Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 2a,3a-Dihydroxyandrostan-16-one

**Li Zhang,[a] Xin Fang,[b] Xiao-Jiang Hao[b] and Yang Lu[a]***

[a]Institute of Materia Medica, Chinese Academy of Medical Sciences, and Peking Union Medical College, 1 Xiannong Tan Street, Beijing 100050, People's Republic of China, and [b]State Key Laboratory of Phytochemistry and Plant Resources in West China, Kunming Institute of Botany, Chinese Academy of Sciences, Kunming 650204, Yunnan, People's Republic of China
Correspondence e-mail: luy@imm.ac.cn

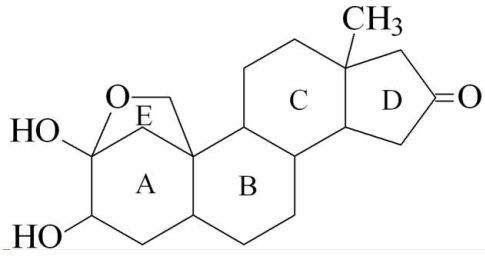
Received 24 June 2009; accepted 24 July 2009

Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.038; $wR$ factor = 0.105; data-to-parameter ratio = 9.9.

The title compound, $C_{19}H_{28}O_4$, is a new androstane steroid derivative. In the crystal, molecules are linked along the $a$ axis by intermolecular O—H···O hydrogen bonds.

## Related literature

The title compound was obtained from the methanol extract of stems of *Trichilia claussenii* by column chromatograph, see: Pupo *et al.* (1997). It shows strong insecticidal activity, see: Champagne *et al.* (1992).




## Experimental

### Crystal data

$C_{19}H_{28}O_4$ $M_r$ = 320.41
Monoclinic, $P2_1$
$a$ = 10.8687 (2) Å
$b$ = 6.3379 (1) Å
$c$ = 12.9038 (2) Å
$\beta$ = 112.882 (1)°
$V$ = 818.93 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 295 K
0.20 × 0.20 × 0.20 mm

### Data collection

MAC DIP 2030K diffractometer
Absorption correction: none
2065 measured reflections
2065 independent reflections
1929 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.015

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
$S$ = 1.14
2065 reflections
208 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.15 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H2$A$···O1$^{i}$ | 0.82 | 2.03 | 2.8088 (19) | 158 |

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

Data collection: *DENZO* (Otwinowski & Minor, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

We acknowledge financial support from the International Centre for Diffraction Data, Pennsylvania, USA.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2087).

## References

Champagne, D. E., Koul, O., Isman, M. B., Scudder, G. G. E. & Towers, G. H. N. (1992). *Phytochemistry*, **31**, 377–394.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Pupo, M. T., Vieira, P. C., Fernandes, J. B., da Silva, M. F. & Fo, R. E. (1997). *Phytochemistry*, **45**, 1495–1500.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2009). E**65**, o2043 [doi:10.1107/S1600536809029493]

## 2a,3a-Dihydroxyandrostan-16-one

**Li Zhang, Xin Fang, Xiao-Jiang Hao and Yang Lu**

### S1. Comment

The title compound,(I), was yielded from the methanol extract of stems of Trichilia claussenii by column chromatographies (Pupo *et al.*, 1997) and recrystasllized from methanol-hexane(1:1). As it shows strong biological activities against insects (Champagne *et al.*, 1992) we have determined its crystal stucture, Fig. 1, Table 1.

In this structure, rings A,B and C adopt chair conformations, and both ring D,*E* adopt envelope conformations. The dihedral angle between the least-squares plane through the 6 atoms of rings A and B is 9.2 (1)°, and that between rings B and C and that between rings C and D are 175.0 (2)° and 2.5 (1)°, respectively.

Besides, the molecules of the title compound are linked into each other along *a* axis, by intermolecular hydrogen bonds O—H···O. Atom O2 acts as hydrogen-bond donor to atom O1 at (-*x*, *y* + 1/2, -*z* + 1). Molecules pack in ribbons along the *b* axis, Fig. 2.

### S2. Experimental

The title compound was prepared according to the procedure of extracting Trichilia claussenii (Pupo *et al.*, 1997). At the temprature of 283 K and unventilated condition, single crystals of (I) were obtained from mixed solvent of methanol-hexane(1:1) within two weeks.

### S3. Refinement

In the absence of significant anomalous dispersion effects, Freidel pairs were merged. The position of the hydroxy H atoms were refined freely along with an isotropic displacement parameter $U_{iso}$(H)= 1.2$U_{eq}$(C). The methyl H atoms were then constrained to an ideal geometry with C—H distances of 0.96 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C), but each group was allowed to rotate freely about its C—C bond. All the other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range of 0.92–0.98 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C).

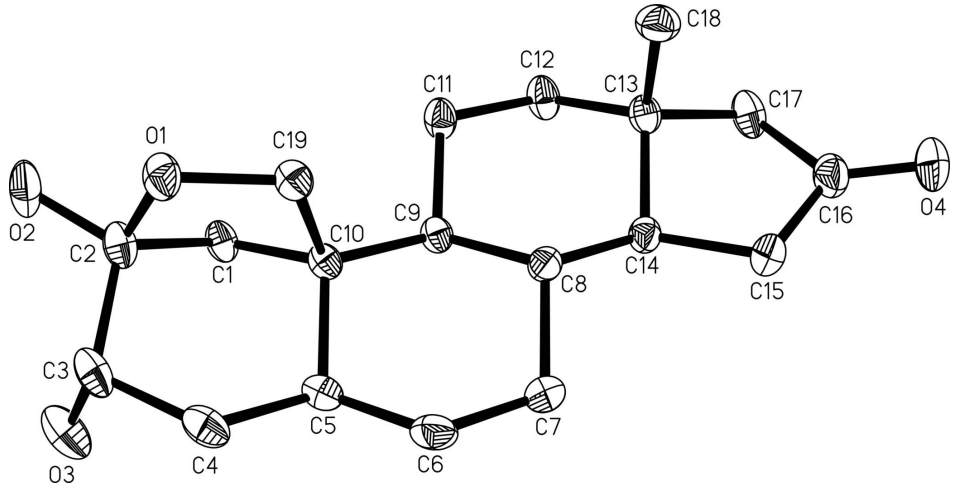



**Figure 1**

View of the molecule showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

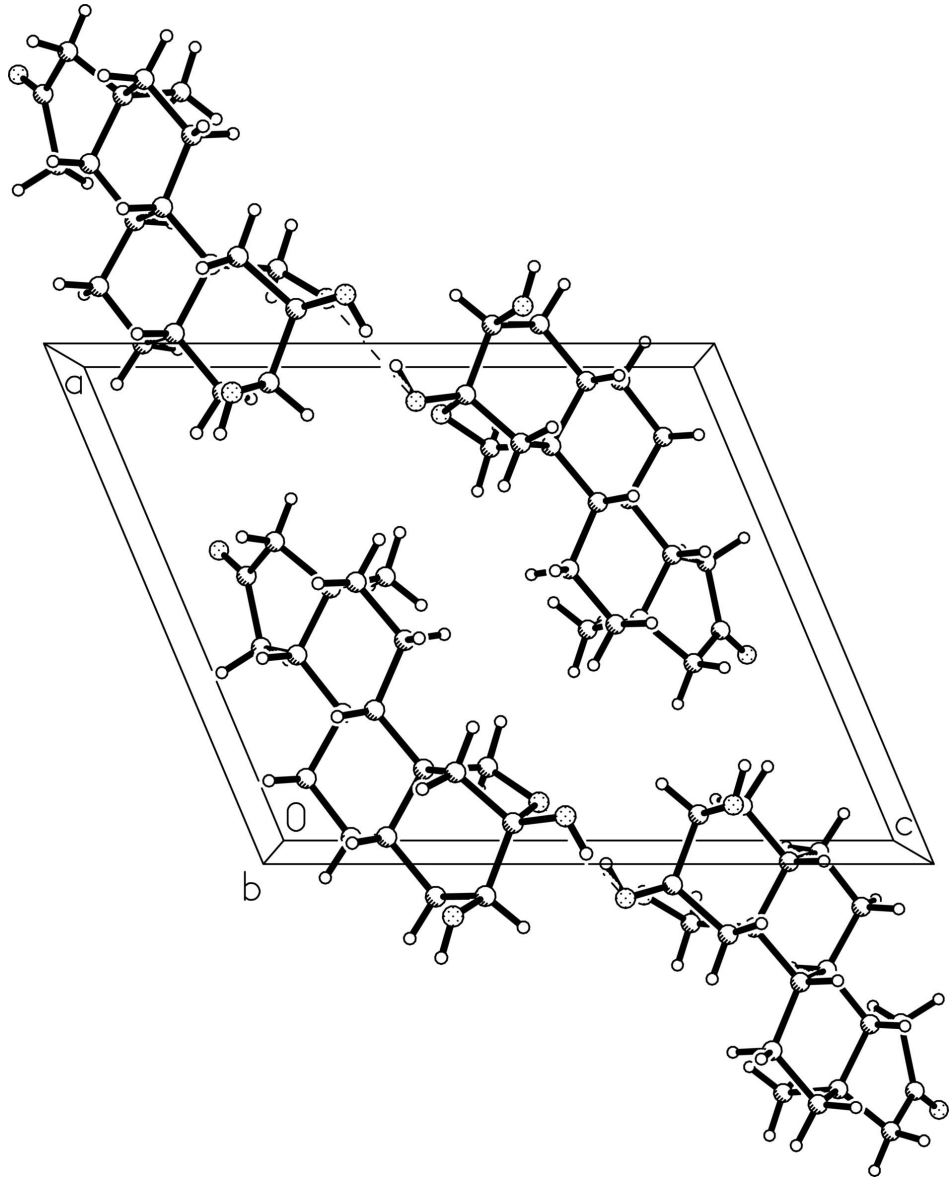



**Figure 2**

The molecular packing of (I) viewed down the *b* axis.

### 2a,3a-Dihydroxyandrostan-16-one

*Crystal data*

$C_{19}H_{28}O_4$
$M_r = 320.41$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 10.8687\ (2)$ Å
$b = 6.3379\ (1)$ Å
$c = 12.9038\ (2)$ Å
$\beta = 112.882\ (1)°$
$V = 818.93\ (2)$ Å$^3$
$Z = 2$
$F(000) = 348$
$D_x = 1.299$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2065 reflections
$\theta = 1.7–27.6°$
$\mu = 0.09$ mm$^{-1}$
$T = 295$ K
Block, colourless
0.20 × 0.20 × 0.20 mm

*Data collection*

MAC DIP 2030K diffractometer
Radiation source: rotate anode
Graphite monochromator
Detector resolution: 0 pixels mm$^{-1}$
$\omega$ scans
2065 measured reflections
2065 independent reflections
1929 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 27.6°$, $\theta_{min} = 1.7°$
$h = -14 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.105$
$S = 1.14$
2065 reflections
208 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.0933P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å$^{-3}$
$\Delta\rho_{min} = -0.15$ 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 | 0.10604 (14) | 0.6665 (3) | 0.44798 (11) | 0.0387 (3) |
| O2 | 0.09261 (16) | 1.0230 (3) | 0.48050 (15) | 0.0530 (5) |
| H2A | 0.0221 | 1.0426 | 0.4882 | 0.080* |
| O3 | −0.09685 (18) | 1.0625 (3) | 0.25157 (18) | 0.0597 (5) |
| H3A | −0.1755 | 1.0667 | 0.2083 | 0.089* |
| O4 | 0.61221 (19) | 0.0525 (4) | 0.09632 (16) | 0.0657 (5) |
| C1 | 0.17314 (18) | 0.9035 (3) | 0.34250 (16) | 0.0321 (4) |
| H1A | 0.2610 | 0.9394 | 0.3976 | 0.038* |
| H1B | 0.1443 | 1.0113 | 0.2846 | 0.038* |
| C2 | 0.07349 (19) | 0.8750 (3) | 0.39679 (18) | 0.0371 (4) |
| C3 | −0.0693 (2) | 0.8638 (4) | 0.3084 (2) | 0.0423 (5) |
| H3B | −0.1314 | 0.8411 | 0.3457 | 0.051* |
| C4 | −0.08066 (18) | 0.6822 (4) | 0.22815 (18) | 0.0419 (5) |
| H4B | −0.1622 | 0.6999 | 0.1618 | 0.050* |
| H4C | −0.0886 | 0.5516 | 0.2642 | 0.050* |
| C5 | 0.03684 (17) | 0.6613 (4) | 0.19065 (15) | 0.0337 (4) |
| H5A | 0.0300 | 0.7763 | 0.1379 | 0.040* |

| | | | | |
|---|---|---|---|---|
| C6 | 0.02645 (19) | 0.4533 (4) | 0.12841 (17) | 0.0418 (5) |
| H6A | 0.0208 | 0.3383 | 0.1760 | 0.050* |
| H6B | −0.0550 | 0.4533 | 0.0611 | 0.050* |
| C7 | 0.14492 (19) | 0.4159 (4) | 0.09552 (16) | 0.0425 (5) |
| H7A | 0.1436 | 0.5195 | 0.0398 | 0.051* |
| H7B | 0.1371 | 0.2771 | 0.0618 | 0.051* |
| C8 | 0.27756 (17) | 0.4311 (3) | 0.19700 (14) | 0.0294 (4) |
| H8A | 0.2807 | 0.3202 | 0.2509 | 0.035* |
| C9 | 0.28860 (17) | 0.6477 (3) | 0.25428 (14) | 0.0278 (4) |
| H9 | 0.2777 | 0.7530 | 0.1958 | 0.033* |
| C10 | 0.17217 (16) | 0.6848 (3) | 0.29237 (14) | 0.0273 (4) |
| C11 | 0.42783 (17) | 0.6878 (4) | 0.34704 (16) | 0.0357 (4) |
| H11A | 0.4325 | 0.8331 | 0.3720 | 0.043* |
| H11B | 0.4390 | 0.5978 | 0.4109 | 0.043* |
| C12 | 0.54289 (19) | 0.6468 (3) | 0.30905 (19) | 0.0383 (5) |
| H12A | 0.5399 | 0.7492 | 0.2523 | 0.046* |
| H12B | 0.6275 | 0.6625 | 0.3725 | 0.046* |
| C13 | 0.53218 (18) | 0.4254 (3) | 0.26115 (15) | 0.0314 (4) |
| C14 | 0.39575 (18) | 0.4035 (3) | 0.16258 (15) | 0.0311 (4) |
| H14 | 0.3903 | 0.5191 | 0.1105 | 0.037* |
| C15 | 0.4097 (2) | 0.2001 (4) | 0.10396 (17) | 0.0429 (5) |
| H15A | 0.3818 | 0.0783 | 0.1349 | 0.051* |
| H15B | 0.3572 | 0.2069 | 0.0237 | 0.051* |
| C16 | 0.5576 (2) | 0.1902 (5) | 0.12818 (18) | 0.0455 (5) |
| C17 | 0.6267 (2) | 0.3747 (4) | 0.2021 (2) | 0.0451 (5) |
| H17A | 0.6348 | 0.4934 | 0.1577 | 0.054* |
| H17B | 0.7147 | 0.3357 | 0.2558 | 0.054* |
| C18 | 0.5558 (2) | 0.2595 (4) | 0.35272 (18) | 0.0428 (5) |
| H18A | 0.4971 | 0.2857 | 0.3910 | 0.064* |
| H18B | 0.6468 | 0.2667 | 0.4056 | 0.064* |
| H18C | 0.5382 | 0.1217 | 0.3192 | 0.064* |
| C19 | 0.17183 (18) | 0.5448 (3) | 0.39026 (15) | 0.0314 (4) |
| H19A | 0.1239 | 0.4144 | 0.3617 | 0.038* |
| H19B | 0.2624 | 0.5108 | 0.4406 | 0.038* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0390 (7) | 0.0436 (8) | 0.0434 (7) | 0.0018 (7) | 0.0270 (6) | 0.0048 (7) |
| O2 | 0.0462 (9) | 0.0557 (11) | 0.0714 (10) | −0.0022 (8) | 0.0384 (8) | −0.0216 (9) |
| O3 | 0.0445 (9) | 0.0469 (10) | 0.0889 (13) | 0.0149 (8) | 0.0274 (9) | 0.0156 (10) |
| O4 | 0.0664 (11) | 0.0689 (13) | 0.0684 (11) | 0.0217 (10) | 0.0334 (9) | −0.0171 (10) |
| C1 | 0.0285 (8) | 0.0285 (10) | 0.0453 (10) | −0.0002 (7) | 0.0209 (8) | 0.0004 (8) |
| C2 | 0.0349 (10) | 0.0361 (11) | 0.0491 (11) | 0.0015 (8) | 0.0260 (9) | −0.0001 (9) |
| C3 | 0.0313 (10) | 0.0423 (12) | 0.0615 (13) | 0.0066 (9) | 0.0271 (10) | 0.0073 (11) |
| C4 | 0.0241 (9) | 0.0481 (12) | 0.0516 (11) | −0.0003 (9) | 0.0128 (8) | 0.0063 (11) |
| C5 | 0.0270 (8) | 0.0364 (10) | 0.0349 (8) | 0.0024 (8) | 0.0088 (7) | 0.0054 (9) |
| C6 | 0.0275 (9) | 0.0485 (13) | 0.0389 (10) | −0.0027 (9) | 0.0015 (8) | −0.0040 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7 | 0.0374 (10) | 0.0499 (13) | 0.0332 (9) | 0.0025 (10) | 0.0062 (8) | −0.0093 (10) |
| C8 | 0.0299 (8) | 0.0310 (9) | 0.0274 (8) | −0.0005 (7) | 0.0113 (7) | −0.0021 (7) |
| C9 | 0.0274 (8) | 0.0281 (9) | 0.0321 (8) | 0.0000 (7) | 0.0160 (7) | 0.0019 (7) |
| C10 | 0.0249 (8) | 0.0268 (8) | 0.0326 (8) | 0.0000 (7) | 0.0138 (6) | 0.0033 (7) |
| C11 | 0.0277 (8) | 0.0367 (10) | 0.0460 (10) | −0.0052 (8) | 0.0177 (8) | −0.0122 (9) |
| C12 | 0.0307 (9) | 0.0337 (11) | 0.0563 (12) | −0.0050 (8) | 0.0235 (9) | −0.0087 (9) |
| C13 | 0.0309 (9) | 0.0295 (9) | 0.0386 (9) | −0.0002 (8) | 0.0187 (8) | −0.0001 (8) |
| C14 | 0.0377 (9) | 0.0296 (10) | 0.0289 (8) | 0.0043 (8) | 0.0162 (7) | 0.0018 (8) |
| C15 | 0.0490 (11) | 0.0444 (12) | 0.0359 (9) | 0.0057 (11) | 0.0173 (9) | −0.0081 (9) |
| C16 | 0.0533 (12) | 0.0482 (13) | 0.0422 (10) | 0.0127 (11) | 0.0264 (9) | 0.0016 (11) |
| C17 | 0.0426 (11) | 0.0412 (12) | 0.0630 (13) | 0.0060 (10) | 0.0331 (10) | −0.0001 (11) |
| C18 | 0.0453 (12) | 0.0409 (11) | 0.0363 (10) | 0.0043 (9) | 0.0093 (9) | 0.0064 (9) |
| C19 | 0.0308 (8) | 0.0320 (10) | 0.0352 (9) | 0.0006 (8) | 0.0171 (7) | 0.0054 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C19 | 1.440 (2) | C8—H8A | 0.9800 |
| O1—C2 | 1.458 (3) | C9—C11 | 1.542 (2) |
| O2—C2 | 1.384 (3) | C9—C10 | 1.543 (2) |
| O2—H2A | 0.8200 | C9—H9 | 0.9800 |
| O3—C3 | 1.429 (3) | C10—C19 | 1.545 (2) |
| O3—H3A | 0.8200 | C11—C12 | 1.533 (2) |
| O4—C16 | 1.214 (3) | C11—H11A | 0.9700 |
| C1—C2 | 1.512 (2) | C11—H11B | 0.9700 |
| C1—C10 | 1.528 (3) | C12—C13 | 1.519 (3) |
| C1—H1A | 0.9700 | C12—H12A | 0.9700 |
| C1—H1B | 0.9700 | C12—H12B | 0.9700 |
| C2—C3 | 1.529 (3) | C13—C18 | 1.527 (3) |
| C3—C4 | 1.521 (4) | C13—C17 | 1.532 (3) |
| C3—H3B | 0.9800 | C13—C14 | 1.539 (3) |
| C4—C5 | 1.537 (3) | C14—C15 | 1.532 (3) |
| C4—H4B | 0.9700 | C14—H14 | 0.9800 |
| C4—H4C | 0.9700 | C15—C16 | 1.515 (3) |
| C5—C6 | 1.525 (3) | C15—H15A | 0.9700 |
| C5—C10 | 1.551 (2) | C15—H15B | 0.9700 |
| C5—H5A | 0.9800 | C16—C17 | 1.512 (4) |
| C6—C7 | 1.523 (3) | C17—H17A | 0.9700 |
| C6—H6A | 0.9700 | C17—H17B | 0.9700 |
| C6—H6B | 0.9700 | C18—H18A | 0.9600 |
| C7—C8 | 1.528 (2) | C18—H18B | 0.9600 |
| C7—H7A | 0.9700 | C18—H18C | 0.9600 |
| C7—H7B | 0.9700 | C19—H19A | 0.9700 |
| C8—C14 | 1.524 (2) | C19—H19B | 0.9700 |
| C8—C9 | 1.542 (3) | | |
| | | | |
| C19—O1—C2 | 109.44 (13) | C1—C10—C9 | 113.66 (15) |
| C2—O2—H2A | 109.5 | C1—C10—C19 | 100.18 (14) |
| C3—O3—H3A | 109.5 | C9—C10—C19 | 115.84 (14) |

| | | | |
|---|---|---|---|
| C2—C1—C10 | 101.51 (15) | C1—C10—C5 | 107.06 (15) |
| C2—C1—H1A | 111.5 | C9—C10—C5 | 109.94 (13) |
| C10—C1—H1A | 111.5 | C19—C10—C5 | 109.50 (15) |
| C2—C1—H1B | 111.5 | C12—C11—C9 | 113.59 (15) |
| C10—C1—H1B | 111.5 | C12—C11—H11A | 108.8 |
| H1A—C1—H1B | 109.3 | C9—C11—H11A | 108.8 |
| O2—C2—O1 | 108.94 (16) | C12—C11—H11B | 108.8 |
| O2—C2—C1 | 111.31 (17) | C9—C11—H11B | 108.8 |
| O1—C2—C1 | 103.11 (15) | H11A—C11—H11B | 107.7 |
| O2—C2—C3 | 114.50 (17) | C13—C12—C11 | 110.12 (16) |
| O1—C2—C3 | 107.03 (17) | C13—C12—H12A | 109.6 |
| C1—C2—C3 | 111.22 (16) | C11—C12—H12A | 109.6 |
| O3—C3—C4 | 111.86 (18) | C13—C12—H12B | 109.6 |
| O3—C3—C2 | 106.94 (18) | C11—C12—H12B | 109.6 |
| C4—C3—C2 | 109.48 (16) | H12A—C12—H12B | 108.2 |
| O3—C3—H3B | 109.5 | C12—C13—C18 | 110.98 (17) |
| C4—C3—H3B | 109.5 | C12—C13—C17 | 115.81 (17) |
| C2—C3—H3B | 109.5 | C18—C13—C17 | 107.28 (17) |
| C3—C4—C5 | 114.94 (17) | C12—C13—C14 | 108.43 (16) |
| C3—C4—H4B | 108.5 | C18—C13—C14 | 113.29 (16) |
| C5—C4—H4B | 108.5 | C17—C13—C14 | 100.79 (15) |
| C3—C4—H4C | 108.5 | C8—C14—C15 | 119.80 (17) |
| C5—C4—H4C | 108.5 | C8—C14—C13 | 113.53 (13) |
| H4B—C4—H4C | 107.5 | C15—C14—C13 | 103.60 (15) |
| C6—C5—C4 | 109.85 (17) | C8—C14—H14 | 106.3 |
| C6—C5—C10 | 112.35 (16) | C15—C14—H14 | 106.3 |
| C4—C5—C10 | 110.84 (14) | C13—C14—H14 | 106.3 |
| C6—C5—H5A | 107.9 | C16—C15—C14 | 103.53 (19) |
| C4—C5—H5A | 107.9 | C16—C15—H15A | 111.1 |
| C10—C5—H5A | 107.9 | C14—C15—H15A | 111.1 |
| C7—C6—C5 | 112.59 (18) | C16—C15—H15B | 111.1 |
| C7—C6—H6A | 109.1 | C14—C15—H15B | 111.1 |
| C5—C6—H6A | 109.1 | H15A—C15—H15B | 109.0 |
| C7—C6—H6B | 109.1 | O4—C16—C17 | 125.6 (2) |
| C5—C6—H6B | 109.1 | O4—C16—C15 | 125.4 (3) |
| H6A—C6—H6B | 107.8 | C17—C16—C15 | 108.98 (18) |
| C6—C7—C8 | 111.69 (15) | C16—C17—C13 | 102.17 (17) |
| C6—C7—H7A | 109.3 | C16—C17—H17A | 111.3 |
| C8—C7—H7A | 109.3 | C13—C17—H17A | 111.3 |
| C6—C7—H7B | 109.3 | C16—C17—H17B | 111.3 |
| C8—C7—H7B | 109.3 | C13—C17—H17B | 111.3 |
| H7A—C7—H7B | 107.9 | H17A—C17—H17B | 109.2 |
| C14—C8—C7 | 111.37 (14) | C13—C18—H18A | 109.5 |
| C14—C8—C9 | 108.93 (15) | C13—C18—H18B | 109.5 |
| C7—C8—C9 | 109.80 (16) | H18A—C18—H18B | 109.5 |
| C14—C8—H8A | 108.9 | C13—C18—H18C | 109.5 |
| C7—C8—H8A | 108.9 | H18A—C18—H18C | 109.5 |
| C9—C8—H8A | 108.9 | H18B—C18—H18C | 109.5 |

| | | | |
|---|---|---|---|
| C8—C9—C11 | 112.89 (15) | O1—C19—C10 | 105.95 (16) |
| C8—C9—C10 | 111.57 (15) | O1—C19—H19A | 110.5 |
| C11—C9—C10 | 113.97 (14) | C10—C19—H19A | 110.5 |
| C8—C9—H9 | 105.9 | O1—C19—H19B | 110.5 |
| C11—C9—H9 | 105.9 | C10—C19—H19B | 110.5 |
| C10—C9—H9 | 105.9 | H19A—C19—H19B | 108.7 |
| | | | |
| C19—O1—C2—O2 | −142.16 (15) | C4—C5—C10—C1 | −60.1 (2) |
| C19—O1—C2—C1 | −23.84 (19) | C6—C5—C10—C9 | 52.7 (2) |
| C19—O1—C2—C3 | 93.54 (17) | C4—C5—C10—C9 | 176.02 (17) |
| C10—C1—C2—O2 | 158.11 (16) | C6—C5—C10—C19 | −75.65 (19) |
| C10—C1—C2—O1 | 41.46 (17) | C4—C5—C10—C19 | 47.7 (2) |
| C10—C1—C2—C3 | −72.9 (2) | C8—C9—C11—C12 | 50.3 (2) |
| O2—C2—C3—O3 | 64.1 (2) | C10—C9—C11—C12 | 178.96 (17) |
| O1—C2—C3—O3 | −175.03 (16) | C9—C11—C12—C13 | −54.3 (2) |
| C1—C2—C3—O3 | −63.1 (2) | C11—C12—C13—C18 | −67.1 (2) |
| O2—C2—C3—C4 | −174.50 (18) | C11—C12—C13—C17 | 170.30 (18) |
| O1—C2—C3—C4 | −53.7 (2) | C11—C12—C13—C14 | 57.9 (2) |
| C1—C2—C3—C4 | 58.3 (2) | C7—C8—C14—C15 | −59.0 (2) |
| O3—C3—C4—C5 | 75.6 (2) | C9—C8—C14—C15 | 179.72 (16) |
| C2—C3—C4—C5 | −42.7 (2) | C7—C8—C14—C13 | 177.94 (17) |
| C3—C4—C5—C6 | 169.86 (18) | C9—C8—C14—C13 | 56.7 (2) |
| C3—C4—C5—C10 | 45.1 (2) | C12—C13—C14—C8 | −61.8 (2) |
| C4—C5—C6—C7 | −176.24 (17) | C18—C13—C14—C8 | 61.8 (2) |
| C10—C5—C6—C7 | −52.4 (2) | C17—C13—C14—C8 | 176.12 (17) |
| C5—C6—C7—C8 | 54.4 (3) | C12—C13—C14—C15 | 166.64 (15) |
| C6—C7—C8—C14 | −177.36 (19) | C18—C13—C14—C15 | −69.71 (19) |
| C6—C7—C8—C9 | −56.6 (2) | C17—C13—C14—C15 | 44.6 (2) |
| C14—C8—C9—C11 | −49.58 (18) | C8—C14—C15—C16 | −156.82 (17) |
| C7—C8—C9—C11 | −171.78 (16) | C13—C14—C15—C16 | −29.09 (19) |
| C14—C8—C9—C10 | −179.45 (14) | C14—C15—C16—O4 | −179.7 (2) |
| C7—C8—C9—C10 | 58.35 (18) | C14—C15—C16—C17 | 2.5 (2) |
| C2—C1—C10—C9 | −166.58 (15) | O4—C16—C17—C13 | −152.8 (2) |
| C2—C1—C10—C19 | −42.37 (17) | C15—C16—C17—C13 | 25.0 (2) |
| C2—C1—C10—C5 | 71.82 (17) | C12—C13—C17—C16 | −158.74 (18) |
| C8—C9—C10—C1 | −176.07 (14) | C18—C13—C17—C16 | 76.7 (2) |
| C11—C9—C10—C1 | 54.6 (2) | C14—C13—C17—C16 | −42.0 (2) |
| C8—C9—C10—C19 | 68.68 (19) | C2—O1—C19—C10 | −3.4 (2) |
| C11—C9—C10—C19 | −60.6 (2) | C1—C10—C19—O1 | 28.71 (17) |
| C8—C9—C10—C5 | −56.09 (19) | C9—C10—C19—O1 | 151.40 (15) |
| C11—C9—C10—C5 | 174.61 (17) | C5—C10—C19—O1 | −83.62 (18) |
| C6—C5—C10—C1 | 176.60 (15) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|

| | | | | |
|---|---|---|---|---|
| O2—H2*A*···O1$^{i}$ | 0.82 | 2.03 | 2.8088 (19) | 158 |

Symmetry code: (i) −*x*, *y*+1/2, −*z*+1.