

The structure is defined in Tables 1 and 2 and Fig. 1.\* There appear to be no intermolecular forces stronger than van der Waals.

**Related literature.** A preliminary description of the molecular structure (attributed to Hanson, unpublished) has been given by Woon, Thompson & Robichaud

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42654 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(1984). Although they describe the material as a hydrate, there is no evidence of hydration in the crystal.

#### References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- LARSON, A. C. (1970). *Crystallographic Computing*, edited by F. R. AHMED, S. R. HALL & C. P. HUBER, pp. 291–294. Munksgaard: Copenhagen.
- LARSON, A. C. & GABE, E. J. (1978). *Computing in Crystallography*, edited by H. SCHENK, R. OLTJOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 81–89. Delft Univ. Press.
- WOON, T. C., THOMPSON, L. K. & ROBICHAUD, P. (1984). *Inorg. Chim. Acta*, **90**, 201–208.

*Acta Cryst.* (1986). **C42**, 503–505

## The Structure of a Trichothecene from *Fusarium roseum*\*

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(Received 26 August 1985; accepted 30 October 1985)

**Abstract.** 3,15-Diacetoxy-7,8-dihydroxy-12,13-epoxytrichothec-9-ene (12,13-epoxytrichothec-9-ene-3,7,8,15-tetrol 3,15-diacetate),  $C_{19}H_{26}O_8$ ,  $M_r = 382.4$ , orthorhombic,  $P2_12_12_1$ ,  $a = 10.972$  (1),  $b = 16.340$  (1),  $c = 10.212$  (1) Å,  $V = 1830.8$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.37$  (1),  $D_x = 1.387$  g cm<sup>-3</sup>,  $Cu K\alpha_1$ ,  $\lambda = 1.54056$  Å,  $\mu = 8.65$  cm<sup>-1</sup>,  $F(000) = 816$ ,  $T = 296$  K, final  $R = 0.042$  for 1420 unique observed reflections. The compound is a toxic metabolite present in substantial amounts in cultures of the fungus *Fusarium roseum*. The analysis confirms the formulation and establishes the stereochemistry. The torsion angle (O–C–C–O) of the hydroxyl-bearing 7,8 linkage is  $47.1$  (4)°. The hydroxyl group at C(8) acts as a proton donor and acceptor, forming an intermolecular hydrogen bond with the carbonyl group of the acetoxy group at C(15), and an intramolecular hydrogen bond with the adjacent hydroxyl group at C(7).

**Experimental.** Tabular crystal [010],  $0.13 \times 0.35 \times 0.35$  mm,  $D_m$  by flotation in carbon tetrachloride and cyclohexane solution. Picker four-circle diffractometer with Ni-filtered  $Cu K\alpha$  radiation,  $\theta$ - $2\theta$  scan,  $2\theta < 130^\circ$ . Cell constants from 30 reflections,  $56 < \theta < 64^\circ$ .

Absorption corrections (Gaussian integration) ranged from 1.117 to 1.312. Max.  $(\sin\theta)/\lambda = 0.588$  Å<sup>-1</sup>,  $h = 0$  to 12,  $k = 0$  to 19,  $l = 0$  to 12. 1 standard reflection (103) measured every 60 reflections, mean intensity 48 750(288) counts; no indication of specimen decay. 1798 unique reflections scanned, of which 378 unobserved [ $I_{net} < 3\sigma(I_{net})$ ;  $\sigma$  evaluated from counting statistics]. Net intensities derived by analysis of individual reflection profiles (Grant & Gabe, 1978). Structure solved by direct methods (*MULTAN80*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refined by block-diagonal least squares, minimizing  $\sum w\Delta F^2$ ,  $w^{-1} = \sigma^2(F_o) + 0.0003F_o^2$ . Where possible, H atoms placed in reasonable positions (C–H = 1.08 Å); otherwise (hydroxyl and methyl groups) located in difference Fourier synthesis. Reasonable isotropic temperature factors assigned:  $U = 0.08$  Å<sup>2</sup> for methyl H, 0.06 Å<sup>2</sup> otherwise. Parameters refined were scale factor, extinction coefficient ( $7.27 \times 10^4$ ; Larson, 1970), atomic coordinates and anisotropic temperature factors for C and O. Final  $R = 0.042$  (0.056 including unobserved reflections);  $wR = 0.057$ ,  $S = 2.10$ . Ratio of the max. shift to e.s.d. in final cycle 0.1. Density in final difference Fourier synthesis did not exceed  $\pm 0.2$  e Å<sup>-3</sup>. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

\* NRCC No. 25174.

Computer program system described by Larson & Gabe (1978). The structure is defined in Tables 1 and 2 and Figs. 1 and 2.\*

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42612 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates (e.s.d.'s) and equivalent isotropic temperature factors for the non-hydrogen atoms

	$x$	$y$	$z$	$B_{eq}(\text{\AA}^2)$
O(1)	0.4142 (2)	0.7888 (1)	0.4412 (3)	3.5
C(2)	0.4251 (3)	0.7115 (2)	0.3779 (5)	3.9
C(3)	0.3467 (4)	0.7043 (2)	0.2552 (5)	4.3
C(4)	0.4200 (4)	0.7460 (3)	0.1466 (4)	4.4
C(5)	0.5488 (3)	0.7667 (2)	0.2073 (4)	3.8
C(6)	0.5479 (3)	0.8547 (2)	0.2765 (4)	3.5
C(7)	0.6554 (3)	0.8637 (2)	0.3745 (4)	3.6
C(8)	0.6545 (4)	0.9459 (2)	0.4484 (4)	4.4
C(9)	0.5297 (4)	0.9695 (2)	0.4954 (4)	4.0
C(10)	0.4315 (3)	0.9307 (2)	0.4544 (4)	3.8
C(11)	0.4310 (3)	0.8610 (2)	0.3589 (4)	3.3
C(12)	0.5499 (4)	0.7041 (2)	0.3161 (4)	3.9
C(13)	0.6556 (4)	0.6662 (3)	0.3813 (5)	5.0
C(14)	0.6502 (4)	0.7565 (3)	0.1063 (5)	5.2
C(15)	0.5560 (3)	0.9248 (3)	0.1759 (4)	3.9
C(16)	0.5264 (5)	1.0402 (3)	0.5895 (5)	6.0
O(17)	0.2302 (2)	0.7449 (2)	0.2753 (3)	5.2
C(18)	0.1297 (3)	0.7054 (2)	0.2472 (4)	3.6
O(19)	0.1272 (3)	0.6369 (2)	0.2134 (5)	7.6
C(20)	0.0187 (4)	0.7582 (3)	0.2652 (4)	4.5
O(21)	0.7713 (3)	0.8526 (2)	0.3151 (3)	5.4
O(22)	0.5874 (3)	0.6219 (2)	0.2824 (3)	5.1
O(23)	0.7032 (3)	1.0107 (2)	0.3694 (4)	6.2
O(24)	0.4611 (3)	0.9181 (2)	0.0799 (3)	4.5
C(25)	0.4681 (4)	0.9673 (2)	-0.0235 (4)	3.7
O(26)	0.5483 (3)	1.0166 (2)	-0.0386 (3)	4.8
C(27)	0.3653 (4)	0.9532 (3)	-0.1169 (5)	5.5

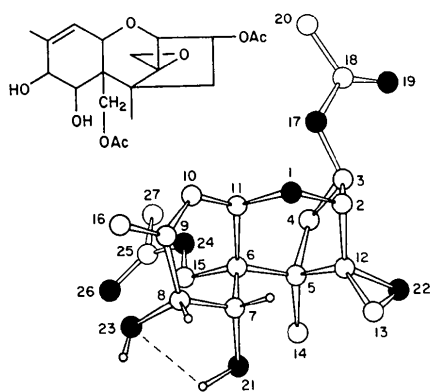


Fig. 1. Structure formula and a general view of the molecule. O atoms are solid circles and H atoms [shown only at C(7), C(8) and hydroxyl groups] are smaller circles. The broken line represents intramolecular hydrogen bonding.

**Related literature.** The production and structure of this and other metabolites of *Fusarium roseum* are discussed by Greenhalgh, Hanson, Miller & Taylor (1984) and by Greenhalgh, Meier, Blackwell, Miller, Taylor &

Table 2. Interatomic distances (including those for two apparent H bonds) and angles and their e.s.d.'s ( $\text{\AA}$ ,  $^\circ$ )

O(1)—C(2)	1.424 (5)	C(8)—O(23)	1.434 (5)
O(1)—C(11)	1.461 (4)	C(9)—C(10)	1.318 (6)
C(2)—C(3)	1.525 (7)	C(9)—C(16)	1.504 (6)
C(2)—C(12)	1.512 (6)	C(10)—C(11)	1.500 (6)
C(3)—C(4)	1.530 (7)	C(12)—C(13)	1.474 (6)
C(3)—O(17)	1.454 (5)	C(12)—O(22)	1.446 (5)
C(4)—C(5)	1.580 (6)	C(13)—O(22)	1.450 (6)
C(5)—C(6)	1.602 (6)	C(15)—O(24)	1.434 (5)
C(5)—C(12)	1.511 (6)	O(17)—C(18)	1.309 (5)
C(5)—C(14)	1.526 (6)	C(18)—O(19)	1.172 (5)
C(6)—C(7)	1.554 (5)	C(18)—C(20)	1.504 (6)
C(6)—C(11)	1.537 (5)	O(24)—C(25)	1.330 (5)
C(6)—C(15)	1.541 (6)	C(25)—O(26)	1.203 (5)
C(7)—C(8)	1.542 (6)	C(25)—O(27)	1.495 (6)
C(7)—O(21)	1.421 (5)	O(21)...O(23)	2.747 (5)
C(8)—C(9)	1.501 (7)	O(23)...O(26)*	2.919 (5)
C(2)—O(1)—C(11)	116.4 (3)	C(9)—C(8)—O(23)	109.3 (4)
O(1)—C(2)—C(3)	113.1 (3)	C(8)—C(9)—C(10)	121.4 (4)
O(1)—C(2)—C(12)	109.6 (3)	C(8)—C(9)—C(16)	115.0 (4)
C(3)—C(2)—C(12)	99.3 (4)	C(10)—C(9)—C(16)	123.5 (4)
C(2)—C(3)—C(4)	105.4 (3)	C(9)—C(10)—C(11)	125.0 (4)
C(2)—C(3)—O(17)	110.2 (4)	O(1)—C(11)—C(6)	111.5 (3)
C(4)—C(3)—O(17)	111.2 (4)	O(1)—C(11)—C(10)	103.9 (3)
C(3)—C(4)—C(5)	106.3 (4)	C(6)—C(11)—C(10)	113.9 (3)
C(4)—C(5)—C(6)	111.1 (3)	C(2)—C(12)—C(5)	104.2 (3)
C(4)—C(5)—C(12)	98.7 (3)	C(2)—C(12)—C(13)	123.9 (4)
C(4)—C(5)—C(14)	111.4 (4)	C(2)—C(12)—O(22)	115.6 (3)
C(6)—C(5)—C(12)	106.4 (3)	C(5)—C(12)—C(13)	128.5 (4)
C(6)—C(5)—C(14)	113.6 (4)	C(5)—C(12)—O(22)	117.1 (4)
C(12)—C(5)—C(14)	114.7 (3)	C(13)—C(12)—O(22)	59.5 (3)
C(5)—C(6)—C(7)	111.4 (3)	C(12)—C(13)—O(22)	59.3 (3)
C(5)—C(6)—C(11)	107.9 (3)	C(6)—C(15)—O(24)	110.9 (3)
C(5)—C(6)—C(15)	111.9 (3)	C(3)—O(17)—C(18)	119.0 (3)
C(7)—C(6)—C(11)	105.9 (3)	O(17)—C(18)—O(19)	123.7 (4)
C(7)—C(6)—C(15)	108.4 (3)	O(17)—C(18)—C(20)	111.9 (3)
C(11)—C(6)—C(15)	111.3 (3)	O(19)—C(18)—C(20)	124.4 (4)
C(6)—C(7)—C(8)	113.1 (3)	C(12)—O(22)—C(13)	61.2 (3)
C(6)—C(7)—O(21)	113.1 (4)	C(15)—O(24)—C(25)	117.1 (3)
C(8)—C(7)—O(21)	109.0 (3)	O(24)—C(25)—O(26)	123.3 (4)
C(7)—C(8)—C(9)	112.7 (3)	O(24)—C(25)—C(27)	111.7 (4)
C(7)—C(8)—O(23)	111.5 (4)	O(26)—C(25)—C(27)	125.0 (4)

\* Atom at the equivalent position  $1\frac{1}{2} - x, 2 - y, \frac{1}{2} + z$ .

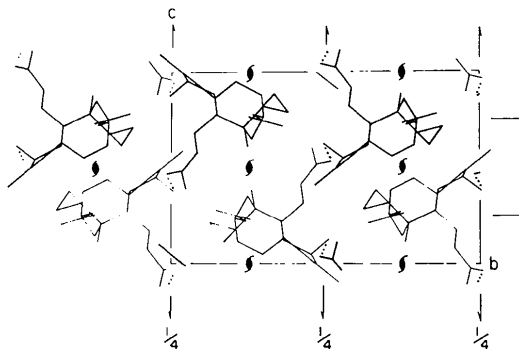


Fig. 2. *a*-axis projection. Intermolecular H bonds are shown as dotted lines.

ApSimon (1984). (In the latter work a *trans* stereochemistry was tentatively assigned to the hydroxyl-bearing 7,8 linkage. This assignment is now shown to be incorrect.)

#### References

- GRANT, D. F. & GABE, E. J. (1978). *J. Appl. Cryst.* **11**, 114–120.  
 GREENHALGH, R., HANSON, A. W., MILLER, J. D. & TAYLOR, A. (1984). *J. Agric. Food Chem.* **32**, 945–948.  
 GREENHALGH, R., MEIER, R.-M., BLACKWELL, B. A., MILLER, J. D., TAYLOR, A. & AP-SIMON, J. W. (1984). *J. Agric. Food Chem.* **32**, 1261–1264.  
 International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)  
 LARSON, A. C. (1970). *Crystallographic Computing*, edited by F. R. AHMED, S. R. HALL & C. P. HUBER, pp. 291–294. Munksgaard: Copenhagen.  
 LARSON, A. C. & GABE, E. J. (1978). *Computing in Crystallography*, edited by H. SCHENK, R. OLT-HOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 81–89. Delft Univ. Press.  
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

*Acta Cryst.* (1986). **C42**, 505–506

## Structure of Bis(1,2,3-triaminoguanidinium) Bis(5-aminotetrazolate) Monohydrate

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(Received 17 September 1984; accepted 11 October 1985)

**Abstract.**  $2\text{CH}_5\text{N}_6^+ \cdot 2\text{CH}_2\text{N}_5^- \cdot \text{H}_2\text{O}$ ,  $M_r = 396.4$ , monoclinic,  $C2/c$ ,  $a = 10.448$  (2),  $b = 10.387$  (1),  $c = 15.544$  (3) Å,  $\beta = 93.11$  (1)°,  $V = 1684.4$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.56$  cm<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.5418$  Å,  $\mu = 10.01$  cm<sup>-1</sup>,  $F(000) = 840$ ,  $T = 296$  (1) K, final  $R = 0.041$  for 956 unique observed reflections. Ions and water molecules are packed in hydrogen-bonded helicoidal chains which are laterally hydrogen-bonded to form an infinite three-dimensional network. Close non-hydrogen-bonded N...N contacts are 3.043 (4) Å between unlike ions, 3.143 (4) Å between cations and 3.114 (4) Å between anions. Short hydrogen-bonded distances are 2.925 (4) Å (N—H...N) between cations, 2.899 (4) Å (N—H...N) between unlike ions, and 2.838 (3) Å (O—H...N) between the water molecule and the anion.

**Experimental.** Title compound [(TAG5AT)<sub>2</sub>·H<sub>2</sub>O]\* recrystallized from water, pale-pink acicular crystal (0.05 × 0.05 × 0.20 mm) mounted on a glass fiber with its long axis approximately coaxial with the  $\phi$  axis of the goniometer. Enraf-Nonius CAD-4 computer-controlled  $\kappa$ -axis diffractometer equipped with a graphite-crystal incident-beam monochromator. Lattice parameters obtained using 25 reflections in range

$6 < \theta < 19^\circ$ . Intensity data\* (upper limit  $150^\circ 2\theta$ ) collected with  $\omega$ - $\theta$  scan technique on 1894 reflections (1733 unique,  $h$  0→13,  $k$  -6→12,  $l$  -19→19). Systematic absences:  $hkl$   $h+k=2n+1$ ;  $h0l$   $l=2n+1$ . 3 standard reflections, 1% intensity variation. Data corrected for Lorentz and polarization effects, no correction for absorption. A secondary-extinction correction was applied (Zachariasen, 1963); final coefficient refined by least squares to  $12.5$  (6)  $\times 10^{-6}$  (absolute units). Structure solved by direct methods using 225 reflections (minimum  $E$  of 1.55) and 2374 relationships yielding 16 phase set having: absolute figure of merit = 1.32, residual = 0.18 and  $\psi_0 = 1.160$ . Remaining atoms located in succeeding difference Fourier synthesis. Non-H atoms refined anisotropically and H atoms isotropically in full-matrix least squares.  $\sum w(|F_o| - |F_c|)^2$  minimized;  $w = 4F_o^2/\sigma^2(F_o^2)$ . Scattering factors from Cromer & Waber (1974), anomalous dispersion included (Ibers & Hamilton, 1964) and  $f'$  and  $f''$  values from Cromer (1974). Refinement of 172 variable parameters using 956

\* Provided by John C. Gray, Rockwell International, Rocketdyne Division, Canoga Park, California.

\* Lists of structure factors, anisotropic thermal parameters and interatomic hydrogen distances and angles, and hydrogen-bonding details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42577 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.