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Eremofortin D, a Valencane-Class Sesquiterpene

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Abstract. $C_{17}H_{24}O_6$, monoclinic, $P2_1$, $a = 11{\cdot}253$ (3), $b = 6{\cdot}341$ (3), $c = 11{\cdot}539$ (5) Å, $\beta = 93{\cdot}0$ (2)°, $Z = 2$. The structure was solved by direct methods and refined to $R = 5{\cdot}7\%$. The molecule is characterized by two *trans* ring junctions.

Introduction. A single crystal of eremofortin D, grown from ethyl acetate–CCl_4, was mounted on a Philips PW 1100 automatic diffractometer. 1565 reflexions were measured with Cu $K\alpha$ radiation, monochromated with a graphite crystal. The structure was solved by *MULTAN* (Germain, Main & Woolfson, 1971). The E map corresponding to the best figure of merit gave all the heavy atoms. Refinement was carried out by full-matrix least squares using a modified version of *ORFLS* (Busing, Martin & Levy, 1962). The thermal parameters of the non-hydrogen atoms were anisotropic. All H atoms were introduced in the refinement procedure at their positions found in electron-density difference maps. Their thermal factors were kept isotropic at the values of the atoms to which they were bonded.

The scattering factors were those of Doyle & Turner (1968) for heavy atoms, and those of Stewart, Davidson & Simpson (1965) for H atoms.

The final R was 5·7%. Fractional coordinates for the heavy atoms are given in Table 1 and those for H in Table 2.*

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32710 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. Eremofortin D has been isolated from a culture of *Penicillium roqueforti* as previously reported (Moreau, Gaudemer, Lablache-Combier & Biguet, 1976): m.p. 209–211 °C, $[\alpha]_D = +91°$ ($c = 1{\cdot}17\%$, $CHCl_3$); $(\varphi)_{589} = +282°$, $(\varphi)_{578} = +295°$, $(\varphi)_{546} = +334°$, $(\varphi)_{436} = +570°$, $(\varphi)_{365} = +891°$; $M_r = 324{\cdot}36$.

Table 1. *Fractional coordinates* (×10⁴) *for the heavy atoms*

The e.s.d.'s are given in parentheses.

	x	y	z
C(1)	6119 (3)	13608 (7)	816 (3)
C(2)	5168 (3)	14545 (7)	1471 (3)
C(3)	5160 (3)	14339 (7)	2767 (3)
C(4)	6333 (3)	13500 (6)	3333 (3)
C(5)	6884 (3)	11646 (6)	2657 (3)
C(6)	8108 (3)	11042 (6)	3266 (3)
C(7)	8738 (3)	9553 (7)	2523 (3)
C(8)	8920 (3)	10257 (6)	1287 (3)
C(9)	7754 (3)	10869 (7)	662 (3)
C(10)	7155 (3)	12501 (7)	1437 (3)
C(11)	9621 (3)	7874 (7)	2766 (3)
C(12)	10246 (4)	7532 (8)	1670 (4)
C(13)	10189 (4)	7274 (9)	3913 (3)
C(14)	6188 (3)	13089 (7)	4633 (3)
C(15)	6076 (3)	9698 (6)	2624 (3)
C(16)	3088 (3)	13750 (8)	2954 (4)
C(17)	2197 (4)	12127 (11)	3299 (5)
O(1)	4952 (2)	12619 (6)	804 (3)
O(2)	8373 (2)	7342 (5)	2556 (3)
O(3)	9718 (2)	11993 (0)	1415 (2)
O(4)	9502 (2)	8528 (5)	761 (2)
O(5)	4192 (2)	12966 (5)	3080 (3)
O(6)	2864 (3)	15496 (7)	2627 (4)

Composition: C 62·95, H 7·46% (calculated for $C_{17}H_{24}O_6$: C 62·95, H 7·46%).

Fig. 1, which shows the *ORTEP* (Johnson, 1965) drawing of the molecule as obtained from our X-ray results, confirms fully the proposed formula. Tables 3 and 4 give the bond distances between heavy atoms and their valency angles.

As shown in Fig. 2, the molecule is characterized by two *trans* ring junctions. The C(14) and C(15) methyl groups are mutually *cis*. With respect to these methyl groups, the C(7)—C(11) epoxide is *syn*. This configuration is characteristic of the valencane class of sesquiterpenes (Overton & Roberts, 1971).

Studies on the toxicity of PR toxin and related compounds (Moreau, Gaudemer, Lablache-Combier & Biguet, 1976; Moreau, Moulé & Bousquet, 1976) revealed the importance of the functional groups at C(7), C(8), C(11) and C(12). A knowledge of the stereochemistry of the sesquiterpenes produced by *P. roqueforti* is thus necessary in order to be able to draw conclusions on the relationships which may exist between chemical structure and biological activity.

Table 2. *Fractional coordinates* ($\times 10^3$) *for the hydrogen atoms*

	x	*y*	*z*
H(1)	632	1439	4
H(2)	486	1550	111
H(3)	509	1613	299
H(4)	692	1475	328
H(6*A*)	795	1051	401
H(6*B*)	860	1227	340
H(9*A*)	793	1152	−6
H(9*B*)	723	938	59
H(10)	775	1352	156
H(12*A*)	1038	598	151
H(12*B*)	1107	808	165
H(13*A*)	970	758	453
H(13*B*)	1028	569	406
H(13*C*)	1104	759	393
H(15*A*)	637	870	220
H(15*B*)	584	942	341
H(15*C*)	547	987	243
H(17*A*)	207	1260	427
H(17*B*)	247	1071	330
H(17*C*)	149	1247	291
H(O3)	1004	1234	71

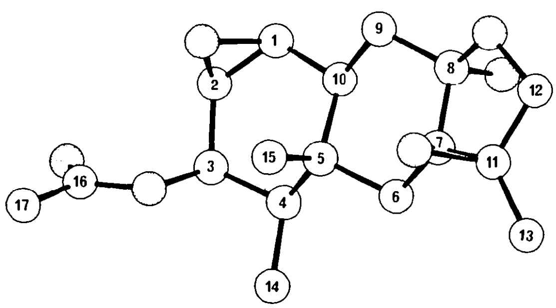

Fig. 1. An *ORTEP* drawing of the molecule.

Table 3. *Intramolecular bond distances* (Å)

The e.s.d.'s in parentheses refer to the last digit.

C(1)—C(2)	1·467 (5)	C(7)—C(11)	1·473 (6)
C(1)—C(10)	1·510 (5)	C(7)—O(2)	1·462 (5)
C(1)—O(1)	1·455 (5)	C(8)—C(9)	1·514 (5)
C(2)—C(3)	1·502 (5)	C(8)—O(3)	1·423 (4)
C(2)—O(1)	1·457 (6)	C(8)—O(4)	1·428 (5)
C(3)—C(4)	1·536 (5)	C(9)—C(10)	1·545 (5)
C(3)—O(5)	1·454 (5)	C(11)—C(12)	1·494 (6)
C(4)—C(5)	1·558 (5)	C(11)—C(13)	1·489 (6)
C(4)—C(14)	1·540 (5)	C(11)—O(2)	1·452 (5)
C(5)—C(6)	1·561 (5)	C(12)—O(4)	1·452 (5)
C(5)—C(10)	1·553 (5)	C(16)—C(17)	1·505 (7)
C(5)—C(15)	1·533 (5)	C(16)—O(5)	1·339 (5)
C(6)—C(7)	1·481 (5)	C(16)—O(6)	1·192 (7)
C(7)—C(8)	1·519 (5)		

Table 4. *Intramolecular bond angles* (°)

The e.s.d.'s in parentheses refer to the last digit.

C(2)—C(1)—C(10)	120·7 (3)	C(7)—C(8)—C(9)	111·5 (3)
C(2)—C(1)—O(1)	59·8 (3)	C(7)—C(8)—O(3)	104·1 (3)
C(10)—C(1)—O(1)	118·6 (3)	C(7)—C(8)—O(4)	105·1 (3)
C(1)—C(2)—C(3)	121·3 (4)	C(9)—C(8)—O(3)	112·2 (3)
C(1)—C(2)—O(1)	59·7 (3)	C(9)—C(8)—O(4)	113·6 (3)
C(3)—C(2)—O(1)	116·3 (3)	O(3)—C(8)—O(4)	109·7 (3)
C(2)—C(3)—C(4)	113·8 (3)	C(8)—C(9)—C(10)	106·8 (3)
C(2)—C(3)—O(5)	110·1 (3)	C(1)—C(10)—C(5)	114·1 (3)
C(4)—C(3)—O(5)	109·0 (3)	C(1)—C(10)—C(9)	112·7 (3)
C(3)—C(4)—C(5)	113·8 (3)	C(5)—C(10)—C(9)	113·8 (3)
C(3)—C(4)—C(14)	109·9 (3)	C(7)—C(11)—C(12)	106·6 (3)
C(5)—C(4)—C(14)	115·1 (3)	C(7)—C(11)—C(13)	127·4 (4)
C(4)—C(5)—C(6)	109·0 (3)	C(7)—C(11)—O(2)	60·0 (2)
C(4)—C(5)—C(10)	107·0 (3)	C(12)—C(11)—C(13)	121·1 (4)
C(4)—C(5)—C(15)	111·7 (3)	C(12)—C(11)—O(2)	108·6 (3)
C(6)—C(5)—C(10)	106·4 (3)	C(13)—C(11)—O(2)	117·1 (3)
C(6)—C(5)—C(15)	108·8 (3)	C(11)—C(12)—O(4)	105·6 (3)
C(10)—C(5)—C(15)	113·8 (3)	C(17)—C(16)—O(5)	110·2 (4)
C(5)—C(6)—C(7)	109·5 (3)	C(17)—C(16)—O(6)	125·9 (4)
C(6)—C(7)—C(8)	116·7 (3)	O(5)—C(16)—O(6)	123·9 (4)
C(6)—C(7)—C(11)	133·5 (3)	C(1)—O(1)—C(2)	60·5 (3)
C(6)—C(7)—O(2)	116·9 (3)	C(7)—O(2)—C(11)	60·7 (3)
C(8)—C(7)—C(11)	105·6 (3)	C(8)—O(4)—C(12)	106·7 (3)
C(8)—C(7)—O(2)	111·0 (3)	C(3)—O(5)—C(16)	117·0 (3)
C(11)—C(7)—O(2)	59·3 (2)		

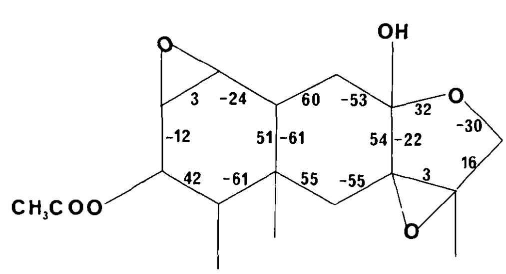

Fig. 2. Torsion angles (°) for eremofortin D.

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Rubidium Tetrachloromanganate

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Abstract. Rb_2MnCl_4, tetragonal, $I4/mmm$, a = 5·05 (2), c = 16·14 (5) Å, D_o = 2·96, D_x = 2·97 g cm^{-3}, Z = 2. Mn ions are octahedrally coordinated by Cl ions. The Cl ions at the equatorial vertices are shared with neighbouring octahedra so that each octahedron is linked to four others to form layers perpendicular to [001], the c dimension accommodating two such layers. The Cl ions at the unshared vertices are closer to the Rb ions than the others. The structure is strictly isomorphous with that of K_2NiF_4.

Introduction. On the basis of powder data, Seifert & Koknat (1965) assigned a body-centred tetragonal cell to the compound Rb_2MnCl_4. The cell parameters [a = 5·051 (5), c = 16·18 (1) Å] and observed density (2·96 g cm^{-3}) indicated two formula units per cell, from this evidence they concluded that the structure might be isomorphous with that found for K_2NiF_4 by Balz & Plieth (1955).

The material examined in this work was prepared by heating stoichiometric amounts of RbCl and $MnCl_2$ in an evacuated silica tube until molten and then cooling the sample at about 5°C h^{-1}. Orange crystals were formed which, although of suitable size for Weissenberg study, were quite irregular in shape. The crystal selected for X-ray examination had to be mounted in a sealed Lindemann-glass tube because of the hygroscopic nature of the material.

The dimensions of the unit cell were determined from α_1–α_2 doublet separations on a zero-layer Weissenberg photograph taken with Cu $K\alpha$ radiation (λ_{α_1} = 1·54051 Å). The unit-cell dimensions and observed density agreed, within experimental error, with the data given by Seifert & Koknat.

Intensity data were collected from equi-inclination photographs taken about the a axis with Mo $K\alpha$ radiation. The intensities of 128 reflexions were measured visually on layer lines 0–4 from accurately timed film exposures. The systematically-absent reflexions were all of the type $h + k + l = 2n + 1$, consistent with the space group ($I4/mmm$) of the K_2NiF_4 structure. These data were corrected for the Lorentz–polarization factor and an approximate correction was made for absorption by assuming the crystal to be cylindrical in shape with a μr value of 1·4.

Both the axial ratio and the number of formula units per cell suggest isomorphism between Rb_2MnCl_4 and K_2NiF_4. If this is so, the a parameter should be twice the Mn–Cl bond length in a $MnCl_6$ octahedron. Goodyear, Steigmann & Ali (1977) found the average value of the latter to be 2·51 Å in the $RbMnCl_3$ structure and this is indeed very nearly equal to $a/2$. Initially, the positional parameters of the K_2NiF_4 structure were assumed and an individual temperature factor of 2·0 Å^2 was assigned to each atom. In the first few cycles of least-squares refinement all the observed structure factors were treated as being symmetrically independent and, because of the uncertain absorption correction, the observed data were scaled to the calculated values for each layer line separately. After several cycles the residual, $R = \Sigma \| F_o| - |F_c\| / \Sigma |F_o|$, reduced to 11·8%. The observed structure factors of symmetrically-equivalent reflexions were then averaged giving 91 independent values for the final cycles of refinement, after which R had decreased to 10·1%. At this stage the shifts in the atomic parameters were all less than 1/30 of a standard deviation. The calculated structure factors of the 42 unobserved reflexions were