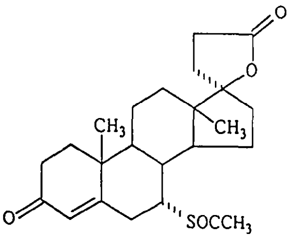
Sugawara, 1983). Atomic parameters are given in Table 1, bond lengths and angles in Table 2.* Fig. 1 shows the molecular structure (*ORTEP*II; Johnson, 1976) and atomic numbering of spironolactone. A *MOLDRAW* (Cense, 1988) stereoview of all the molecules as packed into the unit cell is shown in Fig. 2. The chemical formula is the following:



Related literature. Structure of the first crystalline modification of spironolactone (Dideberg & Dupont 1972). The main differences between the molecule of the present crystalline modification and that of the form described by Dideberg & Dupont lie in the conformations of the *A, D* and *E* rings. Polymorphism of spironolactone (El-dalsh, El-Sayed, Badawi, Khattab & Fouli, 1983; Salole & Al-Sarraj, 1985). Parameters of the *D*-ring configuration (Altona, Geise & Romers, 1968). *Atlas of Steriod Structures* (Duax & Norton, 1975).

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, C—H bond distances, intracyclic torsion angles, interatomic intermolecular distances and distances of atoms from least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51952 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Altona, C., Geise, H. J. & Romers, C. (1968). *Tetrahedron*, **24**, 13–32.

Busing, W. R., Martin, K. O. & Levy, H. A. (1964). *ORFFE*. Report ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, USA.

Cense, J.-M. (1988). *MOLDRAW*. Computer program for plotting molecular and crystal structures. Univ. of Paris-6, ENSCP, France.

Dideberg, O. & Dupont, L. (1972). *Acta Cryst.* **B28**, 3014–3022.

Duax, W. L. & Norton, D. A. (1975). *Atlas of Steroid Structures*, Vol. I. New York: IFI/Plenum.

El-dalsh, S. S., El-Sayed, A. A., Badawi, A. A., Khattab, F. I. & Fouli, A. (1983). *Drug Development Ind. Pharm.* **9**, 877–894.

Frenz, B. A. (1982). *Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Ito, T. & Sugawara, Y. (1983). *Best-Plane Program*. Third version, BP7C, The Institute of Physical & Chemical Research, Wako-Shi, Saitama 351, Japan.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M.M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Salole, E. G. & Al-Sarraj, F. A. (1985). *Drug Development Ind. Pharm.* **11**, 855–864.

Acta Cryst. (1989). C**45**, 1663–1665

Structure of the Simple Trichothecene 7α-Hydroxyscirpene

By Clifford George, Judith L. Flippen-Anderson and Richard Gilardi

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington DC 20375, *USA*

(*Received* 10 *April* 1989; *accepted* 6 *June* 1989)

Abstract. 12,13-Epoxytrichothec-9-en-7α-ol, $C_{15}H_{22}O_3$, $M_r = 250{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}522\,(2)$, $b = 12{\cdot}187\,(2)$, $c = 16{\cdot}647\,(2)$ Å, $V = 1323{\cdot}1\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}257$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 0{\cdot}652$ mm^{-1}, $F(000) = 544$, $T = 295$ K, final $R = 0{\cdot}034$, $wR = 0{\cdot}036$ for 1043 independent reflections. The title compound is produced by deoxygenation of 7α-hydroxytrichodermol which is produced as a minor metabolite of *Myrothecium roridum* along with much larger amounts of macrocyclic roridum and verrucarin trichothecenes. The X-ray structure established the hydroxyl group at the 7α rather than the 8β position. The conformation of the trichothecene ring system matches closely that observed in other simple trichothecenes as well as that observed in a number of macrocyclic trichothecenes. Weak intermolecular hydrogen bonding occurs with the hydroxyl oxygen acting as a donor to the ring oxygen in the six-membered ring.

Experimental. A clear colorless $0{\cdot}13 \times 0{\cdot}15 \times 0{\cdot}34$ mm data crystal was provided by Bruce Jarvis of the University of Maryland. Automated Nicolet *R*3*m* diffractometer with incident beam mono-

0108-2701/89/101663-03$03.00
© 1989 International Union of Crystallography

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$)

Equivalent isotropic U defined as one third of the trace of the orthoganalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	4350 (3)	7656 (2)	2009 (1)	48 (1)
O(2)	2608 (4)	6068 (2)	252 (1)	60 (1)
O(3)	2649 (4)	4374 (2)	2576 (2)	60 (1)
C(2)	3546 (5)	7535 (3)	1210 (2)	50 (1)
C(3)	1531 (7)	8140 (3)	1092 (3)	68 (1)
C(4)	−127 (6)	7336 (3)	1379 (3)	63 (1)
C(5)	994 (5)	6263 (2)	1621 (2)	45 (1)
C(6)	1780 (4)	6323 (2)	2520 (2)	42 (1)
C(7)	3411 (5)	5455 (2)	2732 (2)	43 (1)
C(8)	4107 (7)	5547 (3)	3597 (2)	54 (1)
C(9)	4586 (5)	6698 (3)	3857 (2)	53 (1)
C(10)	3996 (6)	7527 (3)	3409 (2)	55 (1)
C(11)	2863 (5)	7442 (2)	2640 (2)	44 (1)
C(12)	2850 (4)	6363 (2)	1087 (2)	44 (1)
C(13)	4082 (7)	5486 (3)	741 (2)	58 (1)
C(14)	−346 (7)	5268 (4)	1442 (3)	67 (2)
C(15)	−31 (6)	6256 (4)	3100 (3)	63 (1)
C(16)	5729 (8)	6827 (5)	4635 (2)	79 (2)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

O(1)—C(2)	1·437 (4)	O(1)—C(11)	1·453 (4)
O(2)—C(12)	1·445 (3)	O(2)—C(13)	1·446 (5)
O(3)—C(7)	1·431 (4)	C(2)—C(3)	1·520 (6)
C(2)—C(12)	1·513 (4)	C(3)—C(4)	1·536 (6)
C(4)—C(5)	1·552 (5)	C(5)—C(6)	1·584 (4)
C(5)—C(12)	1·506 (4)	C(5)—C(14)	1·525 (5)
C(6)—C(7)	1·541 (4)	C(6)—C(11)	1·548 (4)
C(6)—C(15)	1·527 (5)	C(7)—C(8)	1·515 (4)
C(8)—C(9)	1·500 (5)	C(9)—C(10)	1·314 (5)
C(9)—C(16)	1·502 (5)	C(10)—C(11)	1·482 (4)
C(12)—C(13)	1·456 (5)		

C(2)—O(1)—C(11)	114·0 (2)	C(12)—O(2)—C(13)	60·5 (2)
O(1)—C(2)—C(3)	112·7 (3)	O(1)—C(2)—C(12)	109·3 (2)
C(3)—C(2)—C(12)	100·4 (3)	C(2)—C(3)—C(4)	105·0 (3)
C(3)—C(4)—C(5)	106·7 (3)	C(4)—C(5)—C(6)	111·1 (3)
C(4)—C(5)—C(12)	99·1 (3)	C(6)—C(5)—C(12)	107·1 (2)
C(4)—C(5)—C(14)	110·4 (3)	C(6)—C(5)—C(14)	114·0 (3)
C(12)—C(5)—C(14)	114·2 (3)	C(5)—C(6)—C(7)	114·0 (2)
C(5)—C(6)—C(11)	108·0 (2)	C(7)—C(6)—C(15)	105·1 (2)
C(5)—C(6)—C(15)	110·1 (3)	C(7)—C(6)—C(15)	110·7 (3)
C(11)—C(6)—C(15)	108·6 (3)	O(3)—C(7)—C(6)	110·5 (2)
O(3)—C(7)—C(8)	110·2 (2)	C(6)—C(7)—C(8)	111·9 (3)
C(7)—C(8)—C(9)	114·0 (3)	C(8)—C(9)—C(10)	119·6 (3)
C(8)—C(9)—C(16)	116·7 (3)	C(10)—C(9)—C(16)	123·7 (4)
C(9)—C(10)—C(11)	125·6 (3)	O(1)—C(11)—C(6)	111·7 (2)
O(1)—C(11)—C(10)	106·2 (3)	C(6)—C(11)—C(10)	113·6 (2)
O(2)—C(12)—C(2)	113·5 (2)	O(2)—C(12)—C(5)	117·4 (2)
C(2)—C(12)—C(5)	103·7 (2)	O(2)—C(12)—C(13)	59·8 (2)
C(2)—C(12)—C(13)	125·5 (3)	C(5)—8C(12)—C(13)	128·1 (3)
O(2)—C(13)—C(12)	59·7 (2)		

chromator. 25 centered reflections within $27 \leq 2\theta \leq 78°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}56$ Å^{-1}, range of hkl: $0 \leq h \leq 7$, $0 \leq k \leq 13$, $0 \leq l \leq 18$. Standards 200, 060, 008, monitored every 60 reflections with random variation of 2·5% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]°$, scan rate a function of count rate (4·0° min^{-1} minimum, 30·0° min^{-1} maximum), 1189 reflections measured, 1169 unique, 1043 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization but not for absorption effects. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g.(F_o)^2]$, $g = 0{\cdot}00023$. Secondary-extinction parameter $p = 0{\cdot}016\,(2)$ in $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_o^2/\sin(2\theta)]^{0.25}$. There were 252 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms refined isotropically. $(\Delta/\sigma)_{\max} = 0{\cdot}01$, $R = 0{\cdot}034$, $wR = 0{\cdot}036$, $S = 1{\cdot}48$. Final difference Fourier excursions 0·15 and −0·13 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).† Atom numbering for Tables 1 and 2, atom coordinates, and bond distances and angles, follows that shown in Fig. 1. A packing diagram of the molecule is given in Fig. 2.

Related literature. Details on the preparation of 7α-hydroxyscirpene and 7α-hydroxytrichodermol are given by Jarivs *et al.* (1985). The structures of a number of compounds containing the trichothecene moiety have been reported including: T-2 toxin

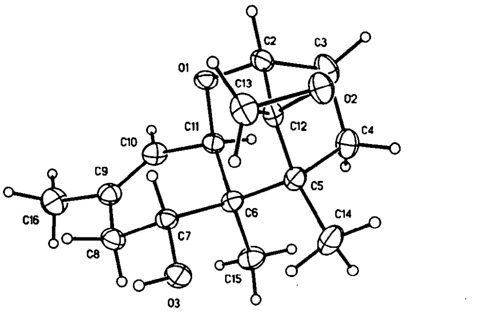


Fig. 1. Thermal-ellipsoid plot of 7α-hydroxyscirpene with ellipsoids drawn at the 20% probability level.

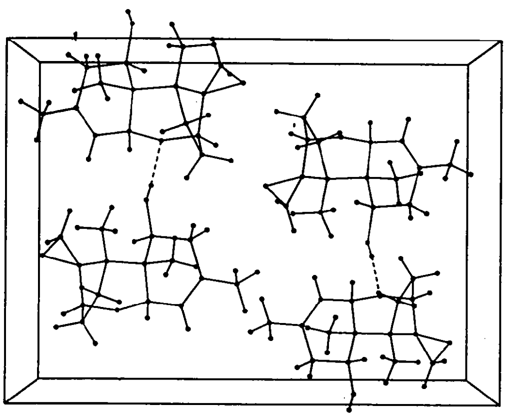


Fig. 2. Packing diagram of 7α-hydroxyscirpene viewed down the *a* axis.

---

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51985 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Gilardi, George & Flippen-Anderson, 1989), 3,15-diacetoxy-7,8-dihydroxy-12,13-epoxytrichothec-9-ene (Hanson, 1986), Myrotoxin A and C (Jarvis, Cömezoglu, Lee, Flippen-Anderson, Gilardi & George, 1986), Verrucarin A (McPhail & Sim, 1966), Verrucarin B (Breitenstein, Tamm, Arnold & Clardy, 1979) and α/β,8β,15-triacetoxy-12,13-epoxytricothec-9-en-3α-ol (Dillen, Gorst-Allman & Steyn, 1986).

This work was supported by USAMRIID contract #85MM5511.

References

Breitenstein, W., Tamm, C., Arnold, E. V. & Clardy, J. (1979). *Helv. Chim. Acta*, **62**, 2697–2703.

Dillen, J. L. M., Gorst-Allman, C. P. & Steyn, P. S. (1986). *S. Afr. J. Chem.* **39**, 111.

Gilardi, R., George, C. & Flippen-Anderson, J. L. (1989). *Acta Cryst.* Accepted.

Hanson, A. W. (1986). *Acta Cryst.* C**42**, 503–505.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Jarvis, B. B., Cömezoglu, F. T., Lee, Y. M., Flippen-Anderson, J. L., Gilardi, R. D. & George, C. F. (1986). *Bull. Soc. Chim. Belg.* **95**, 681–697.

Jarvis, B. B., Lee, Y. M., Yatawara, C. S., Mazzocchi, D. B., Flippen-Anderson, J. L., Gilardi, R. D. & George, C. F. (1985). *Appl. Environ. Microbiol.* **50**, 1225–1228.

McPhail, A. T. & Sim, G. A. (1966). *J. Chem. Soc. C*, pp. 1394–1396.

Sheldrick, G. M. (1980). *SHELXTL80. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1989). C**45**, 1665–1666

Structure of a 1,3-Diphosphetane Derivative

By Peter G. Jones

Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30,
3300 *Braunschweig, Federal Republic of Germany*

Gene B. Carpenter

Department of Chemistry, Brown University, Providence, RI 02912, *USA*

and George M. Sheldrick

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, 3400 *Göttingen,*
Federal Republic of Germany

(*Received* 28 *November* 1988; *accepted* 30 *May* 1989)

Abstract. 2,3,7,8-Tetramethyl-3,8-diaza-1,6-diphosphatricyclo[5.3.0.0^{2,6}]decane, $C_{10}H_{20}N_2P_2$, M_r = 230·23, monoclinic, $P2_1/c$, a = 6·852 (2), b = 13·683 (4), c = 6·844 (2) Å, β = 110·96 (3)°, V = 599·2 Å^3, Z = 2, D_x = 1·276 Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 2·9 mm^{-1}, $F(000)$ = 248, T = 293 K. The structure was refined to R = 0·041 for 948 unique observed reflections. The molecule possesses a crystallographic centre of symmetry. The P—C bond lengths in the four-membered ring are 1·888 (2) and 1·915 (2) Å, with ring bond angles 86·2 (1)° at P and 93·8 (1)° at C.

Experimental. A somewhat cloudy colourless block was mounted in a glass capillary. 1968 profile-fitted intensities (Clegg, 1981) were registered on a Stoe–Siemens four-circle diffractometer using graphite-monochromated Cu $K\alpha$ radiation ($2\theta_{\max}$ = 130°, one hemisphere of data). An absorption correction was applied (crystal size 0·5 × 0·35 × 0·2 mm) that, on the basis of ψ scans, gave transmission factors of 0·59–0·86. Three check reflections decreased in intensity by *ca* 2% and an appropriate correction was applied. Merging equivalents gave 997 unique reflections (R_{int} = 0·059, index ranges after merging h −7 to 7, k 0 to 15, l 0 to 8, 948 of which with $F > 4\sigma(F)$ were used for all calculations [program system *SHELX* (Sheldrick, 1976) locally modified by GMS]. Cell constants were refined from 2θ values of 54 reflections in the range 60–70°. Because the a and c axes are nearly equal, it is possible to construct a centred, metrically orthorhombic cell, but the true Laue symmetry is no higher than monoclinic.

The structure was solved by routine direct methods and subjected to anisotropic full-matrix least-squares refinement on F (H atoms located in

0108-2701/89/101665-02$03.00 © 1989 International Union of Crystallography