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# Aromaticine, a sesquiterpene lactone from *Amblyopappus pusillus*

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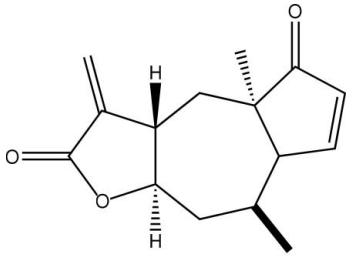
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.058; $wR$ factor = 0.149; data-to-parameter ratio = 10.0.

Aromaticine (systematic name: 4a,8-dimethyl-3-methylene-3,3a,4,4a,7a,8,9,9a-octahydroazuleno[6,5-*b*]furan-2,5-dione), $C_{15}H_{18}O_3$, is a natural lactone isolated from *Amblyopappus pusillus*. The molecular structure and conformation agree with the results of Romo, Joseph-Nathan & Díaz [(1964). *Tetrahedron*, **20**, 79–85]. The fused-ring system contains a seven-membered ring in a twist-boat conformation and two five-membered rings *trans* fused in envelope conformations.

## Related literature

For related literature, see: Allen *et al.* (1987); Bórquez (2006); Bernstein *et al.* (1995); Cremer & Pople (1975); Rodríguez *et al.* (1976); Romo *et al.* (1964).



## Experimental

### Crystal data

$C_{15}H_{18}O_3$
$M_r$ = 246.29
Orthorhombic, $P2_12_12_1$
$a$ = 6.763 (4) Å
$b$ = 9.932 (5) Å
$c$ = 18.685 (7) Å
$V$ = 1255.1 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 295 (2) K
0.40 × 0.10 × 0.08 mm

### Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
16315 measured reflections
1667 independent reflections
1432 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.051

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.148
$S$ = 1.20
1667 reflections
167 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$

Data collection: *COLLECT* (Nonius, 1998); 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: OM2203).

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# supporting information

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## Aromaticine, a sesquiterpene lactone from *Amblyopappus pusillus*

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

### S1. Comment

Sesquiterpene lactones are known to have biological activity, and some guaianolides have antitumor and citotoxic activities (Rodríguez *et al.*, 1976). The title compound was isolated from aerial parts of Amblyopappus pusillus (Bórquez, 2006), which was originally isolated from Helenium Aromaticum (Romo *et al.*, 1964).In order to ascertain the structure and secure the assignment of the stereochemistry, an X-ray structure determination was performed. The absolute configuration was not determined. The structure confirms the previously proposed molecular structure and molecular conformation (Romo *et al.*, 1964). The molecule (Fig. 1) involves a seven-membered ring in a twisted- boat conformation. Deviations of atoms in this ring from the plane (Cremer & Pople, 1975) were less than 0.1 Å for atoms C4 and C9 and more than 0.38 Å, for the remaining atoms of the ring. Using the same definition, for the two five-membered rings, the lactone is present in an envelope conformation with C3a in the flap position and a maximun deviation of -0.144 (3) Å for C3a. The other five-membered ring also adopts an envelope conformation, with C4a 0.137 (3) Å out of the plane. The crystal structure consists of discrete molecules. The molecules are linked into chains by one weak intermolecular C—H···O hydrogen bond [H···O =2.65 Å, C···O = 3.552 (4) Å and C—H···O =163°]. Atom C7 acts as a hydrogen bond donor *via* atom H7 to atom O2 in the molecule at (-*x* + 1/2,-*y* + 1, *z* + 1/2), so generating a C(9) chain running parallel to the [001] direction, (Bernstein, *et al.*, 1995). Bond lengths are within expected ranges (Allen *et al.*, 1987), with average values (Å): O-C$sp^2$ =1.205 (4); C$sp^2$—C$sp^2$ = 1.440 (5); C$sp^3$—C$sp^2$ =1.501 (5) and C$sp^3$—C$sp^3$= 1.516 (4).

### S2. Experimental

The whole fresh plants (200 g) was submerged in chloroform at room temperature for 4 hrs. After filtration, the solvent was avaporated to dryness under reduced pressure, yielding 10 g. The chloroform extract was chromatographed on silica gel column using diethyl ether, giving 800 mg of the title compound (m.p 505–527 K). Optical rotation: $[\alpha]_D^{20}$ +20.6. The title compound was identified by comparing the spectroscopic data with the previously published data (Romo *et al.*, 1964). Crystal suitable for X-ray analysis were obtained for recrystallization from diethyl ether at room temperature.

### S3. Refinement

H atoms were located from difference Fourier maps and placed in geometrically idealized positions (C—H = 0.93–0.98 Å), and were constrained to ride on their parents atoms with $U_{iso}$ (H) = 1.5$U_{eq}$(C) for methyl H atoms and 1.2 $U_{eq}$(C) for the other H atoms.

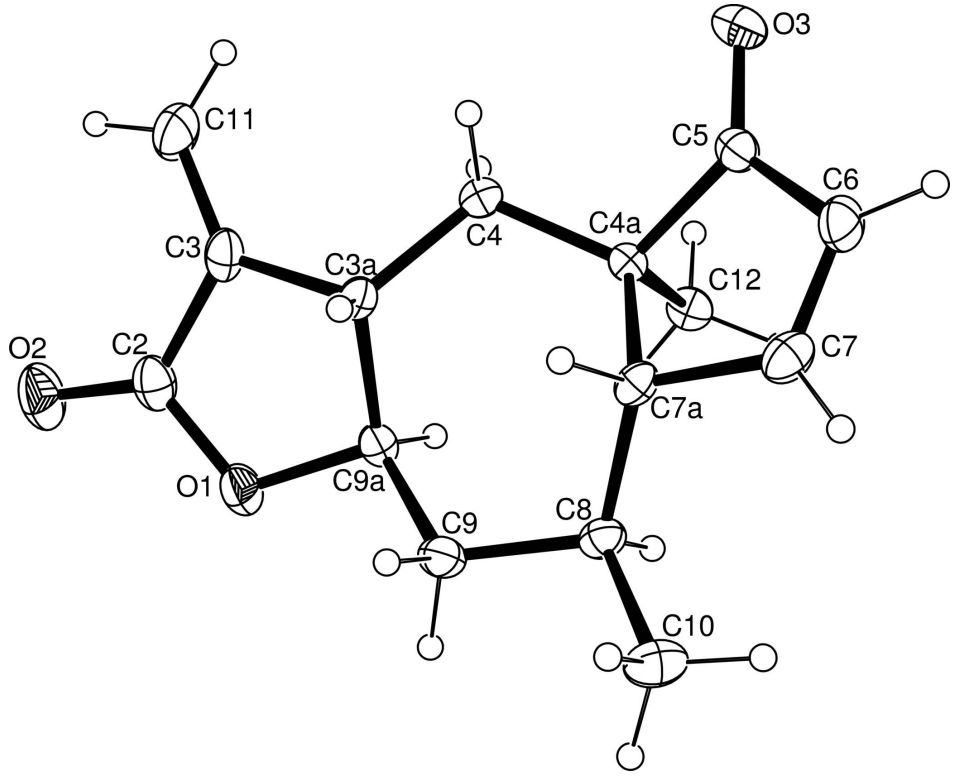


**Figure 1**

View of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level, and H-atom radii are arbitrary.

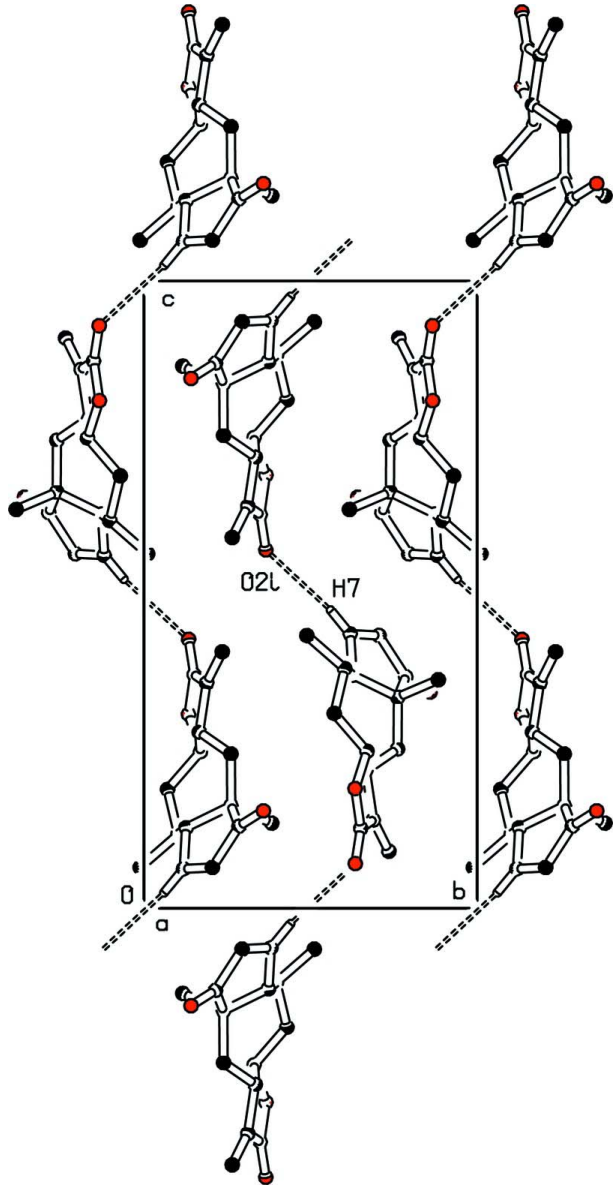


**Figure 2**

Part of the crystal structure showing the formation of C(9) along [001]. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted. [Symmetry code: (i) 1/2 - *x*, 1 - *y*, 1/2 + *z*.]

### 4a,8-dimethyl-3-methylene-3,3a,4,4a,7a,8,9,9a-octahydroazuleno[6,5-*b*]furan-\ 2,5-dione

*Crystal data*

$C_{15}H_{18}O_3$
$M_r = 246.29$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.763$ (4) Å
$b = 9.932$ (5) Å
$c = 18.685$ (7) Å
$V = 1255.1$ (11) Å$^3$
$Z = 4$
$F(000) = 528$
$D_x = 1.303$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 1595 reflections
$\theta = 2.3$–27.5°
$\mu = 0.09$ mm$^{-1}$
$T = 295$ K
Needle, colourless
0.40 × 0.10 × 0.08 mm

*Data collection*

Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans with $\kappa$ offsets
16315 measured reflections
1667 independent reflections

1432 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -8 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -22 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.148$
$S = 1.20$
1667 reflections
167 parameters

0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.1397P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å$^{-3}$
$\Delta\rho_{min} = -0.23$ 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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.5372 (3) | 0.6338 (2) | 0.19079 (12) | 0.0407 (6) |
| O2 | 0.5286 (4) | 0.6343 (3) | 0.07133 (13) | 0.0575 (8) |
| O3 | −0.2760 (3) | 0.8586 (3) | 0.34464 (13) | 0.0411 (6) |
| C2 | 0.4451 (5) | 0.6506 (3) | 0.12748 (18) | 0.0388 (8) |
| C3 | 0.2351 (5) | 0.6883 (3) | 0.14092 (17) | 0.0352 (8) |
| C3A | 0.1973 (4) | 0.6661 (3) | 0.21918 (14) | 0.0266 (7) |
| H3A | 0.147 | 0.5742 | 0.2248 | 0.032 (3)* |
| C4 | 0.0484 (4) | 0.7610 (3) | 0.25401 (15) | 0.0327 (7) |
| H4A | −0.0835 | 0.7355 | 0.2388 | 0.032 (3)* |
| H4B | 0.0728 | 0.8517 | 0.2369 | 0.032 (3)* |
| C4A | 0.0554 (4) | 0.7616 (3) | 0.33662 (15) | 0.0246 (6) |
| C5 | −0.1505 (4) | 0.7855 (3) | 0.36921 (16) | 0.0290 (7) |
| C6 | −0.1602 (5) | 0.7054 (4) | 0.43487 (17) | 0.0398 (8) |
| H6 | −0.257 | 0.7141 | 0.4699 | 0.032 (3)* |
| C7 | −0.0114 (4) | 0.6190 (3) | 0.43729 (17) | 0.0362 (8) |
| H7 | 0.0105 | 0.5602 | 0.4752 | 0.032 (3)* |
| C7A | 0.1190 (4) | 0.6263 (3) | 0.37165 (15) | 0.0270 (7) |
| H7A | 0.0729 | 0.555 | 0.3394 | 0.032 (3)* |
| C8 | 0.3431 (4) | 0.6025 (3) | 0.38199 (16) | 0.0294 (7) |
| H8 | 0.401 | 0.6854 | 0.4012 | 0.032 (3)* |

| | | | | |
|---|---|---|---|---|
| C9 | 0.4455 (5) | 0.5710 (3) | 0.31060 (17) | 0.0335 (7) |
| H9A | 0.5868 | 0.5666 | 0.319 | 0.032 (3)* |
| H9B | 0.4032 | 0.4824 | 0.295 | 0.032 (3)* |
| C9A | 0.4083 (4) | 0.6694 (3) | 0.25025 (15) | 0.0301 (7) |
| H9C | 0.4391 | 0.7608 | 0.2665 | 0.032 (3)* |
| C10 | 0.3856 (5) | 0.4889 (4) | 0.43484 (19) | 0.0461 (9) |
| H10A | 0.3261 | 0.4071 | 0.4179 | 0.045 (4)* |
| H10B | 0.3315 | 0.5116 | 0.4808 | 0.045 (4)* |
| H10C | 0.5259 | 0.4766 | 0.4391 | 0.045 (4)* |
| C11 | 0.1178 (6) | 0.7350 (4) | 0.09104 (18) | 0.0545 (10) |
| H11A | 0.1645 | 0.7459 | 0.0446 | 0.065* |
| H11B | −0.0123 | 0.7572 | 0.102 | 0.065* |
| C12 | 0.1774 (5) | 0.8833 (3) | 0.36430 (17) | 0.0316 (7) |
| H12A | 0.1202 | 0.9654 | 0.3468 | 0.045 (4)* |
| H12B | 0.3112 | 0.876 | 0.3475 | 0.045 (4)* |
| H12C | 0.1764 | 0.8838 | 0.4157 | 0.045 (4)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0379 (12) | 0.0473 (14) | 0.0370 (13) | 0.0043 (12) | 0.0092 (10) | −0.0058 (11) |
| O2 | 0.0744 (17) | 0.0583 (17) | 0.0399 (14) | 0.0075 (16) | 0.0252 (13) | 0.0018 (13) |
| O3 | 0.0294 (11) | 0.0432 (14) | 0.0507 (14) | 0.0076 (11) | 0.0000 (11) | −0.0008 (12) |
| C2 | 0.0501 (19) | 0.0310 (18) | 0.0353 (19) | 0.0002 (16) | 0.0102 (15) | 0.0002 (15) |
| C3 | 0.0500 (19) | 0.0281 (17) | 0.0274 (17) | 0.0009 (15) | 0.0029 (14) | −0.0045 (14) |
| C3A | 0.0305 (14) | 0.0260 (16) | 0.0233 (15) | −0.0010 (13) | −0.0010 (11) | −0.0018 (13) |
| C4 | 0.0326 (16) | 0.0393 (18) | 0.0261 (15) | 0.0081 (14) | −0.0029 (12) | 0.0022 (14) |
| C4A | 0.0240 (13) | 0.0245 (15) | 0.0252 (14) | 0.0024 (12) | 0.0008 (11) | −0.0011 (12) |
| C5 | 0.0255 (14) | 0.0283 (17) | 0.0330 (17) | −0.0014 (13) | −0.0005 (12) | −0.0081 (13) |
| C6 | 0.0352 (16) | 0.054 (2) | 0.0303 (17) | −0.0053 (16) | 0.0080 (13) | 0.0012 (16) |
| C7 | 0.0379 (16) | 0.0404 (19) | 0.0304 (17) | −0.0079 (15) | −0.0037 (13) | 0.0074 (15) |
| C7A | 0.0319 (15) | 0.0249 (16) | 0.0242 (15) | −0.0043 (12) | −0.0046 (12) | 0.0003 (13) |
| C8 | 0.0327 (15) | 0.0254 (16) | 0.0300 (16) | 0.0023 (13) | −0.0054 (12) | 0.0027 (13) |
| C9 | 0.0312 (15) | 0.0315 (17) | 0.0378 (18) | 0.0063 (14) | −0.0044 (14) | −0.0028 (14) |
| C9A | 0.0296 (14) | 0.0312 (17) | 0.0295 (16) | 0.0037 (12) | 0.0026 (12) | −0.0045 (14) |
| C10 | 0.047 (2) | 0.043 (2) | 0.048 (2) | 0.0062 (17) | −0.0088 (18) | 0.0134 (18) |
| C11 | 0.073 (3) | 0.063 (3) | 0.0277 (18) | 0.017 (2) | −0.0028 (17) | 0.0011 (18) |
| C12 | 0.0318 (15) | 0.0259 (17) | 0.0369 (18) | −0.0006 (13) | 0.0002 (13) | 0.0009 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.347 (4) | C7—C7A | 1.512 (4) |
| O1—C9A | 1.456 (3) | C7—H7 | 0.93 |
| O2—C2 | 1.202 (4) | C7A—C8 | 1.546 (4) |
| O3—C5 | 1.208 (4) | C7A—H7A | 0.98 |
| C2—C3 | 1.490 (5) | C8—C10 | 1.526 (4) |
| C3—C11 | 1.309 (5) | C8—C9 | 1.535 (4) |
| C3—C3A | 1.501 (4) | C8—H8 | 0.98 |

| | | | |
|---|---|---|---|
| C3A—C4 | 1.525 (4) | C9—C9A | 1.513 (4) |
| C3A—C9A | 1.541 (4) | C9—H9A | 0.97 |
| C3A—H3A | 0.98 | C9—H9B | 0.97 |
| C4—C4A | 1.544 (4) | C9A—H9C | 0.98 |
| C4—H4A | 0.97 | C10—H10A | 0.96 |
| C4—H4B | 0.97 | C10—H10B | 0.96 |
| C4A—C5 | 1.538 (4) | C10—H10C | 0.96 |
| C4A—C12 | 1.552 (4) | C11—H11A | 0.93 |
| C4A—C7A | 1.555 (4) | C11—H11B | 0.93 |
| C5—C6 | 1.463 (5) | C12—H12A | 0.96 |
| C6—C7 | 1.323 (5) | C12—H12B | 0.96 |
| C6—H6 | 0.93 | C12—H12C | 0.96 |

| | | | |
|---|---|---|---|
| C2—O1—C9A | 111.3 (2) | C7—C7A—H7A | 106.1 |
| O2—C2—O1 | 122.2 (3) | C8—C7A—H7A | 106.1 |
| O2—C2—C3 | 128.9 (3) | C4A—C7A—H7A | 106.1 |
| O1—C2—C3 | 108.9 (3) | C10—C8—C9 | 109.1 (3) |
| C11—C3—C2 | 123.2 (3) | C10—C8—C7A | 112.2 (3) |
| C11—C3—C3A | 130.0 (3) | C9—C8—C7A | 111.4 (2) |
| C2—C3—C3A | 106.8 (3) | C10—C8—H8 | 108 |
| C3—C3A—C4 | 115.9 (3) | C9—C8—H8 | 108 |
| C3—C3A—C9A | 101.9 (2) | C7A—C8—H8 | 108 |
| C4—C3A—C9A | 116.0 (2) | C9A—C9—C8 | 116.2 (2) |
| C3—C3A—H3A | 107.5 | C9A—C9—H9A | 108.2 |
| C4—C3A—H3A | 107.5 | C8—C9—H9A | 108.2 |
| C9A—C3A—H3A | 107.5 | C9A—C9—H9B | 108.2 |
| C3A—C4—C4A | 114.1 (2) | C8—C9—H9B | 108.2 |
| C3A—C4—H4A | 108.7 | H9A—C9—H9B | 107.4 |
| C4A—C4—H4A | 108.7 | O1—C9A—C9 | 108.2 (2) |
| C3A—C4—H4B | 108.7 | O1—C9A—C3A | 105.2 (2) |
| C4A—C4—H4B | 108.7 | C9—C9A—C3A | 114.9 (3) |
| H4A—C4—H4B | 107.6 | O1—C9A—H9C | 109.4 |
| C5—C4A—C4 | 111.6 (2) | C9—C9A—H9C | 109.4 |
| C5—C4A—C12 | 103.2 (2) | C3A—C9A—H9C | 109.4 |
| C4—C4A—C12 | 110.6 (2) | C8—C10—H10A | 109.5 |
| C5—C4A—C7A | 102.5 (2) | C8—C10—H10B | 109.5 |
| C4—C4A—C7A | 115.2 (2) | H10A—C10—H10B | 109.5 |
| C12—C4A—C7A | 112.7 (2) | C8—C10—H10C | 109.5 |
| O3—C5—C6 | 127.9 (3) | H10A—C10—H10C | 109.5 |
| O3—C5—C4A | 125.4 (3) | H10B—C10—H10C | 109.5 |
| C6—C5—C4A | 106.8 (3) | C3—C11—H11A | 120 |
| C7—C6—C5 | 110.3 (3) | C3—C11—H11B | 120 |
| C7—C6—H6 | 124.8 | H11A—C11—H11B | 120 |
| C5—C6—H6 | 124.8 | C4A—C12—H12A | 109.5 |
| C6—C7—C7A | 112.6 (3) | C4A—C12—H12B | 109.5 |
| C6—C7—H7 | 123.7 | H12A—C12—H12B | 109.5 |
| C7A—C7—H7 | 123.7 | C4A—C12—H12C | 109.5 |
| C7—C7A—C8 | 117.6 (2) | H12A—C12—H12C | 109.5 |

| | | | |
|---|---|---|---|
| C7—C7A—C4A | 102.8 (2) | H12B—C12—H12C | 109.5 |
| C8—C7A—C4A | 117.1 (2) | | |
| | | | |
| C9A—O1—C2—O2 | 175.7 (3) | C5—C6—C7—C7A | −1.4 (4) |
| C9A—O1—C2—C3 | −5.1 (4) | C6—C7—C7A—C8 | 145.1 (3) |
| O2—C2—C3—C11 | −13.6 (6) | C6—C7—C7A—C4A | 14.8 (3) |
| O1—C2—C3—C11 | 167.1 (3) | C5—C4A—C7A—C7 | −20.9 (3) |
| O2—C2—C3—C3A | 168.1 (4) | C4—C4A—C7A—C7 | −142.3 (3) |
| O1—C2—C3—C3A | −11.1 (4) | C12—C4A—C7A—C7 | 89.5 (3) |
| C11—C3—C3A—C4 | −30.1 (5) | C5—C4A—C7A—C8 | −151.4 (3) |
| C2—C3—C3A—C4 | 148.0 (3) | C4—C4A—C7A—C8 | 87.2 (3) |
| C11—C3—C3A—C9A | −157.0 (4) | C12—C4A—C7A—C8 | −41.0 (4) |
| C2—C3—C3A—C9A | 21.1 (3) | C7—C7A—C8—C10 | 40.3 (4) |
| C3—C3A—C4—C4A | −164.1 (3) | C4A—C7A—C8—C10 | 163.6 (3) |
| C9A—C3A—C4—C4A | −44.6 (4) | C7—C7A—C8—C9 | 163.0 (3) |
| C3A—C4—C4A—C5 | −147.0 (3) | C4A—C7A—C8—C9 | −73.8 (3) |
| C3A—C4—C4A—C12 | 98.7 (3) | C10—C8—C9—C9A | 176.3 (3) |
| C3A—C4—C4A—C7A | −30.6 (4) | C7A—C8—C9—C9A | 51.9 (4) |
| C4—C4A—C5—O3 | −36.1 (4) | C2—O1—C9A—C9 | 142.0 (3) |
| C12—C4A—C5—O3 | 82.7 (3) | C2—O1—C9A—C3A | 18.7 (3) |
| C7A—C4A—C5—O3 | −160.0 (3) | C8—C9—C9A—O1 | 172.3 (2) |
| C4—C4A—C5—C6 | 144.9 (3) | C8—C9—C9A—C3A | −70.5 (4) |
| C12—C4A—C5—C6 | −96.3 (3) | C3—C3A—C9A—O1 | −23.8 (3) |
| C7A—C4A—C5—C6 | 21.0 (3) | C4—C3A—C9A—O1 | −150.6 (3) |
| O3—C5—C6—C7 | 168.1 (3) | C3—C3A—C9A—C9 | −142.7 (3) |
| C4A—C5—C6—C7 | −13.0 (4) | C4—C3A—C9A—C9 | 90.5 (3) |