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β-Zearalenol Sesquihydrate†

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Abstract

This X-ray diffraction study establishes the molecular structure of the title compound, 3,4,5,6,7,8,9,10-octahydro-7,14,16-trihydroxy-3-methyl-1*H*-2-benzoxacyclotetradecen-1-one sesquihydrate, $C_{18}H_{24}O_5.1.5H_2O$. There are two independent molecules in the asymmetric unit. The molecule consists of a 14-membered lactone ring fused *ortho* to a 1,3-dihydroxybenzene moiety. The crystal structure is stabilized by O—H···O hydrogen bonds. There are three water molecules filling the unit cell and forming a three-dimensional network of hydrogen bonds.

Comment

Zearalenone (Urry, Wehrmeister, Hodge & Hidy, 1966) is a common strogenic secondary metabolite produced by several species of *Fusarium*. Among a number of derivatives of zearalenone isolated from *F. roseum* (Stipanovic & Schroeder, 1975), zearalenol was found to be four times more active than zearalenone in rat uterotropic assay (Pathre & Mirocha, 1976). The present work describes the structure determination and crystal packing of the title compound.

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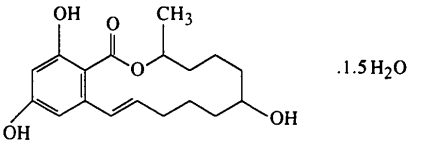

Bond distances and angles are in agreement with the related compound α-zearalenol, reported by Watson, Zabel, Mirocha & Pathre (1982). There are two independent molecules in the asymmetric unit (denoted *A* and *B*) with almost the same conformation. This molecule consists of a phenyl ring (C16–C21) fused to a 14-membered lactone ring (C1–C10, O11, C12, C16, C21). The average C—C bond lengths of the phenyl ring are 1.395 (4) and 1.392 (4) Å for *A* and *B*, respectively. In both molecules, the phenyl ring is planar to within 0.009 (2) Å. The *trans* C1=C2 double bond is twisted out of the plane of the phenyl ring as indicated by the C2—C1—C21—C16 torsion angle [155.8 (3) and 156.7 (3)° for *A* and *B*, respectively] and the C1—C21 distance [1.483 (4) and 1.484 (4) Å for *A* and *B*, respectively], indicating some degree of conjugation. The configuration at C10 is known to be *S* with reference to zearalenone (Kuo *et al.*, 1967; Taub *et al.*, 1968). The configuration at C6 is *S*, confirming the title compound to be β-zearalenol. The torsion angles (see Table 2) for both molecules indicate similar conformation between them and those in α-zearalenol, except that the latter has an *R* configuration at C6.

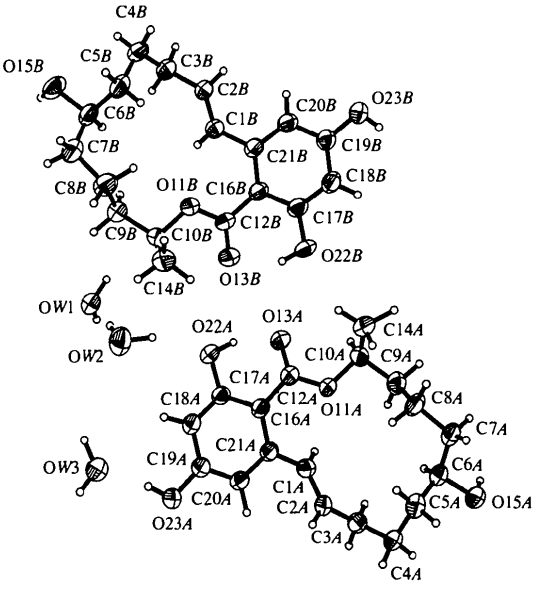

Fig. 1. The molecular structure of the title compound with the atom labelling; 50% probability displacement ellipsoids are shown.

The O—H···O hydrogen bonds are given in Table 3. The molecule contains three hydroxy groups. In both molecules the hydroxy group at C17 interacts with the carbonyl O atom located at C12, forming a strong

intramolecular O—H···O hydrogen bond at distances of 2.592 (3) and 2.580 (4) Å for *A* and *B*, respectively, and forming a six-membered ring which is planar to within 0.08 (2) Å. The O23 hydroxy group participates in an intermolecular O—H···O hydrogen bond with the water atom *OW*3 and a symmetry-related *OW*1 atom ($x, y, z-1$) at distances 2.632 (4) and 2.767 (3) Å in *A* and *B*, respectively. While the hydroxy O15 atom of *A* participates in an O—H···O hydrogen bond with the *OW*2 water atom, O15 of *B* is involved in a hydrogen bond with the symmetry-related O15 atom of *A*. All three water molecules participate in several hydrogen bonds stabilizing the zearalenol molecules in the crystal. Table 3 gives the geometry of these hydrogen bonds.

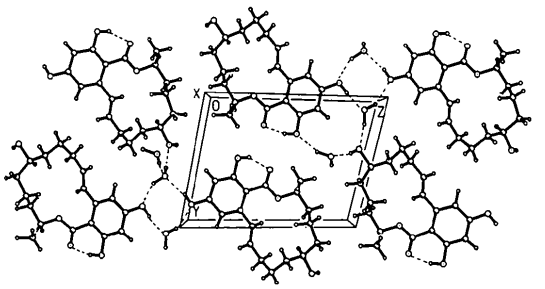

Fig. 2. A unit-cell drawing of the packing arrangement viewed along (011); dashed lines indicate O—H···O hydrogen bonds.

Experimental

The title compound was purchased from Sigma Chemical Company and recrystallized from EtOAc/water (1:1) solution by slow evaporation of the solvent.

Crystal data

$C_{18}H_{24}O_5.1.5H_2O$
M_r = 347.40
Triclinic
$P\bar{1}$
a = 8.398 (2) Å
b = 9.858 (2) Å
c = 12.781 (3) Å
α = 94.89 (1)°
β = 103.94 (2)°
γ = 113.36 (2)°
V = 923.2 (4) Å^3
Z = 2
D_x = 1.250 Mg m^{-3}
D_m = 1.249 Mg m^{-3}
D_m measured by flotation in benzene/chloroform solution

Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 20–50°
μ = 0.783 mm^{-1}
T = 293 (2) K
Rectangular
0.18 × 0.15 × 0.14 mm
Colourless

Data collection

*P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none

θ_{max} = 60.00°
$h = 0 \rightarrow 9$
$k = -10 \rightarrow 9$
$l = -14 \rightarrow 13$
2930 measured reflections
2930 independent reflections
2804 observed reflections [$I > 2\sigma(I)$]
3 standard reflections monitored every 100 reflections
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0342
$wR(F^2)$ = 0.0910
S = 1.068
2930 reflections
658 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0670P)^2 + 0.0331P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.021
$\Delta\rho_{max}$ = 0.125 e Å^{-3}
$\Delta\rho_{min}$ = −0.149 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C1*A*	−0.0661 (4)	−0.2583 (3)	0.3519 (2)	0.0427 (7)
C2*A*	−0.0918 (5)	−0.4008 (4)	0.3314 (2)	0.0466 (7)
C3*A*	−0.2494 (5)	−0.5223 (4)	0.2437 (3)	0.0512 (8)
C4*A*	−0.1945 (6)	−0.6159 (4)	0.1706 (3)	0.0581 (9)
C5*A*	−0.0808 (5)	−0.5231 (4)	0.1028 (3)	0.0557 (8)
C6*A*	−0.1820 (5)	−0.4762 (3)	0.0104 (2)	0.0479 (7)
C7*A*	−0.0599 (6)	−0.3513 (4)	−0.0343 (3)	0.0592 (9)
C8*A*	0.0474 (5)	−0.1970 (4)	0.0443 (3)	0.0549 (8)
C9*A*	−0.0663 (6)	−0.1142 (4)	0.0599 (3)	0.0565 (9)
C10*A*	0.0420 (6)	0.0333 (4)	0.1458 (3)	0.0554 (9)
O11*A*	0.0701 (3)	−0.0176 (2)	0.2511 (2)	0.0485 (5)
C12*A*	0.1122 (5)	0.0779 (3)	0.3433 (2)	0.0445 (7)
O13*A*	0.1281 (4)	0.2072 (2)	0.3447 (2)	0.0614 (7)
C14*A*	0.2227 (8)	0.1338 (4)	0.1329 (4)	0.0760 (13)
O15*A*	−0.2875 (4)	−0.6098 (3)	−0.0778 (2)	0.0559 (6)
C16*A*	0.1445 (4)	0.0170 (3)	0.4440 (2)	0.0409 (6)
C17*A*	0.2595 (4)	0.1221 (3)	0.5425 (2)	0.0398 (6)
C18*A*	0.3130 (4)	0.0760 (3)	0.6383 (3)	0.0441 (7)
C19*A*	0.2504 (4)	−0.0765 (3)	0.6384 (2)	0.0420 (7)
C20*A*	0.1339 (4)	−0.1828 (3)	0.5431 (2)	0.0411 (7)
C21*A*	0.0768 (4)	−0.1398 (3)	0.4456 (2)	0.0399 (6)
O22*A*	0.3246 (3)	0.2743 (2)	0.5486 (2)	0.0496 (6)
O23*A*	0.2924 (4)	−0.1277 (3)	0.7322 (2)	0.0542 (6)
C1*B*	0.5751 (5)	1.0180 (3)	0.4026 (3)	0.0429 (7)
C2*B*	0.6931 (5)	1.1587 (4)	0.4127 (3)	0.0492 (8)
C3*B*	0.7268 (6)	1.2894 (4)	0.4983 (3)	0.0534 (8)
C4*B*	0.9236 (6)	1.3699 (4)	0.5703 (3)	0.0576 (9)
C5*B*	0.9842 (5)	1.2722 (4)	0.6403 (3)	0.0501 (7)
C6*B*	0.9044 (5)	1.2382 (4)	0.7342 (3)	0.0509 (7)
C7*B*	0.9627 (6)	1.1331 (4)	0.7972 (3)	0.0598 (9)
C8*B*	0.8922 (5)	0.9734 (4)	0.7314 (3)	0.0575 (9)
C9*B*	0.6906 (5)	0.8805 (4)	0.7127 (3)	0.0501 (7)
C10*B*	0.6004 (5)	0.7386 (3)	0.6234 (2)	0.0455 (7)
O11*B*	0.5803 (3)	0.7897 (2)	0.5180 (2)	0.0468 (5)
C12*B*	0.4545 (4)	0.6892 (3)	0.4299 (3)	0.0460 (7)
O13*B*	0.3508 (4)	0.5636 (2)	0.4367 (2)	0.0618 (7)
C14*B*	0.7065 (6)	0.6444 (4)	0.6245 (3)	0.0588 (9)
O15*B*	0.9658 (4)	1.3805 (3)	0.8074 (2)	0.0664 (7)
C16*B*	0.4579 (4)	0.7393 (3)	0.3247 (3)	0.0421 (7)
C17*B*	0.3929 (5)	0.6247 (3)	0.2307 (3)	0.0470 (7)
C18*B*	0.4088 (5)	0.6577 (4)	0.1294 (3)	0.0496 (8)
C19*B*	0.4831 (4)	0.8062 (4)	0.1198 (2)	0.0462 (7)
C20*B*	0.5429 (5)	0.9215 (4)	0.2100 (3)	0.0453 (7)
C21*B*	0.5305 (4)	0.8913 (3)	0.3124 (2)	0.0412 (6)
O22*B*	0.3124 (4)	0.4756 (2)	0.2325 (2)	0.0628 (7)
O23*B*	0.4944 (4)	0.8457 (3)	0.0209 (2)	0.0573 (6)
*OW*1	0.2901 (4)	0.6251 (3)	0.8320 (2)	0.0584 (6)
*OW*2	0.4713 (4)	0.4522 (4)	0.7772 (2)	0.0705 (7)
*OW*3	0.5143 (9)	0.0816 (4)	0.9080 (3)	0.146 (3)

Table 2. *Geometric parameters (Å, °)*

C1*A*—C2*A*	1.327 (5)	C1*B*—C2*B*	1.320 (5)
C1*A*—C21*A*	1.483 (4)	C1*B*—C21*B*	1.484 (4)
C2*A*—C3*A*	1.497 (4)	C2*B*—C3*B*	1.503 (4)
C3*A*—C4*A*	1.527 (6)	C3*B*—C4*B*	1.520 (6)
C4*A*—C5*A*	1.529 (5)	C4*B*—C5*B*	1.517 (6)
C5*A*—C6*A*	1.501 (5)	C5*B*—C6*B*	1.510 (5)
C6*A*—O15*A*	1.460 (3)	C6*B*—O15*B*	1.442 (4)
C6*A*—C7*A*	1.516 (5)	C6*B*—C7*B*	1.522 (5)
C7*A*—C8*A*	1.533 (4)	C7*B*—C8*B*	1.527 (5)
C8*A*—C9*A*	1.517 (5)	C8*B*—C9*B*	1.517 (5)
C9*A*—C10*A*	1.531 (5)	C9*B*—C10*B*	1.518 (4)
C10*A*—O11*A*	1.475 (4)	C10*B*—O11*B*	1.474 (3)
C10*A*—C14*A*	1.506 (7)	C10*B*—C14*B*	1.519 (5)
O11*A*—C12*A*	1.322 (4)	O11*B*—C12*B*	1.331 (4)
C12*A*—O13*A*	1.225 (4)	C12*B*—O13*B*	1.224 (4)
C12*A*—C16*A*	1.477 (4)	C12*B*—C16*B*	1.474 (5)
C16*A*—C17*A*	1.411 (4)	C16*B*—C17*B*	1.407 (4)
C16*A*—C21*A*	1.423 (4)	C16*B*—C21*B*	1.416 (4)
C17*A*—O22*A*	1.366 (4)	C17*B*—O22*B*	1.357 (4)
C17*A*—C18*A*	1.379 (5)	C17*B*—C18*B*	1.386 (5)
C18*A*—C19*A*	1.382 (4)	C18*B*—C19*B*	1.375 (5)
C19*A*—O23*A*	1.363 (4)	C19*B*—O23*B*	1.368 (4)
C19*A*—C20*A*	1.390 (4)	C19*B*—C20*B*	1.385 (5)
C20*A*—C21*A*	1.388 (4)	C20*B*—C21*B*	1.385 (5)

C21*A*—C1*A*—C2*A*—C3*A*	172.9 (3)
C1*A*—C2*A*—C3*A*—C4*A*	131.1 (4)
C2*A*—C3*A*—C4*A*—C5*A*	−64.6 (4)
C3*A*—C4*A*—C5*A*—C6*A*	−71.4 (4)
C4*A*—C5*A*—C6*A*—C7*A*	164.1 (3)
C5*A*—C6*A*—C7*A*—C8*A*	−67.3 (5)
C6*A*—C7*A*—C8*A*—C9*A*	−73.8 (5)
C7*A*—C8*A*—C9*A*—C10*A*	175.4 (3)
C8*A*—C9*A*—C10*A*—O11*A*	−71.9 (4)
C9*A*—C10*A*—O11*A*—C12*A*	−160.7 (3)
C10*A*—O11*A*—C12*A*—C16*A*	−177.6 (3)
O11*A*—C12*A*—C16*A*—C21*A*	−21.7 (4)
C12*A*—C16*A*—C21*A*—C1*A*	−14.0 (5)
C2*A*—C1*A*—C21*A*—C16*A*	155.8 (3)
C21*B*—C1*B*—C2*B*—C3*B*	174.5 (3)
C1*B*—C2*B*—C3*B*—C4*B*	122.0 (4)
C2*B*—C3*B*—C4*B*—C5*B*	−64.6 (4)
C3*B*—C4*B*—C5*B*—C6*B*	−71.1 (4)
C4*B*—C5*B*—C6*B*—C7*B*	177.0 (3)
C5*B*—C6*B*—C7*B*—C8*B*	−66.0 (4)
C6*B*—C7*B*—C8*B*—C9*B*	−72.9 (4)
C7*B*—C8*B*—C9*B*—C10*B*	163.9 (3)
C8*B*—C9*B*—C10*B*—O11*B*	−75.2 (4)
C9*B*—C10*B*—O11*B*—C12*B*	−158.3 (3)
C10*B*—O11*B*—C12*B*—C16*B*	−171.7 (3)
O11*B*—C12*B*—C16*B*—C21*B*	−23.6 (5)
C12*B*—C16*B*—C21*B*—C1*B*	−13.9 (5)
C2*B*—C1*B*—C21*B*—C16*B*	156.7 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O22*A*—H23*A*···O13*A*	0.94 (7)	1.69 (6)	2.592 (3)	159 (6)
O23*A*—H24*A*···O*W*3	0.86 (5)	1.77 (5)	2.632 (4)	171 (5)
O15*A*—H20*A*···O*W*2^{i}	0.94 (6)	1.79 (6)	2.698 (4)	162 (5)
O22*B*—H23*B*···O13*B*	0.89 (6)	1.79 (6)	2.580 (4)	147 (5)
O23*B*—H24*B*···O*W*1^{ii}	0.84 (4)	1.94 (4)	2.767 (3)	165 (3)
O15*B*—H20*B*···O15*A*^{iii}	0.86 (5)	2.03 (6)	2.886 (4)	172 (4)
O*W*1—H1O*W*···O23*A*^{iv}	1.02 (6)	1.90 (6)	2.840 (4)	151 (4)
O*W*1—H2O*W*···O15*B*^{v}	0.77 (7)	1.99 (7)	2.754 (4)	171 (7)
O*W*2—H3O*W*···O*W*1	0.90 (7)	1.96 (7)	2.842 (4)	168 (6)
O*W*2—H4O*W*···O22*A*	1.06 (7)	1.96 (7)	2.968 (4)	157 (5)
O*W*3—H5O*W*···O23*B*^{vi}	0.88 (7)	2.03 (6)	2.810 (4)	148 (5)
O*W*3—H6O*W*···O15*A*^{vii}	0.99 (10)	1.82 (10)	2.794 (4)	167 (9)

Symmetry codes: (i) $x-1, y-1, z-1$; (ii) $x, y, z-1$; (iii) $1+x, 2+y, 1+z$; (iv) $x, 1+y, z$; (v) $x-1, y-1, z$; (vi) $x, y-1, 1+z$; (vii) $1+x, 1+y, 1+z$.

Data collection: *P*4 (*XSCANS*; Siemens, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1204). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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17β-Estradiol 3-Benzoate†

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Abstract

In the title compound, 17β-hydroxyestra-1,3,5(10)-trien-3-yl benzoate, $C_{25}H_{28}O_3$, the *B*, *C* and *D* rings adopt envelope, chair and envelope conformations, respectively. Both phenyl rings are planar. The structure

† Contribution No. 1428 of the Instituto de Química, UNAM.