

corrected for this decay and for Lorentz and polarization effects, absorption ignored. 2437 reflections collected, 2338 unique, 1817 considered observed [$I \geq 3\sigma(I)$] and used in the refinement. Direct methods (*MULTAN80*, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), block-matrix anisotropic least squares (*SHELX76*, Sheldrick, 1976), H atoms located in a ΔF map, those of N atoms not found, in the last cycles of refinement 12 reflections probably affected by extinction were excluded, $\sum w\Delta F^2$ minimized, unit weights, $R = 0.075$, $(\Delta/\sigma)_{\max} = 0.09$, the final difference electron density map shows peaks from 0.28 to -0.23 e \AA^{-3} , 167 and 216 parameters refined in two blocks. Atomic scattering factors were from *SHELX76*.

All the calculations were performed on an IBM PS2/80 personal computer with the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreotti, 1987). The final atomic parameters are in Table 1, Table 2 reports bond distances and angles and the molecule is illustrated in Fig. 1.*

Related literature. This paper is strictly related to other systematic analyses of monomers of epoxy resins (Bel'skii, Chernikova, Rotaru & Kruchinin, 1983; Grenier-Loustalot & Bocelli, 1983).

* Lists of structure factors, anisotropic thermal parameters and H coordinates with isotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51933 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Crystalline Modification of Spironolactone

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Abstract. 7α -Acetylthio-3-oxo- 17α -pregn-4-ene-21,17 β -carbolactone, $\text{C}_{24}\text{H}_{32}\text{O}_4\text{S}$, $M_r = 416.6$, orthorhombic, $P2_12_12_1$, $a = 10.584(4)$, $b = 11.005(2)$, $c = 18.996(3) \text{ \AA}$, $V = 2213(2) \text{ \AA}^3$, $Z = 4$, $D_x = 1.25 \text{ Mg m}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.7107 \text{ \AA}$, $\mu = 0.16 \text{ mm}^{-1}$, $F(000) = 896$, $T = 294 \text{ K}$, final $R = 0.047$ for 1237 reflections. The *A*-ring conformation is near that of a sofa, the *B* and *C* rings are chair shaped. The *D* ring is a distorted 13β envelope ($\Delta = 32.5^\circ$, $\varphi_m = 45.9^\circ$) and the *E* ring is almost a plane. All the molecules are held together by van der Waals forces.

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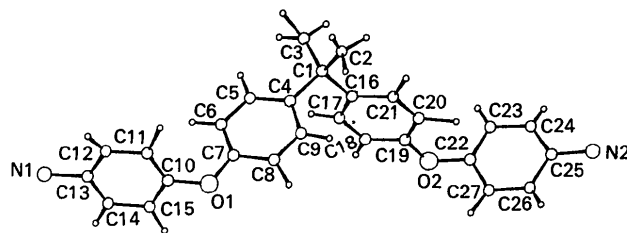


Fig. 1. A perspective view of the molecule with the atomic numbering scheme.

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The bioavailability of spironolactone (a useful diuretic, poorly soluble) depends on its allotropic form. Therefore, this crystalline modification, which is the more thermodynamically stable, has been studied.

Experimental. Single crystals prepared by cooling a supersaturated solution of spironolactone in acetone. Prismatic crystal: $0.22 \times 0.37 \times 0.40 \text{ mm}$. Enraf–Nonius CAD-4 diffractometer; lattice parameters determined from 25 reflections having $5.04 \leq \theta \leq$

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Table 1. Fractional coordinates ($\times 10^4$) and equivalent B 's for non-H atoms with e.s.d.'s in parentheses

$$B_{eq} = \frac{1}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}abc\cos\gamma + \beta_{13}accos\beta + \beta_{23}bccos\alpha).$$

	x	y	z	$B(\text{\AA}^2)$
C(1)	5438 (6)	3878 (5)	1012 (3)	5.2 (1)
C(2)	5457 (7)	3675 (7)	1819 (3)	6.9 (2)
C(3)	5349 (7)	4802 (8)	2193 (3)	8.3 (2)
C(4)	4529 (6)	5719 (6)	1902 (3)	5.9 (2)
C(5)	3976 (5)	5611 (5)	1275 (3)	4.4 (1)
C(6)	2997 (5)	6505 (5)	1027 (3)	5.0 (1)
C(7)	3259 (5)	7015 (5)	318 (3)	4.1 (1)
C(8)	3486 (5)	5979 (5)	-230 (3)	3.9 (1)
C(9)	4512 (5)	5110 (5)	29 (3)	3.9 (1)
C(10)	4236 (5)	4562 (5)	768 (3)	3.9 (1)
C(11)	4823 (6)	4134 (5)	-521 (3)	4.9 (1)
C(12)	5059 (6)	4648 (5)	-1255 (3)	5.2 (1)
C(13)	3983 (5)	5447 (6)	-1506 (3)	4.9 (1)
C(14)	3786 (5)	6442 (5)	-945 (3)	4.2 (1)
C(15)	2897 (6)	7333 (6)	-1325 (3)	6.3 (2)
C(16)	3166 (7)	7209 (8)	-2087 (3)	7.6 (2)
C(17)	4191 (6)	6218 (6)	-2160 (3)	5.9 (2)
C(18)	2794 (6)	4658 (6)	-1613 (3)	6.1 (2)
C(19)	3113 (6)	3713 (6)	759 (3)	4.9 (1)
C(20)	5534 (6)	6730 (7)	-2249 (3)	7.5 (2)
C(21)	6014 (7)	6160 (7)	-2893 (4)	8.5 (2)
C(22)	4991 (6)	5437 (6)	-3200 (3)	5.8 (2)
C(23)	3806 (5)	9451 (5)	356 (3)	4.9 (1)
C(24)	4679 (7)	10504 (5)	450 (3)	6.5 (2)
O(25)	4990 (4)	4879 (5)	-3739 (2)	8.1 (1)
O(26)	3988 (4)	5500 (5)	-2803 (2)	7.8 (1)
O(27)	2700 (4)	9564 (4)	299 (3)	8.6 (1)
O(28)	5898 (6)	4978 (7)	2771 (2)	12.8 (2)
S(29)	4600 (1)	8055 (1)	349.1 (8)	4.46 (3)

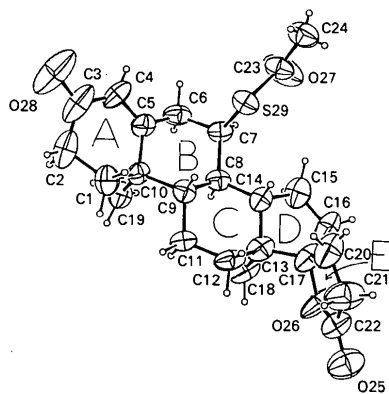


Fig. 1. View of the molecule with the atom numbering.

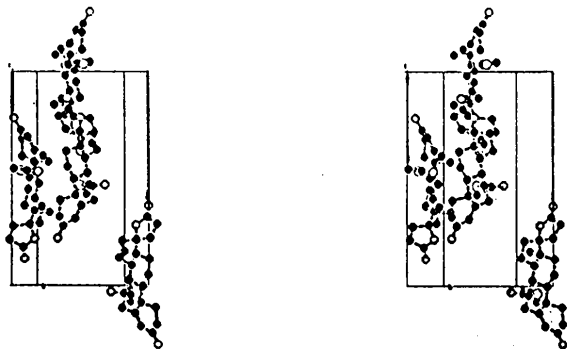


Fig. 2. Stereoview of the spironolactone molecules in the unit cell.

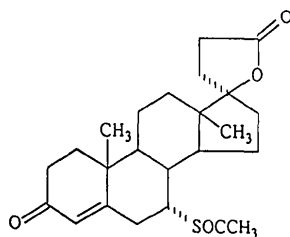
Table 2. Selected bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

C(1)—C(2)	1.549 (8)	C(12)—C(13)	1.516 (8)
C(1)—C(10)	1.549 (8)	C(13)—C(14)	1.542 (8)
C(2)—C(3)	1.434 (11)	C(13)—C(17)	1.520 (8)
C(3)—C(4)	1.441 (10)	C(13)—C(18)	1.542 (8)
C(3)—O(28)	1.258 (8)	C(14)—C(15)	1.538 (8)
C(4)—C(5)	1.334 (8)	C(15)—C(16)	1.482 (9)
C(5)—C(6)	1.504 (8)	C(16)—C(17)	1.545 (10)
C(5)—C(10)	1.528 (8)	C(17)—C(20)	1.539 (9)
C(6)—C(7)	1.484 (8)	C(17)—O(26)	1.470 (7)
C(7)—C(8)	1.563 (7)	C(20)—C(21)	1.466 (10)
C(7)—S(29)	1.824 (5)	C(21)—C(22)	1.464 (10)
C(8)—C(9)	1.528 (7)	C(22)—O(25)	1.194 (8)
C(8)—C(14)	1.485 (7)	C(22)—O(26)	1.304 (7)
C(9)—C(10)	1.556 (7)	C(23)—C(24)	1.492 (8)
C(9)—C(11)	1.535 (8)	C(23)—O(27)	1.183 (7)
C(10)—C(19)	1.512 (8)	C(23)—S(29)	1.752 (6)
C(11)—C(12)	1.525 (8)		

C(2)—C(1)—C(10)	112.1 (5)	C(12)—C(13)—C(14)	107.2 (4)
C(1)—C(2)—C(3)	111.4 (6)	C(12)—C(13)—C(17)	118.2 (5)
C(2)—C(3)—C(4)	117.6 (6)	C(12)—C(13)—C(18)	109.1 (5)
C(2)—C(3)—O(28)	121.9 (7)	C(14)—C(13)—C(17)	100.8 (5)
C(4)—C(3)—O(28)	120.4 (7)	C(14)—C(13)—C(18)	112.4 (5)
C(3)—C(4)—C(5)	123.0 (6)	C(17)—C(13)—C(18)	109.0 (5)
C(4)—C(5)—C(6)	121.6 (5)	C(8)—C(14)—C(13)	114.7 (5)
C(4)—C(5)—C(10)	123.4 (5)	C(8)—C(14)—C(15)	121.1 (5)
C(6)—C(5)—C(10)	114.9 (4)	C(13)—C(14)—C(15)	102.2 (4)
C(5)—C(6)—C(7)	113.7 (5)	C(14)—C(15)—C(16)	106.4 (5)
C(6)—C(7)—C(8)	111.0 (4)	C(15)—C(16)—C(17)	106.7 (5)
C(6)—C(7)—S(29)	110.7 (4)	C(13)—C(17)—C(16)	102.7 (5)
C(8)—C(7)—S(29)	111.0 (3)	C(13)—C(17)—C(20)	115.4 (5)
C(7)—C(8)—C(9)	110.5 (4)	C(13)—C(17)—O(26)	111.0 (5)
C(7)—C(8)—C(14)	113.1 (4)	C(16)—C(17)—C(20)	113.6 (6)
C(9)—C(8)—C(14)	111.0 (4)	C(16)—C(17)—O(26)	110.6 (5)
C(8)—C(9)—C(10)	113.6 (4)	C(20)—C(17)—O(26)	103.9 (5)
C(8)—C(9)—C(11)	111.8 (4)	C(17)—C(20)—C(21)	104.8 (6)
C(10)—C(9)—C(11)	112.6 (4)	C(20)—C(21)—C(22)	108.0 (6)
C(1)—C(10)—C(5)	109.1 (4)	C(21)—C(22)—O(25)	128.5 (6)
C(1)—C(10)—C(9)	107.7 (4)	C(21)—C(22)—O(26)	110.0 (5)
C(1)—C(10)—C(19)	110.5 (5)	O(25)—C(22)—O(26)	121.5 (6)
C(5)—C(10)—C(9)	108.0 (4)	C(24)—C(23)—O(27)	122.8 (5)
C(5)—C(10)—C(19)	109.4 (4)	C(24)—C(23)—S(29)	112.7 (4)
C(9)—C(10)—C(19)	112.1 (4)	O(27)—C(23)—S(29)	124.5 (5)
C(9)—C(11)—C(12)	113.5 (4)	C(17)—O(26)—C(22)	113.0 (5)
C(11)—C(12)—C(13)	112.3 (5)	C(7)—S(29)—C(23)	100.2 (3)

17.36 $^\circ$, θ -2 θ -scan technique; $0.039 \leq (\sin \theta)/\lambda \leq 0.595 \text{ \AA}^{-1}$, $0 \leq h \leq 12$, $0 \leq k \leq 13$, $0 \leq l \leq 22$. No significant deterioration in intensity, average $\sigma(I)/I$ (three standard reflections: $\bar{1}2\bar{3}$, $\bar{1}\bar{1}4$ and $2\bar{3}\bar{3}$) = 0.0022; no correction for absorption, 2227 independent reflections, 990 unobserved reflections [$I < 2\sigma(I)$]. Direct methods, program MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), H atoms located from a Fourier difference map, full-matrix refinements, refined parameters: x , y , z and β_{ij} for C, S and O atoms. H parameters not refined because of insufficient F_o number. For each H atom, B equal to $1 + B_{eq} \text{ \AA}^2$ of bonded heavy atom, $R = 0.047$, $wR = 0.049$, $w = 1/\sigma^2(F)$, $S = 1.57$, maximum shift to e.s.d. ratio $(\Delta/\sigma)_{\max} = 0.03$, $|\Delta\rho|_{\max} = 0.18 (4) e \text{ \AA}^{-3}$. Atomic diffusion factors from *International Tables for X-ray Crystallography* (1974). Computer programs of SDP system (Frenz, 1982); torsion angles: ORFFE program (Busing, Martin & Levy, 1964); angles of the least-squares planes (Ito &

Sugawara, 1983). Atomic parameters are given in Table 1, bond lengths and angles in Table 2.* Fig. 1 shows the molecular structure (ORTEPII; 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,

* 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.

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Structure of the Simple Trichothecene 7 α -Hydroxyscirpene

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Abstract. 12,13-Epoxytrichothec-9-en-7 α -ol, C₁₅H₂₂O₃, $M_r = 250.3$, orthorhombic, $P2_12_12_1$, $a = 6.522(2)$, $b = 12.187(2)$, $c = 16.647(2)$ Å, $V = 1323.1(3)$ Å³, $Z = 4$, $D_x = 1.257$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 0.652$ mm⁻¹, $F(000) = 544$, $T = 295$ K, final $R = 0.034$, $wR = 0.036$ for 1043 independent reflections. The title compound is produced by deoxygenation of 7 α -hydroxytrichodermol which is produced as a minor metabolite of *Myrothecium roridum* along with much larger amounts of macrocyclic roridum and verrucarol trichothecenes. The X-ray structure established the hydroxyl group at the

7 α rather than the 8 β 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 0.13 × 0.15 × 0.34 mm data crystal was provided by Bruce Jarvis of the University of Maryland. Automated Nicolet R3m diffractometer with incident beam mono-