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# 4a-Hydroxy-9-(4-hydroxyphenyl)-4,4a,5,6,9,9a-hexahydro-3*H*-xanthene-1,8(2*H*,7*H*)-dione

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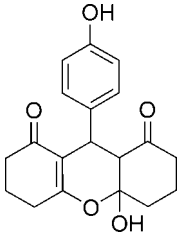
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.042; $wR$ factor = 0.113; data-to-parameter ratio = 8.5.

The title compound, $C_{19}H_{20}O_5$, was synthesized by the reaction of 1,3-cyclohexanedione and 4-hydroxybenzaldehyde in the presence of $PdCl_2$ and thiourea. The tetrahydropyran ring and the six-membered cyclohexene ring adopt envelope conformations, and the six-membered cyclohexane ring is in a chair conformation. The crystal packing is stabilized by classical intermolecular O—H···O hydrogen bonds and weak C—H···O interactions.

## Related literature

For applications of related compounds, see: Menchen *et al.* (2003); Saint-Ruf *et al.* (1972); Reddy *et al.* (2009); Mehdi *et al.* (2011). For the synthesis of related compounds, see: Karade *et al.* (2007); Luna *et al.* (2009). For related structures, see: Loh *et al.* (2011); Yang *et al.* (2011).



## Experimental

### Crystal data

$C_{19}H_{20}O_5$
$M_r$ = 328.35
Monoclinic, $P2_1$
$a$ = 9.014 (4) Å
$b$ = 10.242 (4) Å
$c$ = 9.289 (4) Å
$\beta$ = 108.194 (4)°
$V$ = 814.7 (6) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 295 K
0.35 × 0.30 × 0.20 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min}$ = 0.967, $T_{max}$ = 0.981
4840 measured reflections
1876 independent reflections
1652 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
$S$ = 1.04
1863 reflections
219 parameters
14 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4—H4···O5$^{i}$ | 0.82 | 2.07 | 2.852 (3) | 160 |
| O5—H5···O2$^{ii}$ | 0.82 | 1.94 | 2.758 (4) | 175 |
| C4—H4*B*···O4$^{iii}$ | 0.97 | 2.50 | 3.274 (5) | 137 |
| C8*A*—H13···O3$^{iv}$ | 0.98 | 2.59 | 3.535 (4) | 161 |
| C12—H18···O2$^{ii}$ | 0.93 | 2.55 | 3.251 (4) | 132 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2296).

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# supporting information

*Acta Cryst.* (2011). E**67**, o2751 [https://doi.org/10.1107/S1600536811038335]

## 4a-Hydroxy-9-(4-hydroxyphenyl)-4,4a,5,6,9,9a-hexahydro-3*H*-xanthene-1,8(2*H*,7*H*)-dione

**Liying Wang, Weicheng Lu, Yan Yang and Yulin Zhu**

### S1. Comment

Xanthenes are an important class of heterocyclic compounds which attract researchers by their spectroscopic and biological properties. Their derivatives had been widely used as dyes, fluorescent materials for visualization and in laser technologies (Menchen *et al.*, 2003; Saint-Ruf *et al.*, 1972; Reddy *et al.*, 2009; Mehdi *et al.*, 2011). Due to their wide range of applications, a well established method used for the construction of xanthene unit was set up, which was a Tandem Michael reaction between 1,3-cyclohexanedione and benzaldehyde (Luna *et al.*, 2009; Karade *et al.*, 2007). The reaction between 1,3-cyclohexanedione and 4-hydroxybenzaldehyde in the presence of thiourea and $PdCl_2$ proceeded to give the title compound in isolated yield 86% (Fig. 1).

The molecular structure of the title compound is illustrated in Fig. 2. There are no unusual bond lengths and angles in the molecule. The tricyclo system is connected with a phenly ring at the C9 position. The tetrahydropyran ring (O1/C4B/C8A/C9/C9A/C4A) and the six-membered cyclohexene ring (C1–C4/C4A/C9A) adopt envelope conformations, the other six-membered cyclohexane ring (C4B/C5–C8/C8A) is in a chair conformation. Other than the published structure 4*a*-hydroxy-9-(2-methoxyphenyl)-4,4*a*,5,6,7,8,9,9*a*-octahydro-3*H*-xanthene-1,8(2*H*)-dione or 3,4,4*a*,5,6,7,9,9*a*-octahydro-4*a*-hydroxyl-9-(4-chlorophenyl)-1*H*-xanthene-1,8(2*H*)-dione (Loh *et al.*, 2011; Yang *et al.*, 2011), the main structure of this compound is a derivated xanthene–dione fused tricyclo system with a hydroxyl group at its C4b position. The hydroxy group in phenly ring, tetrahydropyran ring with a hydroxyl group and carbonyl O atom allow the formation of two intermolecular O4—H4···O5$^{i}$ and O5—H5···O2$^{ii}$ hydrogen bonds. There are weak intermolecular C4—H4B···O4$^{iii}$, C8A—H13···O3$^{iv}$ and C12—H18···O2$^{ii}$ interactions which link molecules into chains. Symmetyry codes: (i) *x*+1, *y*, *z*+1; (ii) -*x*, *y*+1/2, -*z*+1; (iii) -*x*+1, *y*+1/2, -*z*+2; (iv) -*x*, *y*+1/2, -*z*+2.

### S2. Experimental

A mixture of 1,3-cyclohexanedione (1.12 g, 10 mmol), 4-hydroxybenzaldehyde (0.61 g, 5 mmol), thiourea (0.76 g,10 mmol) and $PdCl_2$ (0.0020 mg) was refluxed in anhydrous acetonitrile (12 ml) at 373 K for 4 h. After being cooled to room temperature, the reaction mixture was poured into water. The white precipitate was filtered off with a silica pad, washed twice with anhydrous ethanol, and the filtrate was then dried under vacuum to yield the product in yield of 86%. Single crystals of the title compound were obtained by slow evaporation from anhydrous ethanol at room temperature to yield colourless, block-shaped crystal.

### S3. Refinement

The H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98Å and O—H = 0.82Å, respectively. The $U_{iso}$ = 1.2$U_{eq}$(C) and $U_{iso}$ = 1.5$U_{eq}$(O). 3350 Friedel pairs were merged during the refinement.

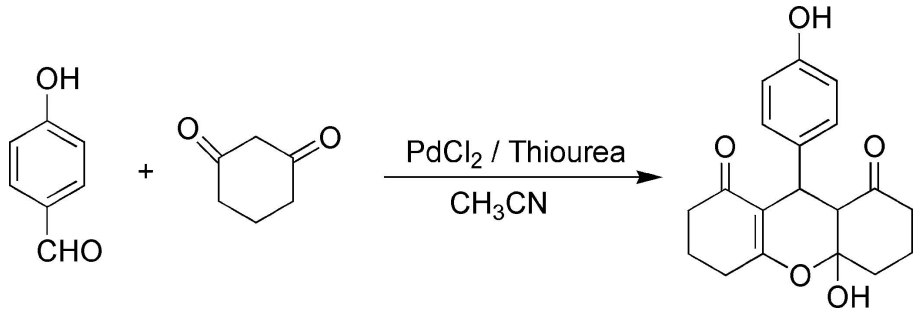


**Figure 1**

Palladium(II) chloride catalyzed synthesis of the title compound.

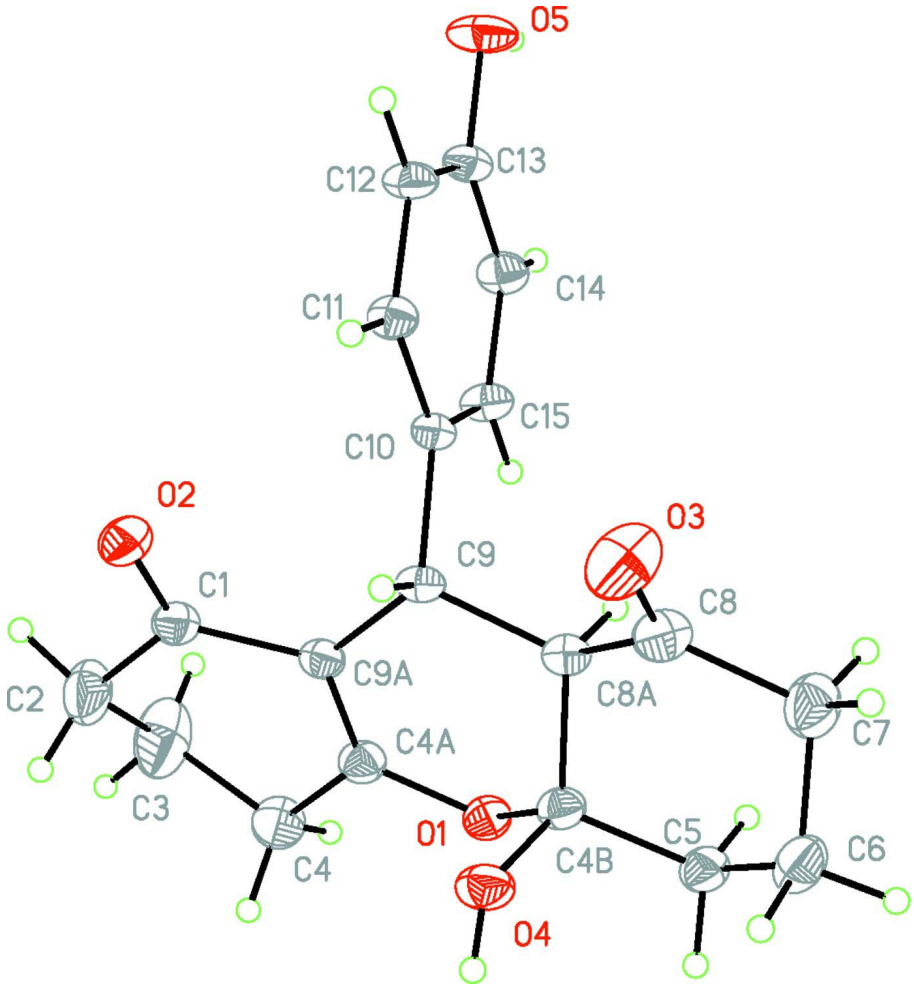


**Figure 2**

The molecular structure of title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

## 4a-Hydroxy-9-(4-hydroxyphenyl)-4,4a,5,6,9,9a-hexahydro-3*H*-xanthene- 1,8(2*H*,7*H*)-dione

*Crystal data*

$C_{19}H_{20}O_5$
$M_r = 328.35$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 9.014$ (4) Å
$b = 10.242$ (4) Å
$c = 9.289$ (4) Å
$\beta = 108.194$ (4)°
$V = 814.7$ (6) Å$^3$
$Z = 2$
$F(000) = 348.0$
$D_x = 1.339$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1885 reflections
$\theta = 2.4$–$26.8°$
$\mu = 0.10$ mm$^{-1}$
$T = 295$ K
Block, colourless
0.35 × 0.30 × 0.20 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$– and $\omega$–scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.967$, $T_{max} = 0.981$
4840 measured reflections
1876 independent reflections
1652 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 12$
$l = -10 \rightarrow 11$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.113$
$S = 1.04$
1863 reflections
219 parameters
14 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.1291P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å$^{-3}$
$\Delta\rho_{min} = -0.19$ e Å$^{-3}$

*Special details*

**Geometry**. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor w*R* 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 $F^2 > 2\sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C9 | 0.1903 (3) | 0.1561 (2) | 0.8768 (3) | 0.0289 (5) |
| H9 | 0.1954 | 0.0612 | 0.8912 | 0.035* |
| C9A | 0.3440 (3) | 0.2024 (3) | 0.8614 (3) | 0.0307 (5) |
| C4A | 0.4337 (3) | 0.2922 (3) | 0.9542 (3) | 0.0354 (6) |

| | | | | |
|---|---|---|---|---|
| C8A | 0.1606 (3) | 0.2192 (3) | 1.0149 (3) | 0.0307 (6) |
| H13 | 0.1037 | 0.3003 | 0.9782 | 0.037* |
| C5 | 0.2735 (3) | 0.3317 (3) | 1.2653 (3) | 0.0407 (7) |
| H5A | 0.2174 | 0.4116 | 1.2264 | 0.049* |
| H5B | 0.3707 | 0.3551 | 1.3417 | 0.049* |
| C8 | 0.0566 (3) | 0.1394 (3) | 1.0823 (3) | 0.0432 (7) |
| C6 | 0.1760 (4) | 0.2471 (4) | 1.3358 (4) | 0.0483 (8) |
| H6A | 0.1504 | 0.2962 | 1.4142 | 0.058* |
| H6B | 0.2362 | 0.1712 | 1.3826 | 0.058* |
| C1 | 0.4046 (3) | 0.1347 (3) | 0.7535 (3) | 0.0375 (6) |
| C4B | 0.3076 (3) | 0.2586 (3) | 1.1383 (3) | 0.0338 (6) |
| C4 | 0.5792 (4) | 0.3490 (4) | 0.9360 (4) | 0.0492 (8) |
| H4A | 0.6694 | 0.3118 | 1.0114 | 0.059* |
| H4B | 0.5803 | 0.4426 | 0.9525 | 0.059* |
| C7 | 0.0251 (4) | 0.2026 (4) | 1.2164 (4) | 0.0540 (9) |
| H7A | −0.0287 | 0.1409 | 1.2619 | 0.065* |
| H7B | −0.0428 | 0.2774 | 1.1820 | 0.065* |
| C2 | 0.5553 (4) | 0.1824 (4) | 0.7353 (5) | 0.0656 (11) |
| H2A | 0.6403 | 0.1290 | 0.7971 | 0.079* |
| H2B | 0.5513 | 0.1715 | 0.6304 | 0.079* |
| C3 | 0.5892 (6) | 0.3217 (5) | 0.7787 (6) | 0.0818 (15) |
| H3A | 0.5151 | 0.3768 | 0.7058 | 0.098* |
| H3B | 0.6930 | 0.3433 | 0.7760 | 0.098* |
| O1 | 0.4005 (2) | 0.34289 (19) | 1.0760 (2) | 0.0357 (4) |
| O2 | 0.3391 (3) | 0.0380 (2) | 0.6848 (3) | 0.0487 (6) |
| O4 | 0.3938 (2) | 0.1433 (2) | 1.1914 (2) | 0.0435 (5) |
| H4 | 0.4795 | 0.1624 | 1.2506 | 0.065* |
| O5 | −0.3220 (3) | 0.2708 (3) | 0.3538 (3) | 0.0548 (6) |
| H5 | −0.3326 | 0.3497 | 0.3398 | 0.082* |
| O3 | 0.0004 (4) | 0.0370 (3) | 1.0282 (3) | 0.0698 (8) |
| C13 | −0.1962 (3) | 0.2473 (3) | 0.4795 (3) | 0.0340 (6) |
| C11 | 0.0184 (3) | 0.3158 (3) | 0.6905 (3) | 0.0350 (6) |
| H19 | 0.0782 | 0.3830 | 0.7472 | 0.042* |
| C10 | 0.0564 (3) | 0.1873 (3) | 0.7339 (3) | 0.0275 (5) |
| C14 | −0.1588 (3) | 0.1177 (3) | 0.5195 (3) | 0.0376 (6) |
| H16 | −0.2179 | 0.0507 | 0.4616 | 0.045* |
| C15 | −0.0334 (3) | 0.0891 (3) | 0.6455 (3) | 0.0323 (6) |
| H15 | −0.0086 | 0.0024 | 0.6717 | 0.039* |
| C12 | −0.1073 (3) | 0.3459 (3) | 0.5641 (3) | 0.0365 (6) |
| H18 | −0.1312 | 0.4325 | 0.5367 | 0.044* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C9 | 0.0264 (11) | 0.0269 (13) | 0.0275 (13) | −0.0013 (10) | 0.0000 (10) | 0.0017 (10) |
| C9A | 0.0270 (11) | 0.0307 (13) | 0.0306 (13) | 0.0014 (10) | 0.0034 (10) | 0.0007 (11) |
| C4A | 0.0330 (12) | 0.0342 (14) | 0.0358 (14) | −0.0032 (11) | 0.0062 (11) | −0.0044 (12) |
| C8A | 0.0306 (12) | 0.0304 (14) | 0.0278 (13) | −0.0016 (10) | 0.0045 (10) | 0.0013 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0407 (14) | 0.0428 (17) | 0.0348 (14) | 0.0025 (13) | 0.0064 (11) | −0.0078 (13) |
| C8 | 0.0421 (14) | 0.0477 (17) | 0.0379 (15) | −0.0097 (14) | 0.0099 (12) | 0.0021 (14) |
| C6 | 0.0621 (19) | 0.0514 (19) | 0.0340 (15) | 0.0075 (16) | 0.0186 (14) | 0.0012 (14) |
| C1 | 0.0362 (13) | 0.0389 (15) | 0.0330 (14) | 0.0042 (12) | 0.0044 (11) | −0.0019 (12) |
| C4B | 0.0330 (13) | 0.0341 (14) | 0.0301 (14) | −0.0014 (11) | 0.0038 (11) | −0.0037 (11) |
| C4 | 0.0409 (15) | 0.0504 (18) | 0.0570 (19) | −0.0156 (14) | 0.0165 (14) | −0.0104 (16) |
| C7 | 0.0548 (19) | 0.061 (2) | 0.053 (2) | −0.0116 (17) | 0.0268 (16) | −0.0054 (18) |
| C2 | 0.0526 (19) | 0.079 (3) | 0.075 (3) | −0.0129 (19) | 0.0353 (19) | −0.029 (2) |
| C3 | 0.089 (3) | 0.077 (3) | 0.105 (3) | −0.036 (3) | 0.066 (3) | −0.027 (3) |
| O1 | 0.0346 (9) | 0.0329 (10) | 0.0368 (10) | −0.0065 (8) | 0.0074 (8) | −0.0081 (8) |
| O2 | 0.0484 (12) | 0.0473 (13) | 0.0444 (13) | 0.0007 (10) | 0.0059 (10) | −0.0167 (10) |
| O4 | 0.0444 (11) | 0.0390 (11) | 0.0378 (11) | 0.0083 (9) | −0.0005 (8) | 0.0008 (9) |
| O5 | 0.0443 (11) | 0.0498 (14) | 0.0473 (13) | 0.0012 (10) | −0.0190 (9) | 0.0032 (11) |
| O3 | 0.0865 (18) | 0.0670 (18) | 0.0645 (18) | −0.0429 (15) | 0.0359 (15) | −0.0146 (14) |
| C13 | 0.0264 (12) | 0.0407 (15) | 0.0285 (13) | 0.0015 (11) | −0.0008 (10) | 0.0030 (11) |
| C11 | 0.0354 (13) | 0.0309 (14) | 0.0306 (13) | −0.0029 (11) | −0.0014 (10) | 0.0005 (11) |
| C10 | 0.0235 (11) | 0.0307 (13) | 0.0250 (12) | −0.0002 (10) | 0.0031 (9) | 0.0025 (10) |
| C14 | 0.0300 (12) | 0.0396 (16) | 0.0351 (14) | −0.0079 (11) | −0.0017 (11) | −0.0065 (12) |
| C15 | 0.0319 (12) | 0.0268 (13) | 0.0345 (14) | −0.0013 (10) | 0.0050 (11) | 0.0004 (11) |
| C12 | 0.0384 (13) | 0.0300 (14) | 0.0344 (14) | 0.0038 (11) | 0.0017 (11) | 0.0051 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C9—C9A | 1.513 (3) | C4—C3 | 1.518 (6) |
| C9—C10 | 1.523 (3) | C4—H4A | 0.9700 |
| C9—C8A | 1.533 (4) | C4—H4B | 0.9700 |
| C9—H9 | 0.9800 | C7—H7A | 0.9700 |
| C9A—C4A | 1.345 (4) | C7—H7B | 0.9700 |
| C9A—C1 | 1.458 (4) | C2—C3 | 1.487 (6) |
| C4A—O1 | 1.360 (3) | C2—H2A | 0.9700 |
| C4A—C4 | 1.492 (4) | C2—H2B | 0.9700 |
| C8A—C4B | 1.512 (3) | C3—H3A | 0.9700 |
| C8A—C8 | 1.518 (4) | C3—H3B | 0.9700 |
| C8A—H13 | 0.9800 | O4—H4 | 0.8200 |
| C5—C4B | 1.508 (4) | O5—C13 | 1.371 (3) |
| C5—C6 | 1.521 (5) | O5—H5 | 0.8200 |
| C5—H5A | 0.9700 | C13—C12 | 1.373 (4) |
| C5—H5B | 0.9700 | C13—C14 | 1.391 (4) |
| C8—O3 | 1.204 (4) | C11—C12 | 1.388 (4) |
| C8—C7 | 1.507 (5) | C11—C10 | 1.388 (4) |
| C6—C7 | 1.532 (5) | C11—H19 | 0.9300 |
| C6—H6A | 0.9700 | C10—C15 | 1.387 (4) |
| C6—H6B | 0.9700 | C14—C15 | 1.381 (4) |
| C1—O2 | 1.225 (4) | C14—H16 | 0.9300 |
| C1—C2 | 1.502 (5) | C15—H15 | 0.9300 |
| C4B—O4 | 1.416 (4) | C12—H18 | 0.9300 |
| C4B—O1 | 1.443 (3) | | |

| | | | |
|---|---|---|---|
| C9A—C9—C10 | 110.6 (2) | C3—C4—H4A | 109.5 |
| C9A—C9—C8A | 110.6 (2) | C4A—C4—H4B | 109.5 |
| C10—C9—C8A | 110.0 (2) | C3—C4—H4B | 109.5 |
| C9A—C9—H9 | 108.5 | H4A—C4—H4B | 108.1 |
| C10—C9—H9 | 108.5 | C8—C7—C6 | 111.9 (3) |
| C8A—C9—H9 | 108.5 | C8—C7—H7A | 109.2 |
| C4A—C9A—C1 | 119.1 (2) | C6—C7—H7A | 109.2 |
| C4A—C9A—C9 | 122.6 (2) | C8—C7—H7B | 109.2 |
| C1—C9A—C9 | 117.8 (2) | C6—C7—H7B | 109.2 |
| C9A—C4A—O1 | 123.3 (2) | H7A—C7—H7B | 107.9 |
| C9A—C4A—C4 | 124.6 (3) | C3—C2—C1 | 113.4 (3) |
| O1—C4A—C4 | 112.0 (2) | C3—C2—H2A | 108.9 |
| C4B—C8A—C8 | 109.8 (2) | C1—C2—H2A | 108.9 |
| C4B—C8A—C9 | 114.0 (2) | C3—C2—H2B | 108.9 |
| C8—C8A—C9 | 114.2 (2) | C1—C2—H2B | 108.9 |
| C4B—C8A—H13 | 106.0 | H2A—C2—H2B | 107.7 |
| C8—C8A—H13 | 106.0 | C2—C3—C4 | 111.7 (4) |
| C9—C8A—H13 | 106.0 | C2—C3—H3A | 109.3 |
| C4B—C5—C6 | 109.9 (3) | C4—C3—H3A | 109.3 |
| C4B—C5—H5A | 109.7 | C2—C3—H3B | 109.3 |
| C6—C5—H5A | 109.7 | C4—C3—H3B | 109.3 |
| C4B—C5—H5B | 109.7 | H3A—C3—H3B | 107.9 |
| C6—C5—H5B | 109.7 | C4A—O1—C4B | 114.3 (2) |
| H5A—C5—H5B | 108.2 | C4B—O4—H4 | 109.5 |
| O3—C8—C7 | 123.7 (3) | C13—O5—H5 | 109.5 |
| O3—C8—C8A | 122.0 (3) | O5—C13—C12 | 122.5 (3) |
| C7—C8—C8A | 114.2 (3) | O5—C13—C14 | 117.5 (2) |
| C5—C6—C7 | 111.1 (3) | C12—C13—C14 | 120.0 (2) |
| C5—C6—H6A | 109.4 | C12—C11—C10 | 121.2 (3) |
| C7—C6—H6A | 109.4 | C12—C11—H19 | 119.4 |
| C5—C6—H6B | 109.4 | C10—C11—H19 | 119.4 |
| C7—C6—H6B | 109.4 | C15—C10—C11 | 118.0 (2) |
| H6A—C6—H6B | 108.0 | C15—C10—C9 | 121.4 (2) |
| O2—C1—C9A | 121.3 (3) | C11—C10—C9 | 120.6 (2) |
| O2—C1—C2 | 120.7 (3) | C15—C14—C13 | 119.6 (3) |
| C9A—C1—C2 | 117.9 (3) | C15—C14—H16 | 120.2 |
| O4—C4B—O1 | 108.4 (2) | C13—C14—H16 | 120.2 |
| O4—C4B—C5 | 111.4 (2) | C14—C15—C10 | 121.3 (3) |
| O1—C4B—C5 | 107.6 (2) | C14—C15—H15 | 119.3 |
| O4—C4B—C8A | 107.3 (2) | C10—C15—H15 | 119.3 |
| O1—C4B—C8A | 109.7 (2) | C13—C12—C11 | 119.8 (3) |
| C5—C4B—C8A | 112.4 (2) | C13—C12—H18 | 120.1 |
| C4A—C4—C3 | 110.8 (3) | C11—C12—H18 | 120.1 |
| C4A—C4—H4A | 109.5 | | |

| | | | |
|---|---|---|---|
| C10—C9—C9A—C4A | 118.8 (3) | C9—C8A—C4B—C5 | 174.6 (2) |
| C8A—C9—C9A—C4A | −3.4 (3) | C9A—C4A—C4—C3 | 15.5 (5) |
| C10—C9—C9A—C1 | −68.9 (3) | O1—C4A—C4—C3 | −163.9 (3) |

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| C8A—C9—C9A—C1 | 169.0 (2) | O3—C8—C7—C6 | 132.1 (4) |
| C1—C9A—C4A—O1 | −167.6 (3) | C8A—C8—C7—C6 | −50.7 (4) |
| C9—C9A—C4A—O1 | 4.7 (4) | C5—C6—C7—C8 | 52.4 (4) |
| C1—C9A—C4A—C4 | 13.1 (4) | O2—C1—C2—C3 | 159.2 (4) |
| C9—C9A—C4A—C4 | −174.7 (3) | C9A—C1—C2—C3 | −24.1 (5) |
| C9A—C9—C8A—C4B | −26.3 (3) | C1—C2—C3—C4 | 52.2 (6) |
| C10—C9—C8A—C4B | −148.8 (2) | C4A—C4—C3—C2 | −47.4 (5) |
| C9A—C9—C8A—C8 | −153.6 (2) | C9A—C4A—O1—C4B | 25.7 (4) |
| C10—C9—C8A—C8 | 83.9 (3) | C4—C4A—O1—C4B | −154.9 (2) |
| C4B—C8A—C8—O3 | −131.1 (3) | O4—C4B—O1—C4A | 62.6 (3) |
| C9—C8A—C8—O3 | −1.7 (4) | C5—C4B—O1—C4A | −176.7 (2) |
| C4B—C8A—C8—C7 | 51.6 (3) | C8A—C4B—O1—C4A | −54.2 (3) |
| C9—C8A—C8—C7 | −179.0 (3) | C12—C11—C10—C15 | 1.1 (4) |
| C4B—C5—C6—C7 | −56.4 (4) | C12—C11—C10—C9 | −177.3 (2) |
| C4A—C9A—C1—O2 | 167.8 (3) | C9A—C9—C10—C15 | 117.6 (3) |
| C9—C9A—C1—O2 | −4.8 (4) | C8A—C9—C10—C15 | −120.0 (2) |
| C4A—C9A—C1—C2 | −8.8 (4) | C9A—C9—C10—C11 | −64.0 (3) |
| C9—C9A—C1—C2 | 178.6 (3) | C8A—C9—C10—C11 | 58.4 (3) |
| C6—C5—C4B—O4 | −61.1 (3) | O5—C13—C14—C15 | −179.8 (3) |
| C6—C5—C4B—O1 | −179.8 (2) | C12—C13—C14—C15 | 0.9 (4) |
| C6—C5—C4B—C8A | 59.3 (3) | C13—C14—C15—C10 | 0.2 (4) |
| C8—C8A—C4B—O4 | 67.0 (3) | C11—C10—C15—C14 | −1.1 (4) |
| C9—C8A—C4B—O4 | −62.6 (3) | C9—C10—C15—C14 | 177.3 (2) |
| C8—C8A—C4B—O1 | −175.5 (2) | O5—C13—C12—C11 | 179.8 (3) |
| C9—C8A—C4B—O1 | 54.9 (3) | C14—C13—C12—C11 | −0.9 (4) |
| C8—C8A—C4B—C5 | −55.9 (3) | C10—C11—C12—C13 | −0.1 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O4—H4···O5$^{i}$ | 0.82 | 2.07 | 2.852 (3) | 160 |
| O5—H5···O2$^{ii}$ | 0.82 | 1.94 | 2.758 (4) | 175 |
| C4—H4*B*···O4$^{iii}$ | 0.97 | 2.50 | 3.274 (5) | 137 |
| C8*A*—H13···O3$^{iv}$ | 0.98 | 2.59 | 3.535 (4) | 161 |
| C12—H18···O2$^{ii}$ | 0.93 | 2.55 | 3.251 (4) | 132 |

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