title compound, $C_{20}H_{32}O_4$, is built up from three fused five-membered, six-membered and seven-membered rings. The five-membered ring has an envelope conformation, whereas the six- and seven-membered rings have chair conformations. The crystal structure is stabilized by strong intermolecular O—H···O hydrogen bonds, forming a three-dimensional network. The absolute configuration was assigned on the basis of earlier chemical studies.

## Related literature

For related literature, see: Araya *et al.* (2003); Cremer & Pople (1975); Fuentes *et al.* (2005); Loyola *et al.* (1996, 2004); Wickens (1995).




## Experimental

### Crystal data

$C_{20}H_{32}O_4$
$M_r$ = 336.46
Orthorhombic, $P2_12_12_1$
$a$ = 11.094 (7) Å
$b$ = 12.728 (10) Å
$c$ = 13.8776 (11) Å
$V$ = 1959.6 (19) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 298 (2) K
0.30 × 0.20 × 0.10 mm

### Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
9149 measured reflections
1922 independent reflections
1836 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.072

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.117
$S$ = 1.13
1922 reflections
226 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O4$^{i}$ | 0.82 | 1.80 | 2.613 (3) | 170 |
| O3—H3···O2$^{ii}$ | 0.82 | 2.01 | 2.825 (3) | 173 |
| O4—H4···O3$^{iii}$ | 0.82 | 1.94 | 2.752 (3) | 172 |
| C1—H1*B*···O2 | 0.97 | 2.44 | 2.889 (4) | 108 |
| C5—H5*B*···O1 | 0.97 | 2.51 | 3.054 (4) | 116 |
| C6—H6*B*···O4 | 0.97 | 2.52 | 2.901 (3) | 104 |
| C16—H16*A*···O3 | 0.96 | 2.46 | 2.809 (4) | 101 |

Symmetry codes: (i) $-x+\frac{1}{2}, -y+2, z-\frac{1}{2}$; (ii) $-x+\frac{1}{2}, -y+2, z+\frac{1}{2}$; (iii) $x+\frac{1}{2}, -y+\frac{5}{2}, -z+2$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2730).

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# supporting information

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# (3*S*,3a*S*,5a*S*,7*S*,8*S*,10a*S*,10b*R*)-7,8-Dihydroxy-3-isopropyl-5a,8-dimethyl-2,3,4,5,5a,6,7,8,10a,10b-decahydrocyclohepta[*e*]indene-3a(1*H*)-carboxylic acid

**Iván Brito, Jorge Bórquez, Luis Alberto Loyola, Alejandro Cárdenas and Matías López-Rodríguez**

## S1. Comment

Azorella compacta is a compact resinous cushion shrub that grows in the Andes of Peru, Bolivia, Argentina and Chile and has been used in folk medicine. The common name llareta is used for several species of the genus Azorella (Wickens, 1995). Mulinane diterpenes exhibits antiplasmodial (Loyola *et al.*, 2004), anti-Tripanosoma cruzi (Araya *et al.*, 2003) and antihyperglycemic (Fuentes *et al.*, 2005) activities.We have undertaken the X-ray crystal-structure determination of the title compound in order to establish its molecular conformation and relative stereochemistry. We are not able to determine the absolute stereochemistry by X-ray methods and the configuration shown here was chosen to be in accord with that reported in previous chemical studies (Loyola *et al.*, 1996). The structure consists of a mulinic acid skeleton and the isopropyl, methyl groups and carboxylic acid at C3, C5a, C8 and C3b are $\alpha$-oriented respectively, whereas the hydroxyl groups at C8 and C7 are $\beta$-oriented. The cyclopentane (A), cyclohexane (B) and cycloheptene (C) rings are in an envelope, chair and chair conformation respectively [$Q_2$ = 0.435 (2) Å, $\varphi_2$= 118.7 (3)° for rig A; $Q_T$= 0.581 (2) Å, $\theta$ = 174.4 (2)°, $\varphi$=131 (2)° for ring B; $Q_T$= 0.634 (2) Å, $\varphi_2$=78.4 (6)°, for ring C] (Cremer & Pople, 1975). The A and B and B and C rings are *trans* and *cis*-fused respectively. The molecular conformation is stabilized by four intramolecular hydrogen bonds and the crystal structure is stabilized by three intermolecular hydrogen bonds (Table 1).

## S2. Experimental

Dried and finely powdered whole plant of Azorella compacta (3,0 kg) were extracted with petroleum ether at room temperature. After filtration, the solvent was evaporated in vacuum yielding a gum (220 g). The concentrated petrol ether extract was adsorbed on silica gel (300 g) and slurried onto the top of a column containing silica gel (2.0 kg) in petroleum ether, and eluted with a petroleum ether/ethyl acetate gradient with increasing amounts of ethyl acetate to produce six fractions. Fraction 2 (100 g) eluted with petroleum ether/ethyl acetate(18:2) was further separated and purified by silica gel column chromatography(petroleum ether/ethyl acetate), 19:1) to give 600 mg of the title compound. The structure were elucidated by analysis of their spectroscopic data. Recrystallization from hexane-ethyl acetate (7:3) at room temperature afforded colourless crystals suitable for X-ray diffraction analysis.

## S3. Refinement

All H atoms were located on a difference Fourier map and then treated as riding atoms, with C - H bond lengths in the range 0.96 - 0.98 Å and O - H distances of 0.82 Å. For methyl atoms, $U_{iso}$(H) = 1.5$U_{eq}$(C), while for other H atoms, $U_{iso}$(H) = 1.2$U_{eq}$(C, O). In the absence of significant anomalous scattering effects, Friedel pairs were averaged. The absolute configuration shown here was chosen to be in accord with that reported in previous chemical studies (Loyola *et al.*, 1996).

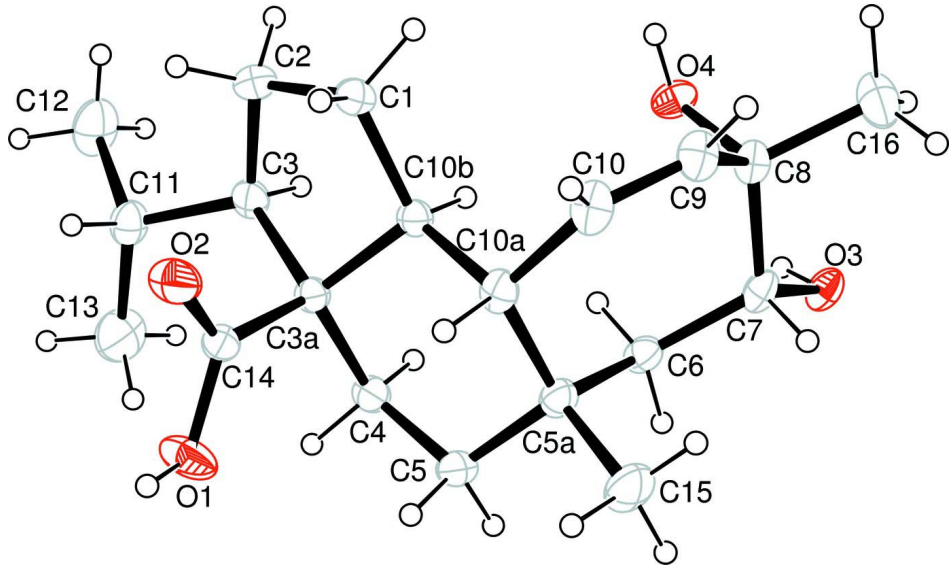


**Figure 1**

The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

**(3*S*,3a*S*,5a*S*,7*S*,8*S*,10a*S*,10b*R*)-7,8-Dihydroxy-3-isopropyl-5a,8-dimethyl-2,3,4,5,5a,6,7,8,10a,10b-decahydrocyclohepta[*e*]indene-3a(1*H*)-carboxylic acid**

*Crystal data*

$C_{20}H_{32}O_4$
$M_r = 336.46$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 11.094\ (7)$ Å
$b = 12.728\ (10)$ Å
$c = 13.8776\ (11)$ Å
$V = 1959.6\ (19)$ Å$^3$
$Z = 4$
$F(000) = 736$
$D_x = 1.14$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5686 reflections
$\theta = 2.3–25.2°$
$\mu = 0.08$ mm$^{-1}$
$T = 298$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

*Data collection*

Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ scans, and $\omega$ scans with $\kappa$ offsets
9149 measured reflections
1922 independent reflections
1836 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 25.2°$, $\theta_{min} = 2.4°$
$h = -9\rightarrow13$
$k = -11\rightarrow15$
$l = -15\rightarrow16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.117$
$S = 1.13$
1922 reflections
226 parameters
0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0775P)^2 + 0.1473P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.005$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.14$ 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.

**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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.22790 (18) | 0.80481 (16) | 0.67764 (12) | 0.0550 (5) |
| H1 | 0.2371 | 0.7969 | 0.6195 | 0.066 (2)* |
| O2 | 0.39658 (15) | 0.89409 (15) | 0.66282 (11) | 0.0497 (5) |
| O3 | 0.01429 (13) | 1.24195 (13) | 1.01859 (10) | 0.0361 (4) |
| H3 | 0.0408 | 1.1986 | 1.0568 | 0.066 (2)* |
| O4 | 0.27135 (13) | 1.21546 (12) | 0.99028 (10) | 0.0352 (4) |
| H4 | 0.3422 | 1.2321 | 0.9835 | 0.066 (2)* |
| C1 | 0.44994 (19) | 1.03680 (17) | 0.81893 (16) | 0.0368 (5) |
| H1A | 0.4778 | 1.0971 | 0.8554 | 0.046 (2)* |
| H1B | 0.4745 | 1.0446 | 0.7522 | 0.046 (2)* |
| C2 | 0.4986 (2) | 0.93369 (19) | 0.86208 (19) | 0.0431 (6) |
| H2A | 0.5396 | 0.9477 | 0.9224 | 0.046 (2)* |
| H2B | 0.5554 | 0.9014 | 0.818 | 0.046 (2)* |
| C3 | 0.3898 (2) | 0.85939 (16) | 0.87930 (14) | 0.0338 (5) |
| H3A | 0.3642 | 0.8699 | 0.9462 | 0.041* |
| C3A | 0.28837 (18) | 0.90550 (15) | 0.81408 (13) | 0.0278 (4) |
| C4 | 0.15886 (19) | 0.88598 (16) | 0.84672 (15) | 0.0330 (4) |
| H4A | 0.1524 | 0.8994 | 0.9153 | 0.046 (2)* |
| H4B | 0.1381 | 0.813 | 0.8355 | 0.046 (2)* |
| C5 | 0.0704 (2) | 0.95643 (19) | 0.79272 (17) | 0.0407 (5) |
| H5A | −0.0097 | 0.9439 | 0.8182 | 0.046 (2)* |
| H5B | 0.0698 | 0.9354 | 0.7255 | 0.046 (2)* |
| C5A | 0.09599 (19) | 1.07500 (17) | 0.79757 (15) | 0.0343 (5) |
| C6 | 0.06692 (18) | 1.11212 (16) | 0.90075 (14) | 0.0329 (4) |
| H6A | −0.0136 | 1.0875 | 0.9159 | 0.046 (2)* |
| H6B | 0.1217 | 1.0758 | 0.9438 | 0.046 (2)* |
| C7 | 0.07182 (19) | 1.22815 (17) | 0.92647 (14) | 0.0322 (4) |
| H7 | 0.0232 | 1.2658 | 0.8787 | 0.039* |
| C8 | 0.1969 (2) | 1.27901 (16) | 0.92731 (15) | 0.0332 (5) |
| C9 | 0.2535 (2) | 1.28268 (17) | 0.82860 (15) | 0.0377 (5) |
| H9 | 0.2827 | 1.3481 | 0.8099 | 0.045* |
| C10 | 0.2678 (2) | 1.20670 (18) | 0.76493 (15) | 0.0390 (5) |
| H10 | 0.3086 | 1.2265 | 0.7093 | 0.047* |
| C10A | 0.22889 (19) | 1.09300 (16) | 0.76724 (13) | 0.0319 (4) |
| H10A | 0.2354 | 1.0675 | 0.7008 | 0.038* |

| | | | | |
|---|---|---|---|---|
| C10B | 0.31312 (18) | 1.02422 (15) | 0.82710 (13) | 0.0286 (4) |
| H10B | 0.2936 | 1.0397 | 0.8945 | 0.034* |
| C11 | 0.4236 (2) | 0.74293 (17) | 0.86931 (15) | 0.0398 (5) |
| H11 | 0.4606 | 0.7335 | 0.8058 | 0.048* |
| C12 | 0.5173 (3) | 0.7132 (3) | 0.9447 (2) | 0.0599 (8) |
| H12A | 0.5844 | 0.7607 | 0.9408 | 0.066 (2)* |
| H12B | 0.5448 | 0.6427 | 0.9331 | 0.066 (2)* |
| H12C | 0.482 | 0.7173 | 1.0077 | 0.066 (2)* |
| C13 | 0.3176 (3) | 0.6675 (2) | 0.8759 (3) | 0.0675 (9) |
| H13A | 0.2766 | 0.6777 | 0.9362 | 0.066 (2)* |
| H13B | 0.3463 | 0.5965 | 0.8721 | 0.066 (2)* |
| H13C | 0.2628 | 0.6808 | 0.8238 | 0.066 (2)* |
| C14 | 0.31036 (18) | 0.86941 (16) | 0.71064 (13) | 0.0301 (4) |
| C15 | 0.0128 (3) | 1.1305 (2) | 0.72577 (19) | 0.0545 (7) |
| H15A | 0.0256 | 1.1022 | 0.6625 | 0.066 (2)* |
| H15B | 0.0303 | 1.2044 | 0.7254 | 0.066 (2)* |
| H15C | −0.0696 | 1.1198 | 0.7444 | 0.066 (2)* |
| C16 | 0.1909 (3) | 1.39039 (18) | 0.9685 (2) | 0.0525 (6) |
| H16A | 0.1586 | 1.388 | 1.0326 | 0.066 (2)* |
| H16B | 0.1399 | 1.433 | 0.9285 | 0.066 (2)* |
| H16C | 0.2704 | 1.42 | 0.9702 | 0.066 (2)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0571 (10) | 0.0727 (11) | 0.0351 (9) | −0.0269 (10) | 0.0080 (8) | −0.0216 (8) |
| O2 | 0.0426 (9) | 0.0711 (12) | 0.0353 (8) | −0.0131 (8) | 0.0105 (7) | −0.0178 (8) |
| O3 | 0.0291 (7) | 0.0475 (9) | 0.0318 (8) | 0.0110 (6) | 0.0051 (6) | −0.0001 (6) |
| O4 | 0.0272 (7) | 0.0449 (8) | 0.0335 (7) | −0.0028 (7) | 0.0012 (6) | 0.0082 (6) |
| C1 | 0.0314 (11) | 0.0364 (10) | 0.0427 (11) | −0.0054 (9) | 0.0010 (9) | −0.0081 (9) |
| C2 | 0.0300 (10) | 0.0459 (12) | 0.0534 (14) | 0.0010 (10) | −0.0109 (10) | −0.0080 (10) |
| C3 | 0.0382 (11) | 0.0376 (10) | 0.0255 (9) | 0.0046 (9) | −0.0058 (8) | −0.0040 (8) |
| C3A | 0.0273 (9) | 0.0316 (9) | 0.0245 (9) | −0.0005 (8) | 0.0013 (8) | −0.0027 (7) |
| C4 | 0.0304 (9) | 0.0366 (10) | 0.0320 (9) | −0.0052 (8) | 0.0054 (8) | −0.0059 (8) |
| C5 | 0.0281 (10) | 0.0482 (12) | 0.0457 (12) | −0.0008 (9) | −0.0036 (9) | −0.0128 (10) |
| C5A | 0.0308 (10) | 0.0420 (11) | 0.0302 (10) | 0.0071 (9) | −0.0045 (8) | −0.0040 (8) |
| C6 | 0.0252 (9) | 0.0400 (10) | 0.0337 (10) | 0.0016 (8) | 0.0015 (8) | −0.0007 (8) |
| C7 | 0.0290 (9) | 0.0411 (10) | 0.0264 (9) | 0.0112 (9) | 0.0033 (8) | 0.0017 (8) |
| C8 | 0.0369 (10) | 0.0317 (9) | 0.0310 (10) | 0.0064 (9) | 0.0034 (8) | 0.0015 (8) |
| C9 | 0.0434 (12) | 0.0321 (9) | 0.0377 (11) | 0.0021 (9) | 0.0084 (9) | 0.0100 (8) |
| C10 | 0.0462 (12) | 0.0413 (11) | 0.0294 (10) | 0.0074 (10) | 0.0113 (9) | 0.0084 (8) |
| C10A | 0.0372 (10) | 0.0383 (10) | 0.0201 (8) | 0.0061 (9) | 0.0026 (8) | −0.0012 (7) |
| C10B | 0.0285 (10) | 0.0310 (9) | 0.0263 (9) | −0.0004 (8) | 0.0019 (7) | −0.0029 (7) |
| C11 | 0.0444 (12) | 0.0388 (11) | 0.0362 (11) | 0.0101 (10) | −0.0013 (10) | 0.0028 (8) |
| C12 | 0.0706 (18) | 0.0638 (16) | 0.0454 (13) | 0.0282 (15) | −0.0104 (13) | 0.0082 (11) |
| C13 | 0.0611 (18) | 0.0407 (13) | 0.101 (2) | 0.0003 (13) | 0.0004 (17) | 0.0144 (14) |
| C14 | 0.0301 (9) | 0.0331 (9) | 0.0272 (9) | 0.0010 (8) | −0.0024 (8) | −0.0027 (7) |
| C15 | 0.0484 (13) | 0.0693 (16) | 0.0458 (13) | 0.0188 (13) | −0.0178 (12) | −0.0044 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.0619 (15) | 0.0367 (12) | 0.0588 (15) | −0.0006 (11) | 0.0168 (13) | −0.0067 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C14 | 1.312 (3) | C6—C7 | 1.520 (3) |
| O1—H1 | 0.82 | C6—H6A | 0.97 |
| O2—C14 | 1.206 (3) | C6—H6B | 0.97 |
| O3—C7 | 1.440 (2) | C7—C8 | 1.531 (3) |
| O3—H3 | 0.82 | C7—H7 | 0.98 |
| O4—C8 | 1.449 (3) | C8—C9 | 1.508 (3) |
| O4—H4 | 0.82 | C8—C16 | 1.530 (3) |
| C1—C10B | 1.531 (3) | C9—C10 | 1.319 (3) |
| C1—C2 | 1.540 (3) | C9—H9 | 0.93 |
| C1—H1A | 0.97 | C10—C10A | 1.510 (3) |
| C1—H1B | 0.97 | C10—H10 | 0.93 |
| C2—C3 | 1.551 (3) | C10A—C10B | 1.526 (3) |
| C2—H2A | 0.97 | C10A—H10A | 0.98 |
| C2—H2B | 0.97 | C10B—H10B | 0.98 |
| C3—C11 | 1.535 (3) | C11—C12 | 1.523 (3) |
| C3—C3A | 1.559 (3) | C11—C13 | 1.521 (4) |
| C3—H3A | 0.98 | C11—H11 | 0.98 |
| C3A—C4 | 1.527 (3) | C12—H12A | 0.96 |
| C3A—C14 | 1.527 (3) | C12—H12B | 0.96 |
| C3A—C10B | 1.546 (3) | C12—H12C | 0.96 |
| C4—C5 | 1.526 (3) | C13—H13A | 0.96 |
| C4—H4A | 0.97 | C13—H13B | 0.96 |
| C4—H4B | 0.97 | C13—H13C | 0.96 |
| C5—C5A | 1.537 (3) | C15—H15A | 0.96 |
| C5—H5A | 0.97 | C15—H15B | 0.96 |
| C5—H5B | 0.97 | C15—H15C | 0.96 |
| C5A—C15 | 1.531 (3) | C16—H16A | 0.96 |
| C5A—C6 | 1.542 (3) | C16—H16B | 0.96 |
| C5A—C10A | 1.550 (3) | C16—H16C | 0.96 |
| | | | |
| C14—O1—H1 | 109.5 | O4—C8—C9 | 109.10 (16) |
| C7—O3—H3 | 109.5 | O4—C8—C16 | 108.5 (2) |
| C8—O4—H4 | 109.5 | C9—C8—C16 | 109.20 (18) |
| C10B—C1—C2 | 103.26 (18) | O4—C8—C7 | 106.57 (16) |
| C10B—C1—H1A | 111.1 | C9—C8—C7 | 112.58 (18) |
| C2—C1—H1A | 111.1 | C16—C8—C7 | 110.82 (19) |
| C10B—C1—H1B | 111.1 | C10—C9—C8 | 129.5 (2) |
| C2—C1—H1B | 111.1 | C10—C9—H9 | 115.3 |
| H1A—C1—H1B | 109.1 | C8—C9—H9 | 115.3 |
| C1—C2—C3 | 107.89 (18) | C9—C10—C10A | 130.8 (2) |
| C1—C2—H2A | 110.1 | C9—C10—H10 | 114.6 |
| C3—C2—H2A | 110.1 | C10A—C10—H10 | 114.6 |
| C1—C2—H2B | 110.1 | C10—C10A—C10B | 112.72 (18) |
| C3—C2—H2B | 110.1 | C10—C10A—C5A | 114.76 (18) |

| | | | |
|---|---|---|---|
| H2A—C2—H2B | 108.4 | C10B—C10A—C5A | 110.47 (16) |
| C11—C3—C2 | 112.66 (19) | C10—C10A—H10A | 106.1 |
| C11—C3—C3A | 119.15 (17) | C10B—C10A—H10A | 106.1 |
| C2—C3—C3A | 104.03 (17) | C5A—C10A—H10A | 106.1 |
| C11—C3—H3A | 106.8 | C10A—C10B—C1 | 120.45 (18) |
| C2—C3—H3A | 106.8 | C10A—C10B—C3A | 112.84 (16) |
| C3A—C3—H3A | 106.8 | C1—C10B—C3A | 105.65 (16) |
| C4—C3A—C14 | 112.35 (16) | C10A—C10B—H10B | 105.6 |
| C4—C3A—C10B | 106.94 (16) | C1—C10B—H10B | 105.6 |
| C14—C3A—C10B | 112.05 (16) | C3A—C10B—H10B | 105.6 |
| C4—C3A—C3 | 116.49 (17) | C12—C11—C13 | 109.3 (2) |
| C14—C3A—C3 | 108.49 (16) | C12—C11—C3 | 110.2 (2) |
| C10B—C3A—C3 | 99.89 (15) | C13—C11—C3 | 114.5 (2) |
| C3A—C4—C5 | 111.34 (17) | C12—C11—H11 | 107.5 |
| C3A—C4—H4A | 109.4 | C13—C11—H11 | 107.5 |
| C5—C4—H4A | 109.4 | C3—C11—H11 | 107.5 |
| C3A—C4—H4B | 109.4 | C11—C12—H12A | 109.5 |
| C5—C4—H4B | 109.4 | C11—C12—H12B | 109.5 |
| H4A—C4—H4B | 108 | H12A—C12—H12B | 109.5 |
| C4—C5—C5A | 115.89 (18) | C11—C12—H12C | 109.5 |
| C4—C5—H5A | 108.3 | H12A—C12—H12C | 109.5 |
| C5A—C5—H5A | 108.3 | H12B—C12—H12C | 109.5 |
| C4—C5—H5B | 108.3 | C11—C13—H13A | 109.5 |
| C5A—C5—H5B | 108.3 | C11—C13—H13B | 109.5 |
| H5A—C5—H5B | 107.4 | H13A—C13—H13B | 109.5 |
| C15—C5A—C5 | 108.27 (19) | C11—C13—H13C | 109.5 |
| C15—C5A—C6 | 109.69 (18) | H13A—C13—H13C | 109.5 |
| C5—C5A—C6 | 107.63 (18) | H13B—C13—H13C | 109.5 |
| C15—C5A—C10A | 109.16 (19) | O2—C14—O1 | 121.61 (18) |
| C5—C5A—C10A | 107.99 (17) | O2—C14—C3A | 124.46 (18) |
| C6—C5A—C10A | 113.94 (17) | O1—C14—C3A | 113.90 (17) |
| C7—C6—C5A | 120.54 (18) | C5A—C15—H15A | 109.5 |
| C7—C6—H6A | 107.2 | C5A—C15—H15B | 109.5 |
| C5A—C6—H6A | 107.2 | H15A—C15—H15B | 109.5 |
| C7—C6—H6B | 107.2 | C5A—C15—H15C | 109.5 |
| C5A—C6—H6B | 107.2 | H15A—C15—H15C | 109.5 |
| H6A—C6—H6B | 106.8 | H15B—C15—H15C | 109.5 |
| O3—C7—C6 | 108.13 (17) | C8—C16—H16A | 109.5 |
| O3—C7—C8 | 110.08 (17) | C8—C16—H16B | 109.5 |
| C6—C7—C8 | 116.42 (17) | H16A—C16—H16B | 109.5 |
| O3—C7—H7 | 107.3 | C8—C16—H16C | 109.5 |
| C6—C7—H7 | 107.3 | H16A—C16—H16C | 109.5 |
| C8—C7—H7 | 107.3 | H16B—C16—H16C | 109.5 |

| | | | |
|---|---|---|---|
| C10B—C1—C2—C3 | 8.2 (2) | C9—C10—C10A—C10B | −79.8 (3) |
| C1—C2—C3—C11 | 149.26 (18) | C9—C10—C10A—C5A | 47.8 (3) |
| C1—C2—C3—C3A | 18.8 (2) | C15—C5A—C10A—C10 | 60.6 (2) |
| C11—C3—C3A—C4 | 81.3 (2) | C5—C5A—C10A—C10 | 178.06 (18) |

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|---|---|---|---|
| C2—C3—C3A—C4 | −152.25 (18) | C6—C5A—C10A—C10 | −62.4 (2) |
| C11—C3—C3A—C14 | −46.6 (2) | C15—C5A—C10A—C10B | −170.67 (17) |
| C2—C3—C3A—C14 | 79.8 (2) | C5—C5A—C10A—C10B | −53.2 (2) |
| C11—C3—C3A—C10B | −164.02 (18) | C6—C5A—C10A—C10B | 66.3 (2) |
| C2—C3—C3A—C10B | −37.57 (19) | C10—C10A—C10B—C1 | −43.1 (2) |
| C14—C3A—C4—C5 | −68.2 (2) | C5A—C10A—C10B—C1 | −172.95 (17) |
| C10B—C3A—C4—C5 | 55.2 (2) | C10—C10A—C10B—C3A | −169.03 (16) |
| C3—C3A—C4—C5 | 165.83 (16) | C5A—C10A—C10B—C3A | 61.1 (2) |
| C3A—C4—C5—C5A | −55.1 (2) | C2—C1—C10B—C10A | −162.12 (17) |
| C4—C5—C5A—C15 | 170.12 (19) | C2—C1—C10B—C3A | −32.9 (2) |
| C4—C5—C5A—C6 | −71.4 (2) | C4—C3A—C10B—C10A | −60.5 (2) |
| C4—C5—C5A—C10A | 52.0 (2) | C14—C3A—C10B—C10A | 63.1 (2) |
| C15—C5A—C6—C7 | −56.7 (3) | C3—C3A—C10B—C10A | 177.76 (15) |
| C5—C5A—C6—C7 | −174.27 (18) | C4—C3A—C10B—C1 | 165.97 (16) |
| C10A—C5A—C6—C7 | 66.0 (2) | C14—C3A—C10B—C1 | −70.5 (2) |
| C5A—C6—C7—O3 | 166.72 (16) | C3—C3A—C10B—C1 | 44.2 (2) |
| C5A—C6—C7—C8 | −68.8 (2) | C2—C3—C11—C12 | 62.0 (3) |
| O3—C7—C8—O4 | 69.69 (19) | C3A—C3—C11—C12 | −175.7 (2) |
| C6—C7—C8—O4 | −53.8 (2) | C2—C3—C11—C13 | −174.3 (2) |
| O3—C7—C8—C9 | −170.74 (16) | C3A—C3—C11—C13 | −52.1 (3) |
| C6—C7—C8—C9 | 65.8 (2) | C4—C3A—C14—O2 | 165.1 (2) |
| O3—C7—C8—C16 | −48.1 (2) | C10B—C3A—C14—O2 | 44.7 (3) |
| C6—C7—C8—C16 | −171.59 (18) | C3—C3A—C14—O2 | −64.7 (3) |
| O4—C8—C9—C10 | 67.3 (3) | C4—C3A—C14—O1 | −17.0 (2) |
| C16—C8—C9—C10 | −174.3 (3) | C10B—C3A—C14—O1 | −137.48 (19) |
| C7—C8—C9—C10 | −50.8 (3) | C3—C3A—C14—O1 | 113.2 (2) |
| C8—C9—C10—C10A | 2.5 (4) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O4$^{i}$ | 0.82 | 1.80 | 2.613 (3) | 170 |
| O3—H3···O2$^{ii}$ | 0.82 | 2.01 | 2.825 (3) | 173 |
| O4—H4···O3$^{iii}$ | 0.82 | 1.94 | 2.752 (3) | 172 |
| C1—H1*B*···O2 | 0.97 | 2.44 | 2.889 (4) | 108 |
| C5—H5*B*···O1 | 0.97 | 2.51 | 3.054 (4) | 116 |
| C6—H6*B*···O4 | 0.97 | 2.52 | 2.901 (3) | 104 |
| C16—H16*A*···O3 | 0.96 | 2.46 | 2.809 (4) | 101 |

Symmetry codes: (i) −*x*+1/2, −*y*+2, *z*−1/2; (ii) −*x*+1/2, −*y*+2, *z*+1/2; (iii) *x*+1/2, −*y*+5/2, −*z*+2.