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

# 1,6,6-Trimethyl-6,7,8,9-tetrahydrophenanthro[1,2-*b*]furan-10,11-dione

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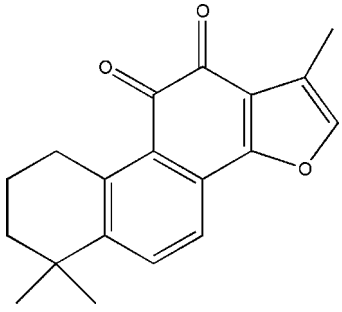
Received 23 April 2007; accepted 30 April 2007

Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.057; $wR$ factor = 0.139; data-to-parameter ratio = 10.1.

The title compound, $C_{19}H_{18}O_3$, also known as tanshinone IIA, contains a four-ring system. The crystal packing involves $\pi$–$\pi$ stacking interactions of 3.279 Å and C—H···O hydrogen bonds. The molecule lies on a crystallographic mirror plane, which involves disorder of the cyclohexene ring across this plane.

## Related literature

For related literature, see: Chang *et al.* (1991); Ryu *et al.* (1997); Xue *et al.* (1999).



## Experimental

### *Crystal data*

$C_{19}H_{18}O_3$
$M_r$ = 294.33
Orthorhombic, *Pmna*
$a$ = 6.5579 (11) Å
$b$ = 9.1687 (15) Å
$c$ = 24.328 (4) Å
$V$ = 1462.8 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 113 (2) K
0.24 × 0.20 × 0.10 mm

### *Data collection*

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)
$T_{min}$ = 0.968, $T_{max}$ = 0.991
10602 measured reflections
1404 independent reflections
1343 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.044

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.139
$S$ = 1.17
1404 reflections
139 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2283).

## References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chang, H. M., Chui, K. Y., Tan, F. W. L., Yang, Y., Zhong, Z. P., Lee, C. M., Sham, H. L. & Wang, H. N. C. (1991). *J. Med. Chem.* **34**, 1675–1692.
Ryu, S. Y., Lee, C. O. & Choi, A. U. (1997). *Planta Med.* **63**, 339–342.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xue, M., Cui, Y., Wang, H. Q., Hu, H. Y. & Zhang, B. (1999). *J. Pharm. Biomed. Anal.* **21**, 207–213.

 doi:10.1107/S1600536807021320  

# supplementary materials

*Acta Cryst.* (2007). E**63**, o2831 [ **doi:10.1107/S1600536807021320** ]

# 1,6,6-Trimethyl-6,7,8,9-tetrahydrophenanthro[1,2-*b*]furan-10,11-dione

**X.-Q. Liu and W.-Y. Gao**

## Comment

Tanshinone IIA is one of the major active constitutes isolated from the traditional Chinese medicinal herb, Salvia miltiorrhiza Bunge, which has been widely used in China to treat coronary heart diseases (Chang *et al.*, 1991), antitumour (Ryu *et al.*, 1997), angina pectoris and myocardial infarction (Xue *et al.*, 1999). Tanshinone IIA is also most effective and has been used as a qualitycontroller for some medicine. We report here the crystal structure (I)(Fig. 1).

The crystal structure of (I) illustrated in Fig. 1 shows that the C12—C17 ring has a twisted conformation. Also, the C4—C9 are essentially coplanar with the aromatic rings. An intermolecular C—H···O hydrogen bond links the molecules into a chain extending along the b axis. No π-π interactions are observed.

## Experimental

Dried powder of Salvia miltiorrhiza Bunge was exacted with EtOH and the extract was concentrated in vacuo. The residue was subjected to silical-gel coloumn chromatography. Elution with petroleum ether-ethyl acetate (9:1v/v) yielded the title compound. The identity of (I) was confirmed by NMR spectroscopy. $^{1}$H NMR in $CDCl_3$ (400 MHz):1.30 (s, 6H), 1.65 (m, 2H), 1.78 (m, 2H), 2.25(d,1.3 Hz, 3H), 3.18 (t, 6 Hz, 2H), 7.21 (q,1.3 Hz,1*H*), 7.53 (d, AB, 8 Hz,1*H*), 7.62(d, AB, 8 Hz,1*H*). $^{13}$C NMR in $CDCl_3$ (100 MHz): 8.96, 19.33, 30.01, 32.03, 34.86, 38.07, 120.11, 120.43, 121.35, 126.71, 127.66, 133.64, 141.49, 144.66, 150.35, 161.91, 175.96, 183.83.

## Refinement

All H atoms were positioned geometrically and refined using a riding model, in the range of 0.95-0.98 Å , with $U_{iso}$(H) = 1.2$U_{eq}$(carrier) or $U_{iso}$(H) = 1.5$U_{eq}$(methyl C). The methylene groups C15 and C16, and five hydrogens H3a, H3b, H3c, H14a, H14b are found to be disordered. The molecular were located on mirror plane (the molecule has no such symmetry), so C15, C16 were disordered in two position. All the two site occupancies were refined to 0.5:0.5.

## Figures

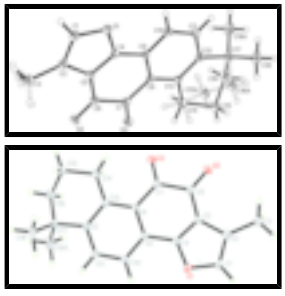


Fig. 1. A view of the molecular of (I). Displacement ellipsoids are drawn at the 30% probability level. One disorder component is shown for clarity.

### 1,6,6-Trimethyl-6,7,8,9-tetrahydrophenanthro[1,2-b]furan-10,11-dione

*Crystal data*

| | |
|---|---|
| $C_{19}H_{18}O_3$ | $D_x = 1.337$ Mg m$^{-3}$ |
| $M_r = 294.33$ | Melting point: 209.0-210.0 K |
| Orthorhombic, *Pmna* | Mo *K*α radiation<br>λ = 0.71070 Å |
| Hall symbol: -P 2ac 2 | Cell parameters from 2823 reflections |
| $a = 6.5579$ (11) Å | θ = 2.2–27.9º |
| $b = 9.1687$ (15) Å | μ = 0.09 mm$^{-1}$ |
| $c = 24.328$ (4) Å | $T = 113$ (2) K |
| $V = 1462.8$ (4) Å$^3$ | Prism, colourless |
| $Z = 4$ | 0.24 × 0.20 × 0.10 mm |
| $F_{000} = 624$ | |

*Data collection*

| | |
|---|---|
| Rigaku Saturn diffractometer | 1343 reflections with $I > 2\sigma(I)$ |
| Radiation source: rotating anode | $R_{int} = 0.044$ |
| Monochromator: confocal | $\theta_{max} = 25.0$º |
| $T = 113$(2) K | $\theta_{min} = 2.2$º |
| ω scans | $h = -7 \rightarrow 7$ |
| Absorption correction: multi-scan (SADABS; Sheldrick,1996) | $k = -10 \rightarrow 10$ |
| $T_{min} = 0.968$, $T_{max} = 0.991$ | $l = -28 \rightarrow 28$ |
| 10602 measured reflections | Standard reflections: ? |
| 1404 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.057$ | $w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.879P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.139$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $S = 1.17$ | $\Delta\rho_{max} = 0.22$ e Å$^{-3}$ |
| 1404 reflections | $\Delta\rho_{min} = -0.22$ e Å$^{-3}$ |
| 139 parameters | Extinction correction: none |
| 1 restraint | |
| Primary atom site location: structure-invariant direct methods | |
| Secondary atom site location: difference Fourier map | |

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.0000 | 0.8407 (3) | 0.29609 (10) | 0.0249 (6) | |
| H1 | 0.0000 | 0.9428 | 0.3029 | 0.030* | |
| C2 | 0.0000 | 0.7377 (3) | 0.33594 (11) | 0.0240 (6) | |
| C3 | 0.0000 | 0.7590 (3) | 0.39690 (11) | 0.0315 (7) | |
| H3A | 0.0243 | 0.8620 | 0.4053 | 0.047* | 0.50 |
| H3B | 0.1081 | 0.6995 | 0.4134 | 0.047* | 0.50 |
| H3C | −0.1323 | 0.7293 | 0.4119 | 0.047* | 0.50 |
| C4 | 0.0000 | 0.6329 (3) | 0.25212 (10) | 0.0194 (6) | |
| C5 | 0.0000 | 0.6018 (3) | 0.30665 (10) | 0.0200 (6) | |
| C6 | 0.0000 | 0.4508 (3) | 0.32377 (10) | 0.0210 (6) | |
| C7 | 0.0000 | 0.3346 (3) | 0.27671 (10) | 0.0202 (6) | |
| C8 | 0.0000 | 0.3812 (3) | 0.21762 (10) | 0.0189 (6) | |
| C9 | 0.0000 | 0.5325 (3) | 0.20633 (10) | 0.0184 (6) | |
| C10 | 0.0000 | 0.5814 (3) | 0.15243 (10) | 0.0221 (6) | |
| H10 | 0.0000 | 0.6830 | 0.1448 | 0.026* | |
| C11 | 0.0000 | 0.4819 (3) | 0.10997 (10) | 0.0243 (6) | |
| H11 | 0.0000 | 0.5166 | 0.0732 | 0.029* | |
| C12 | 0.0000 | 0.3313 (3) | 0.11940 (10) | 0.0221 (6) | |
| C13 | 0.0000 | 0.2793 (3) | 0.17363 (10) | 0.0195 (6) | |
| C14 | 0.0000 | 0.1179 (3) | 0.18488 (11) | 0.0353 (8) | |
| H14A | −0.1365 | 0.0861 | 0.1926 | 0.042* | 0.50 |
| H14B | 0.0825 | 0.0981 | 0.2166 | 0.042* | 0.50 |
| C15 | 0.0843 (7) | 0.0305 (4) | 0.13403 (15) | 0.0428 (12) | 0.50 |
| H15A | 0.0783 | −0.0727 | 0.1404 | 0.051* | 0.50 |
| H15B | 0.2232 | 0.0575 | 0.1268 | 0.051* | 0.50 |
| C16 | −0.0492 (11) | 0.0744 (4) | 0.08622 (15) | 0.049 (3) | 0.50 |
| H16A | −0.1896 | 0.0678 | 0.0971 | 0.059* | 0.50 |
| H16B | −0.0278 | 0.0091 | 0.0559 | 0.059* | 0.50 |
| C17 | 0.0000 | 0.2317 (3) | 0.06866 (11) | 0.0294 (7) | |
| C18 | 0.1883 (4) | 0.2607 (3) | 0.03377 (9) | 0.0509 (7) | |
| H18A | 0.1895 | 0.1939 | 0.0023 | 0.076* | |
| H18B | 0.3110 | 0.2451 | 0.0560 | 0.076* | |
| H18C | 0.1856 | 0.3616 | 0.0205 | 0.076* | |

| | | | | |
|---|---|---|---|---|
| O1 | 0.0000 | 0.77918 (18) | 0.24416 (7) | 0.0236 (5) |
| O2 | 0.0000 | 0.4085 (2) | 0.37113 (7) | 0.0291 (5) |
| O3 | 0.0000 | 0.2080 (2) | 0.29159 (7) | 0.0293 (5) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0282 (15) | 0.0191 (13) | 0.0275 (14) | 0.000 | 0.000 | −0.0064 (11) |
| C2 | 0.0228 (14) | 0.0236 (13) | 0.0257 (13) | 0.000 | 0.000 | −0.0040 (11) |
| C3 | 0.0406 (18) | 0.0298 (15) | 0.0241 (14) | 0.000 | 0.000 | −0.0053 (12) |
| C4 | 0.0178 (13) | 0.0180 (12) | 0.0224 (13) | 0.000 | 0.000 | 0.0002 (10) |
| C5 | 0.0171 (13) | 0.0208 (14) | 0.0220 (13) | 0.000 | 0.000 | −0.0002 (10) |
| C6 | 0.0180 (13) | 0.0215 (13) | 0.0235 (13) | 0.000 | 0.000 | −0.0014 (10) |
| C7 | 0.0162 (13) | 0.0202 (14) | 0.0240 (13) | 0.000 | 0.000 | 0.0018 (11) |
| C8 | 0.0165 (13) | 0.0189 (12) | 0.0215 (13) | 0.000 | 0.000 | 0.0013 (10) |
| C9 | 0.0146 (12) | 0.0209 (13) | 0.0198 (13) | 0.000 | 0.000 | −0.0010 (10) |
| C10 | 0.0226 (14) | 0.0214 (13) | 0.0222 (13) | 0.000 | 0.000 | 0.0030 (11) |
| C11 | 0.0290 (15) | 0.0244 (14) | 0.0195 (12) | 0.000 | 0.000 | 0.0008 (10) |
| C12 | 0.0219 (14) | 0.0233 (13) | 0.0212 (13) | 0.000 | 0.000 | −0.0028 (11) |
| C13 | 0.0177 (13) | 0.0181 (13) | 0.0228 (13) | 0.000 | 0.000 | −0.0011 (10) |
| C14 | 0.061 (2) | 0.0192 (14) | 0.0254 (14) | 0.000 | 0.000 | −0.0005 (11) |
| C15 | 0.076 (3) | 0.0224 (19) | 0.030 (2) | 0.0069 (19) | 0.001 (2) | −0.0050 (16) |
| C16 | 0.091 (9) | 0.0283 (18) | 0.0280 (18) | −0.015 (3) | −0.002 (2) | −0.0086 (15) |
| C17 | 0.0432 (18) | 0.0248 (14) | 0.0202 (13) | 0.000 | 0.000 | −0.0055 (11) |
| C18 | 0.0437 (14) | 0.0737 (17) | 0.0352 (12) | −0.0013 (13) | 0.0074 (10) | −0.0271 (12) |
| O1 | 0.0293 (11) | 0.0185 (9) | 0.0231 (9) | 0.000 | 0.000 | −0.0011 (7) |
| O2 | 0.0401 (12) | 0.0273 (11) | 0.0200 (9) | 0.000 | 0.000 | 0.0029 (8) |
| O3 | 0.0413 (12) | 0.0201 (10) | 0.0266 (10) | 0.000 | 0.000 | 0.0026 (8) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.354 (4) | C12—C13 | 1.403 (3) |
| C1—O1 | 1.384 (3) | C12—C17 | 1.535 (3) |
| C1—H1 | 0.9500 | C13—C14 | 1.504 (4) |
| C2—C5 | 1.435 (4) | C14—C15$^{i}$ | 1.574 (4) |
| C2—C3 | 1.496 (4) | C14—C15 | 1.575 (4) |
| C3—H3A | 0.9800 | C14—H14A | 0.9600 |
| C3—H3B | 0.9800 | C14—H14B | 0.9602 |
| C3—H3C | 0.9800 | C15—C15$^{i}$ | 1.106 (10) |
| C4—O1 | 1.355 (3) | C15—C16$^{i}$ | 1.252 (5) |
| C4—C5 | 1.357 (3) | C15—C16 | 1.511 (6) |
| C4—C9 | 1.445 (3) | C15—H15A | 0.9601 |
| C5—C6 | 1.445 (3) | C15—H15B | 0.9600 |
| C6—O2 | 1.216 (3) | C16—C16$^{i}$ | 0.646 (15) |
| C6—C7 | 1.564 (3) | C16—C15$^{i}$ | 1.252 (5) |
| C7—O3 | 1.216 (3) | C16—C17 | 1.539 (5) |
| C7—C8 | 1.500 (3) | C16—H16A | 0.9600 |
| C8—C9 | 1.414 (3) | C16—H16B | 0.9600 |

| | | | |
|---|---|---|---|
| C8—C13 | 1.421 (4) | C17—C18 | 1.522 (3) |
| C9—C10 | 1.386 (3) | C17—C18$^{i}$ | 1.522 (3) |
| C10—C11 | 1.378 (4) | C17—C16$^{i}$ | 1.539 (5) |
| C10—H10 | 0.9500 | C18—H18A | 0.9800 |
| C11—C12 | 1.399 (4) | C18—H18B | 0.9800 |
| C11—H11 | 0.9500 | C18—H18C | 0.9800 |
| C2—C1—O1 | 111.7 (2) | C15—C14—H14B | 109.7 |
| C2—C1—H1 | 124.2 | H14A—C14—H14B | 108.1 |
| O1—C1—H1 | 124.2 | C15$^{i}$—C15—C16$^{i}$ | 79.4 (4) |
| C1—C2—C5 | 104.5 (2) | C15$^{i}$—C15—C16 | 54.6 (3) |
| C1—C2—C3 | 128.2 (2) | C16$^{i}$—C15—C16 | 24.9 (6) |
| C5—C2—C3 | 127.3 (2) | C15$^{i}$—C15—C14 | 69.43 (17) |
| C2—C3—H3A | 109.5 | C16$^{i}$—C15—C14 | 120.1 (4) |
| C2—C3—H3B | 109.5 | C16—C15—C14 | 105.4 (3) |
| H3A—C3—H3B | 109.5 | C15$^{i}$—C15—H15A | 87.7 |
| C2—C3—H3C | 109.5 | C16$^{i}$—C15—H15A | 117.4 |
| H3A—C3—H3C | 109.5 | C16—C15—H15A | 111.3 |
| H3B—C3—H3C | 109.5 | C14—C15—H15A | 111.1 |
| O1—C4—C5 | 110.3 (2) | C15$^{i}$—C15—H15B | 161.6 |
| O1—C4—C9 | 121.4 (2) | C16$^{i}$—C15—H15B | 85.5 |
| C5—C4—C9 | 128.3 (2) | C16—C15—H15B | 109.9 |
| C4—C5—C2 | 107.6 (2) | C14—C15—H15B | 110.3 |
| C4—C5—C6 | 118.9 (2) | H15A—C15—H15B | 108.8 |
| C2—C5—C6 | 133.5 (2) | C16$^{i}$—C16—C15$^{i}$ | 100.6 (4) |
| O2—C6—C5 | 125.3 (2) | C16$^{i}$—C16—C15 | 54.6 (3) |
| O2—C6—C7 | 118.5 (2) | C15$^{i}$—C16—C15 | 46.0 (4) |
| C5—C6—C7 | 116.2 (2) | C16$^{i}$—C16—C17 | 77.9 (3) |
| O3—C7—C8 | 123.9 (2) | C15$^{i}$—C16—C17 | 126.7 (3) |
| O3—C7—C6 | 115.6 (2) | C15—C16—C17 | 110.0 (4) |
| C8—C7—C6 | 120.5 (2) | C16$^{i}$—C16—H16A | 163.5 |
| C9—C8—C13 | 119.9 (2) | C15$^{i}$—C16—H16A | 63.0 |
| C9—C8—C7 | 117.8 (2) | C15—C16—H16A | 109.1 |
| C13—C8—C7 | 122.3 (2) | C17—C16—H16A | 109.7 |
| C10—C9—C8 | 120.1 (2) | C16$^{i}$—C16—H16B | 81.6 |
| C10—C9—C4 | 121.5 (2) | C15$^{i}$—C16—H16B | 122.7 |
| C8—C9—C4 | 118.4 (2) | C15—C16—H16B | 109.9 |
| C11—C10—C9 | 119.7 (2) | C17—C16—H16B | 109.9 |
| C11—C10—H10 | 120.2 | H16A—C16—H16B | 108.3 |
| C9—C10—H10 | 120.2 | C18—C17—C18$^{i}$ | 108.5 (3) |
| C10—C11—C12 | 122.0 (2) | C18—C17—C12 | 110.17 (15) |
| C10—C11—H11 | 119.0 | C18$^{i}$—C17—C12 | 110.17 (15) |
| C12—C11—H11 | 119.0 | C18—C17—C16$^{i}$ | 98.5 (3) |
| C11—C12—C13 | 119.3 (2) | C18$^{i}$—C17—C16$^{i}$ | 119.2 (3) |
| C11—C12—C17 | 117.1 (2) | C12—C17—C16$^{i}$ | 109.5 (2) |

| | | | |
|---|---|---|---|
| C13—C12—C17 | 123.6 (2) | C18—C17—C16 | 119.2 (3) |
| C12—C13—C8 | 119.0 (2) | C18$^{i}$—C17—C16 | 98.5 (3) |
| C12—C13—C14 | 120.4 (2) | C12—C17—C16 | 109.5 (2) |
| C8—C13—C14 | 120.6 (2) | C16$^{i}$—C17—C16 | 24.2 (5) |
| C13—C14—C15$^{i}$ | 111.0 (2) | C17—C18—H18A | 109.5 |
| C13—C14—C15 | 111.0 (2) | C17—C18—H18B | 109.5 |
| C15$^{i}$—C14—C15 | 41.1 (3) | H18A—C18—H18B | 109.5 |
| C13—C14—H14A | 109.5 | C17—C18—H18C | 109.5 |
| C15$^{i}$—C14—H14A | 70.8 | H18A—C18—H18C | 109.5 |
| C15—C14—H14A | 109.1 | H18B—C18—H18C | 109.5 |
| C13—C14—H14B | 109.4 | C4—O1—C1 | 105.86 (19) |
| C15$^{i}$—C14—H14B | 137.2 | | |
| O1—C1—C2—C5 | 0.0 | C9—C8—C13—C14 | 180.0 |
| O1—C1—C2—C3 | 180.000 (1) | C7—C8—C13—C14 | 0.0 |
| O1—C4—C5—C2 | 0.0 | C12—C13—C14—C15$^{i}$ | −22.10 (19) |
| C9—C4—C5—C2 | 180.0 | C8—C13—C14—C15$^{i}$ | 157.90 (19) |
| O1—C4—C5—C6 | 180.0 | C12—C13—C14—C15 | 22.10 (19) |
| C9—C4—C5—C6 | 0.000 (1) | C8—C13—C14—C15 | −157.90 (19) |
| C1—C2—C5—C4 | 0.0 | C13—C14—C15—C15$^{i}$ | −98.27 (12) |
| C3—C2—C5—C4 | 180.0 | C13—C14—C15—C16$^{i}$ | −34.7 (6) |
| C1—C2—C5—C6 | 180.000 (1) | C15$^{i}$—C14—C15—C16$^{i}$ | 63.6 (5) |
| C3—C2—C5—C6 | 0.000 (1) | C13—C14—C15—C16 | −56.5 (4) |
| C4—C5—C6—O2 | 180.0 | C15$^{i}$—C14—C15—C16 | 41.8 (4) |
| C2—C5—C6—O2 | 0.000 (1) | C15$^{i}$—C15—C16—C16$^{i}$ | 179.998 (2) |
| C4—C5—C6—C7 | 0.0 | C14—C15—C16—C16$^{i}$ | 130.1 (3) |
| C2—C5—C6—C7 | 180.0 | C16$^{i}$—C15—C16—C15$^{i}$ | 180.002 (1) |
| O2—C6—C7—O3 | 0.0 | C14—C15—C16—C15$^{i}$ | −49.9 (3) |
| C5—C6—C7—O3 | 180.0 | C15$^{i}$—C15—C16—C17 | 122.2 (4) |
| O2—C6—C7—C8 | 180.0 | C16$^{i}$—C15—C16—C17 | −57.8 (4) |
| C5—C6—C7—C8 | 0.0 | C14—C15—C16—C17 | 72.2 (4) |
| O3—C7—C8—C9 | 180.0 | C11—C12—C17—C18 | 59.82 (17) |
| C6—C7—C8—C9 | 0.0 | C13—C12—C17—C18 | −120.18 (17) |
| O3—C7—C8—C13 | 0.0 | C11—C12—C17—C18$^{i}$ | −59.82 (17) |
| C6—C7—C8—C13 | 180.0 | C13—C12—C17—C18$^{i}$ | 120.18 (17) |
| C13—C8—C9—C10 | 0.0 | C11—C12—C17—C16$^{i}$ | 167.1 (3) |
| C7—C8—C9—C10 | 180.0 | C13—C12—C17—C16$^{i}$ | −12.9 (3) |
| C13—C8—C9—C4 | 180.0 | C11—C12—C17—C16 | −167.1 (3) |
| C7—C8—C9—C4 | 0.0 | C13—C12—C17—C16 | 12.9 (3) |
| O1—C4—C9—C10 | 0.0 | C16$^{i}$—C16—C17—C18 | 33.80 (18) |
| C5—C4—C9—C10 | 180.0 | C15$^{i}$—C16—C17—C18 | 128.1 (6) |
| O1—C4—C9—C8 | 180.0 | C15—C16—C17—C18 | 78.6 (4) |
| C5—C4—C9—C8 | 0.0 | C16$^{i}$—C16—C17—C18$^{i}$ | 150.60 (15) |
| C8—C9—C10—C11 | 0.0 | C15$^{i}$—C16—C17—C18$^{i}$ | −115.1 (7) |

| | | | |
|---|---|---|---|
| C4—C9—C10—C11 | 180.0 | C15—C16—C17—C18$^{i}$ | −164.6 (3) |
| C9—C10—C11—C12 | 0.0 | C16$^{i}$—C16—C17—C12 | −94.37 (11) |
| C10—C11—C12—C13 | 0.0 | C15$^{i}$—C16—C17—C12 | −0.1 (8) |
| C10—C11—C12—C17 | 180.0 | C15—C16—C17—C12 | −49.5 (4) |
| C11—C12—C13—C8 | 0.0 | C15$^{i}$—C16—C17—C16$^{i}$ | 94.3 (7) |
| C17—C12—C13—C8 | 180.0 | C15—C16—C17—C16$^{i}$ | 44.8 (4) |
| C11—C12—C13—C14 | 180.0 | C5—C4—O1—C1 | 0.0 |
| C17—C12—C13—C14 | 0.0 | C9—C4—O1—C1 | 180.0 |
| C9—C8—C13—C12 | 0.0 | C2—C1—O1—C4 | 0.0 |
| C7—C8—C13—C12 | 180.0 | | |

Symmetry codes: (i) −*x*, *y*, *z*.

**Fig. 1**

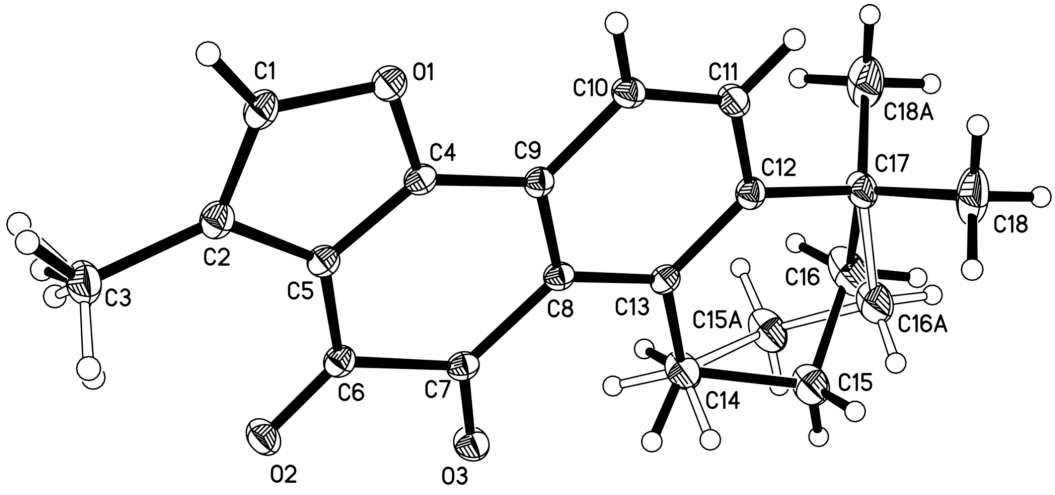
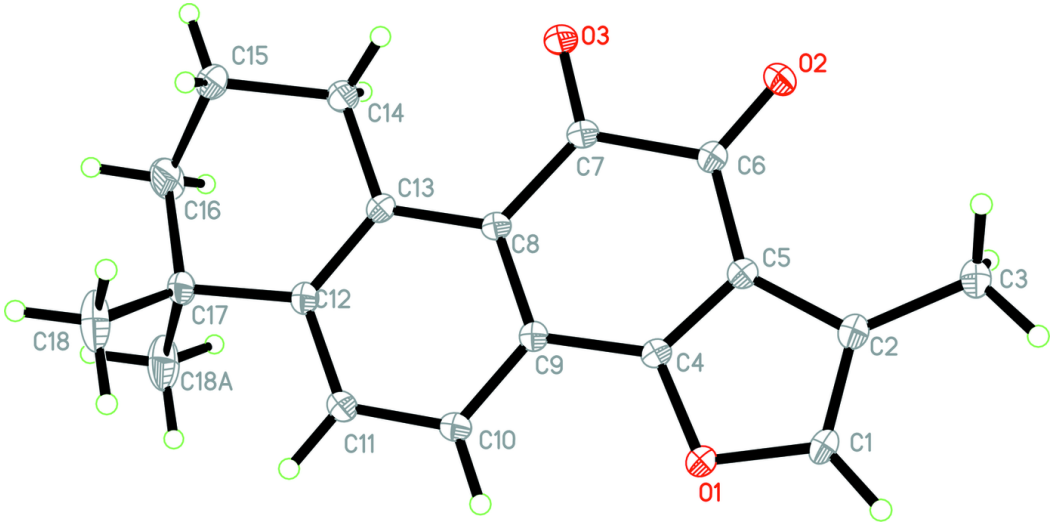

C1
O1
C10
C11
C18A
C4
C9
C12
C17
C18
C2
C16
C3
C5
C8
C13
C15A
C16A
C6
C7
C14
C15
O2
O3

**Fig. 2**


C15
C14
O3
O2
C7
C6
C13
C16
C8
C5
C3
C12
C17
C18
C18A
C9
C4
C2
C11
C10
C1
O1