C11	0.43392 (15)	0.44285 (10)	0.09394 (10)	0.0245 (5)
C12	0.4525 (2)	0.37152 (10)	0.04472 (10)	0.0282 (5)
C13	0.3702 (2)	0.30256 (10)	0.04282 (10)	0.0286 (5)
C14	0.2649 (2)	0.30498 (10)	0.08777 (10)	0.0257 (5)
C15	0.19232 (14)	0.41037 (9)	0.27802 (10)	0.0221 (5)
C16	0.28290 (14)	0.47666 (9)	0.28591 (10)	0.0218 (5)
C17	0.34900 (15)	0.49961 (10)	0.36757 (10)	0.0248 (5)
C18	0.3235 (2)	0.45674 (10)	0.44180 (10)	0.0287 (5)
C19	0.2334 (2)	0.39171 (10)	0.43397 (10)	0.0288 (5)
C20	0.1674 (2)	0.36805 (10)	0.35188 (10)	0.0271 (5)
C21	0.03189 (15)	0.31977 (10)	0.17223 (10)	0.0259 (5)
C22	−0.1849 (2)	0.28788 (11)	0.18757 (11)	0.0320 (5)
C23	−0.2986 (2)	0.32389 (11)	0.22254 (11)	0.0344 (6)
C24	−0.3783 (2)	0.35232 (12)	0.35365 (13)	0.0396 (6)
C25	−0.3484 (2)	0.33713 (12)	0.44923 (13)	0.0447 (7)
C26	0.39102 (14)	0.59181 (9)	0.20536 (10)	0.0240 (5)
C27	0.6112 (2)	0.63049 (11)	0.24087 (11)	0.0326 (5)
C28	0.7454 (2)	0.59421 (11)	0.26214 (11)	0.0316 (5)
C29	0.8870 (2)	0.52072 (11)	0.37232 (12)	0.0352 (5)
C30	1.0073 (2)	0.57196 (11)	0.37320 (12)	0.0368 (6)

Table 2. *Selected geometric parameters (Å, °)*

F1—C25	1.393 (2)	O2—C24	1.419 (2)
F2—C30	1.403 (2)	O3—C26	1.421 (2)
O1—C21	1.421 (2)	O3—C27	1.416 (2)
O1—C22	1.419 (2)	O4—C28	1.417 (2)
O2—C23	1.416 (2)	O4—C29	1.425 (2)
C21—O1—C22	112.6 (1)	O2—C24—C25	108.1 (2)
C23—O2—C24	112.8 (1)	F1—C25—C24	110.1 (2)
C26—O3—C27	111.8 (1)	O3—C26—C2	110.1 (1)
C28—O4—C29	112.9 (1)	O3—C27—C28	109.1 (1)
O1—C21—C1	109.5 (1)	O4—C28—C27	109.6 (2)
O1—C22—C23	108.7 (1)	O4—C29—C30	113.9 (1)
O2—C23—C22	109.3 (2)	F2—C30—C29	110.0 (2)
O1—C22—C23—O2	−68.2 (2)	O3—C27—C28—O4	−70.0 (2)
O2—C24—C25—F1	−66.4 (2)	O4—C29—C30—F2	61.1 (2)

Anisotropic displacement parameters were used for all non-H atoms. All H atoms were placed in calculated positions, refined using a riding model and given isotropic displacement parameters equal to 1.2 times those of the atoms to which they are attached.

Data collection and cell refinement were performed using *CAD-4/PC* (Enraf–Nonius, 1993), and data reduction by *XCAD*4 (Harms, 1995). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990), and refined using *SHELXL*93 (Sheldrick, 1993). *SHELXTL* (Siemens, 1990) was used for molecular graphics, and *PLATON* (Spek, 1990) for preparation of the CIF and geometry analysis.

This research was sponsored by the Division of Chemical Sciences, Office of Basic Energy Sciences, US Department of Energy, under contract number DE-AC05-96OR22464 with Oak Ridge National Laboratory, managed by Lockheed Martin Energy Research Corp.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1273). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bakshi, P. K., Linden, A., Vincent, B. R., Roe, S. P., Adhikesavalu, D., Cameron, T. S. & Knop, O. (1994). *Can. J. Chem.* **72**, 1273–1293.

Enraf–Nonius (1993). *CAD-4/PC*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Gakh, A. A., Sachleben, R. A., Bryan, J. C. & Moyer, B. A. (1995). *Tetrahedron Lett.* **36**, 8163–8166.

Harms, K. (1995). *XCAD4. Program for the Lp-Correction of Enraf–Nonius Four-Circle Diffractometer Data.* Universität Marburg, Germany.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Siemens (1990). *SHELXTL* 5.03/*IRIX*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Acta Cryst. (1996). C**52**, 3095–3097

3,4,5,6,9,10-Hexahydro-14,16-dihydroxy-3-methyl-1*H*-2-benzoxacyclotetradecin-1,7(8*H*)-dione (Zearalenone)†

Kaliyamoorthy Panneerselvam, Enrique Rudiño-Piñera and Manuel Soriano-García*

Instituto de Química, Circuito Exterior, Ciudad Universitaria, Delegación Coyoacán, México DF 04510, México. E-mail: soriano@servidor.unam.mx

(*Received 18 March 1996; accepted 9 September 1996*)

Abstract

This X-ray diffraction study establishes the molecular structure of the title compound, $C_{18}H_{22}O_5$. The molecule consists of a 14-membered lactone ring fused to a benzene ring. The crystal structure is stabilized by O—H···O and C—H···O hydrogen bonds.

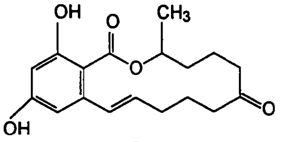
Comment

The compound zearalenone is a micotoxin produced by *Giberella zeare* when the fungus has the proper moisture and temperature conditions. This molecule is responsible for an estrogenic syndrome which attacks the genital system of male and female laboratory animals (Taylor & Watson, 1976). Related molecules have been used to produced curvularin macrolites (Ellestad, Lowell, Perkinson, Hargreaves & McGahren, 1978) and recently it has been recognized that related compounds have a potent activity against P388 leukemia (Agatsuma, Takahashi, Kabuto & Nozoe, 1993). The crystal and molecular structure of the title compound, (I), was first reported by Griffin, Duax, Strong & Mirocha (1981)

---

† Contribution No. 1468 of the Instituto de Química, UNAM.

without atomic coordinates. The present work describes in detail the structure determination and crystal packing of the title compound.



(I)

Bond distances and angles are in agreement with those of related structures reported by Taylor & Watson (1976) and Ellestad *et al.* (1978). This molecule consists of one phenyl (C16–C21) ring fused to a 14-membered lactone ring (C1–C10, O11, C12, C16, C21). The average phenyl C—C bond length is 1.391 (7) Å. The phenyl ring is planar within 0.02 Å. The configuration at the C10 atom is known to be *S* in zearalenone (Kuo *et al.*, 1967; Taub *et al.*, 1968).

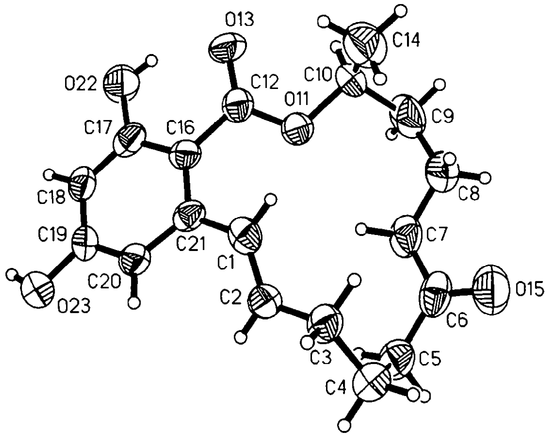


Fig. 1. The molecular structure of the title compound with the atom labelling and 50% probability displacement ellipsoids.

The hydrogen-bonding scheme is given in Table 3. The molecule contains two hydroxy groups. One of the hydroxy groups located at C17 interacts with the carbonyl-O atom located at C12 forming a strong intramolecular O—H···O hydrogen bond at a distance of 2.561 (7) Å and making a six-membered ring. This ring is planar within 0.07 Å. The second hydroxy group, O23, participates in an intermolecular O—H···O hydrogen bond with a symmetry-related O15 atom ($x + 1$, y, $z - 1$) at a distance of 2.732 (6) Å (Allen, Kennard & Taylor, 1983). There are four intramolecular C—H···O hydrogen bonds (2.652–3.184 Å) which internally stabilize the conformation of the molecule. In addition, there are four intermolecular C—H···O hydrogen bonds with C···O distances in the range 3.113–3.499 Å (Desiraju, 1991). In summary, the molecules in the crystal are stabilized by a network of O—H···O and C—H···O hydrogen bonds.

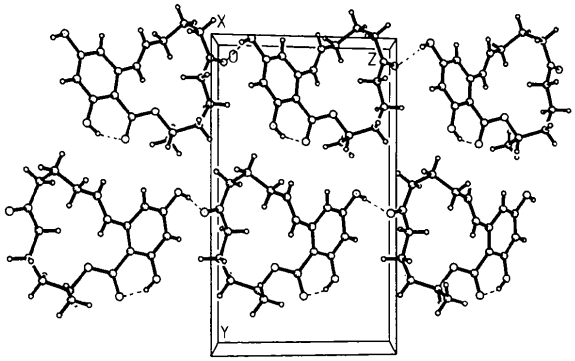


Fig. 2. A unit-cell drawing of the packing arrangement along (011), with dashed lines indicating O—H···O hydrogen bonds.

Experimental

The title compound was purchased from the Sigma Chemical Company and crystallized from chloroform by slow evaporation of the solvent.

Crystal data

$C_{18}H_{22}O_5$
$M_r = 318.36$
Monoclinic
$P2_1$
$a = 5.202\,(1)$ Å
$b = 16.329\,(5)$ Å
$c = 9.745\,(7)$ Å
$\beta = 98.33\,(4)°$
$V = 819.0\,(7)$ Å^3
$Z = 2$
$D_x = 1.291$ Mg m^{-3}
$D_m = 1.294$ Mg m^{-3}
D_m measured by flotation in benzene/bromoform solution

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 20$–$50°$
$\mu = 0.770$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.25 × 0.18 × 0.17 mm
Pale yellow

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1415 measured reflections
1262 independent reflections
969 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0424$

$\theta_{\rm max} = 60°$
$h = -5 \rightarrow 5$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 10$
3 standard reflections monitored every 100 reflections intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0503$
$wR(F^2) = 0.0852$
$S = 1.166$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.181$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.165$ e Å^{-3}
Extinction correction: none

1262 reflections
208 parameters
H atoms refined with a constant isotropic displacement parameter of 0.08 Å^2
$w = 1/[\sigma^2(F_o^2) + 0.3473P]$ where $P = (F_o^2 + 2F_c^2)/3$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.1 (5)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	$U_{\rm eq}$
C1	0.1243 (11)	0.0816 (3)	0.5484 (5)	0.0464 (15)
C2	0.0796 (12)	0.0075 (4)	0.5961 (5)	0.055 (2)
C3	−0.1241 (11)	−0.0134 (3)	0.6806 (5)	0.053 (2)
C4	−0.0303 (14)	−0.0690 (4)	0.8050 (6)	0.070 (2)
C5	0.1969 (14)	−0.0345 (4)	0.9039 (6)	0.067 (2)
C6	0.1432 (13)	0.0480 (4)	0.9597 (6)	0.060 (2)
C7	0.2865 (13)	0.1191 (4)	0.9184 (6)	0.062 (2)
C8	0.1946 (13)	0.2019 (4)	0.9612 (6)	0.070 (2)
C9	0.3337 (14)	0.2745 (4)	0.9063 (6)	0.075 (2)
C10	0.2457 (14)	0.3010 (4)	0.7555 (6)	0.060 (2)
O11	0.3054 (8)	0.2328 (2)	0.6636 (4)	0.0547 (11)
C12	0.3578 (11)	0.2533 (4)	0.5397 (5)	0.050 (2)
O13	0.3794 (10)	0.3253 (2)	0.5062 (4)	0.0741 (15)
C14	−0.0296 (14)	0.3226 (5)	0.7197 (7)	0.082 (2)
O15	−0.0294 (10)	0.0565 (3)	1.0312 (4)	0.0803 (14)
C16	0.4138 (11)	0.1843 (3)	0.4497 (5)	0.0440 (14)
C17	0.5791 (12)	0.2016 (3)	0.3501 (5)	0.053 (2)
C18	0.6627 (12)	0.1403 (4)	0.2706 (6)	0.052 (2)
C19	0.5738 (12)	0.0617 (4)	0.2842 (6)	0.050 (2)
C20	0.4012 (12)	0.0442 (4)	0.3716 (5)	0.052 (2)
C21	0.3172 (11)	0.1038 (3)	0.4602 (5)	0.0425 (14)
O22	0.6612 (10)	0.2783 (2)	0.3261 (5)	0.081 (2)
O23	0.6461 (9)	−0.0020 (2)	0.2051 (4)	0.0769 (14)

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

C1—C2	1.330 (8)	C10—O11	1.490 (6)
C1—C21	1.458 (7)	O11—C12	1.319 (6)
C2—C3	1.473 (8)	C12—O13	1.229 (7)
C3—C4	1.537 (7)	C12—C16	1.482 (7)
C4—C5	1.521 (8)	C16—C21	1.415 (7)
C5—C6	1.494 (9)	C16—C17	1.416 (7)
C6—O15	1.222 (7)	C17—O22	1.354 (6)
C6—C7	1.467 (9)	C17—C18	1.374 (8)
C7—C8	1.513 (8)	C18—C19	1.377 (8)
C8—C9	1.525 (9)	C19—C20	1.354 (8)
C9—C10	1.536 (8)	C19—O23	1.379 (7)
C10—C14	1.467 (8)	C20—C21	1.412 (7)
C2—C1—C21	127.1 (6)	O13—C12—C16	122.4 (5)
C1—C2—C3	125.6 (6)	O11—C12—C16	115.7 (5)
C2—C3—C4	113.9 (5)	C21—C16—C17	119.5 (5)
C5—C4—C3	114.2 (5)	C21—C16—C12	124.1 (5)
C6—C5—C4	113.3 (6)	C17—C16—C12	116.4 (5)
O15—C6—C7	120.7 (7)	O22—C17—C18	116.3 (5)
O15—C6—C5	120.1 (7)	O22—C17—C16	122.8 (5)
C7—C6—C5	119.0 (5)	C18—C17—C16	120.9 (5)
C6—C7—C8	116.0 (5)	C17—C18—C19	119.2 (6)
C7—C8—C9	114.4 (5)	C20—C19—C18	121.3 (6)
C8—C9—C10	117.6 (6)	C20—C19—O23	116.8 (6)
C14—C10—O11	108.6 (5)	C18—C19—O23	121.8 (5)
C14—C10—C9	116.2 (6)	C19—C20—C21	122.1 (6)
O11—C10—C9	107.7 (5)	C20—C21—C16	116.8 (5)
C12—O11—C10	116.7 (5)	C20—C21—C1	119.3 (5)
O13—C12—O11	121.6 (5)	C16—C21—C1	123.7 (5)

C21—C1—C2—C3	−177.4 (5)
C1—C2—C3—C4	−135.1 (6)
C2—C3—C4—C5	57.1 (7)
C3—C4—C5—C6	56.5 (7)
C4—C5—C6—C7	−112.5 (7)
C5—C6—C7—C8	171.2 (5)
C6—C7—C8—C9	−175.2 (6)
C7—C8—C9—C10	79.9 (8)
C8—C9—C10—O11	−64.7 (8)
C9—C10—O11—C12	−150.1 (5)
C10—O11—C12—C16	−179.4 (5)
O11—C12—C16—C21	26.7 (8)
C12—C16—C21—C1	8.3 (8)
C2—C1—C21—C16	−158.5 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O22—H22···O13	0.82	1.839	2.561 (7)	146.2
O23—H23···O15$^{\rm i}$	0.82	1.913	2.732 (6)	175.4
C1—H1···O11	0.93	2.394	2.817 (6)	107.5
C3—H31···O13$^{\rm ii}$	0.97	2.550	3.364 (7)	141.6
C4—H42···O22$^{\rm iii}$	0.97	2.600	3.499 (8)	154.2
C7—H71···O11	0.97	2.416	3.113 (7)	128.4
C10—H10···O13	0.98	2.302	2.652 (7)	99.9
C10—H10···O23$^{\rm iv}$	0.98	2.504	3.278 (7)	135.8
C14—H141···O13	0.96	2.728	3.184 (8)	109.8
C18—H18···O15$^{\rm i}$	0.93	2.629	3.311 (8)	130.7

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

Data collection: *P*4 diffractometer software. Cell refinement: *XSCANS* (Siemens, 1992). 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.

KP acknowledges the CONACYT (Cátedra Patrimonial Nivel II) for fellowship assistance. The authors thanks the Instituto de Biotecnología, UNAM, for data collection, and the DGAPA, UNAM (Project No. IN201395), for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1200). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Agatsuma, S., Takahashi, A., Kabuto, C. & Nozoe, S. (1993). *Chem. Pharm. Bull.* **41**, 373–375.

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.

Ellestad, G. A., Lowell, F. M., Perkinson, N. A., Hargreaves, R. T. & McGahren, W. J. (1978). *J. Org. Chem.* **43**, 2339–2343.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Griffin, J. F., Duax, W. L., Strong, P. D. & Mirocha, C. J. (1981). *Am. Cryst. Assoc. Ser.* 2, **9**, 35.

Kuo, C. H., Taub, D., Hoffsommer, R. D., Wendler, N. L., Urry, W. H. & Mullenbach, G. (1967). *Chem. Commun.* pp. 761–762.

Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1990*b*). *SHELXTL-Plus*. Release 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. University of Göttingen, Germany.

Siemens (1992). *XSCANS User's Manual*. Version 2.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Taub, D., Girotra, N. N., Hoffsommer, R. D., Kuo, C. H., Slates, H. L., Weber, S. & Wendler, N. L. (1968). *Tetrahedron*, **24**, 2443–2461.

Taylor, I. F. & Watson, W. H. (1976). *Acta Cryst.* B**32**, 710–714.