PANAGIOTOPOULOS, N. C. & BROWN, I. D. (1972*b*). *Acta Cryst.* B**28**, 2880–2882.
PANAGIOTOPOULOS, N. C. & BROWN, I. D. (1973). *Acta Cryst.* B**29**, 890–894.
RAMACHANDRAN, G. N. (1964). *Advanced Methods of Crystallography*, pp. 37–46. London: Academic Press.
STEPHENS, J. S. & CRUICKSHANK, D. W. J. (1970). *Acta Cryst.* B**26**, 222–226.
STĘPIEŃ, A. (1973). *The Crystal Structure of Guanidinium Trichromate.* Thesis, Łódź Univ.
WAJSMAN, E., CYGLER, M., GRABOWSKI, M. J. & STĘPIEŃ, A. (1976). *Roczn. Chem.* **50**, 1587–1592.

Acta Cryst. (1977). B**33**, 2927–2930

Dendrosterone, a Stigmastane-Type Steroid

BY JAN-ERIC BERG, BENGT KARLSSON, ANNE-MARIE PILOTTI AND ANNE-CHARLOTTE SÖDERHOLM

*Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-*106 91 *Stockholm, Sweden*

(*Received* 1 *April* 1977; *accepted* 12 *May* 1977)

Abstract. $C_{29}H_{44}O_5$, monoclinic, $P2_1$, $a = 17{\cdot}551$ (2), $b = 7{\cdot}280$ (1), $c = 10{\cdot}654$ (3) Å, $\beta = 104{\cdot}64$ (1)°, $Z = 2$, $V = 1317{\cdot}4$ Å^3, $\rho_c = 1{\cdot}19$ g cm^{-3}. Dendrosterone has been isolated as a glucoside from *Dendrobium ochreatum* Lindl. The X-ray determination proved that dendrosterone has a steroid skeleton.

Introduction. Dendrosterone has been isolated as a glucoside from *Dendrobium ochreatum* Lindl. (Behr, Berg, Karlsson, Leander, Pilotti & Wiehager, 1975). Detailed spectroscopic studies and chemical degradation of dendrosterone indicated that it must have either a steroid or a triterpene skeleton, but all attempts to determine a complete structure were unsuccessful. An X-ray analysis of dendrosterone was therefore undertaken. Three-dimensional data were collected from a crystal of approximate volume 0·002 mm^3; a Philips PW 1100 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, and a θ–2θ scan procedure were used. Intensities were measured for all independent reflexions with $2\theta \leq 140°$. Of these, 1697 reflexions had $I_{\rm net} \geq 3\sigma(I_{\rm net})$ and were used in the structure refinement. The usual Lorentz and polarization corrections were applied to the data. Lattice constants were obtained from least-squares refinement of the 2θ angles for 25 reflexions.

The structure has been solved with the multisolution program *MULTAN* of Germain, Main & Woolfson (1971). A number of E maps gave probable partial structures (the A, B and C ring system, see Fig. 2) located in different parts of the unit cell. Phases from one of these partial structures could be used as a basic set for further refinements. The structure was refined by a least-squares procedure with the weighting scheme of Hughes (1941). Except for the H atom bonded to O(21), which could not be located, the positions of the H atoms were partly deduced from a difference synthesis and partly estimated from chemical considerations. The H positional and thermal parameters

Table 1. *Positional parameters* (×10^4) *of the non-hydrogen atoms with e.s.d.'s in parentheses*

	x	y	z
C(1)	6260 (3)	10816 (10)	3526 (6)
C(2)	7110 (3)	10991 (11)	3429 (6)
C(3)	7558 (3)	9196 (11)	3761 (5)
C(4)	7162 (3)	7670 (11)	2902 (6)
C(5)	6308 (3)	7445 (9)	2986 (5)
C(6)	5872 (3)	5885 (10)	2200 (6)
C(7)	5064 (3)	5519 (10)	2384 (6)
C(8)	4560 (3)	7273	2204 (5)
C(9)	5012 (3)	8852 (9)	3028 (5)
C(10)	5806 (3)	9244 (10)	2683 (5)
C(11)	4500 (3)	10563 (10)	3028 (7)
C(12)	3715 (3)	10114 (10)	3365 (6)
C(13)	3254 (3)	8640 (10)	2461 (5)
C(14)	3787 (3)	6938 (10)	2603 (5)
C(15)	3234 (4)	5417 (11)	1912 (7)
C(16)	2446 (4)	5857 (10)	2244 (6)
C(17)	2548 (3)	7805 (10)	2900 (5)
C(18)	2975 (3)	9372 (12)	1081 (6)
C(19)	5675 (3)	9768 (11)	1250 (5)
O(20)	8358 (2)	9359 (10)	3695 (5)
O(21)	6152 (3)	4967 (10)	1481 (5)
C(22)	1760 (3)	8835 (10)	2613 (5)
C(23)	1810 (4)	10686 (11)	3329 (8)
C(24)	1140 (3)	7700 (10)	3021 (5)
O(25)	1274 (2)	7009 (9)	4090 (4)
C(26)	358 (3)	7529 (10)	2055 (5)
C(27)	−265 (3)	6486 (10)	2532 (5)
C(28)	−64 (3)	4426 (10)	2744 (5)
C(29)	−460 (4)	3576 (11)	1484 (5)
O(30)	−1007 (2)	4695 (9)	784 (4)
C(31)	−1067 (3)	6386 (10)	1532 (5)
C(32)	−353 (5)	3543 (12)	3828 (6)
O(33)	−348 (3)	2062 (9)	1093 (5)
C(34)	−1296 (4)	7952 (11)	620 (7)

Table 2. *Positional* ($\times 10^3$) *and isotropic thermal* ($\times 10^2$) *parameters of the hydrogen atoms*

	x	y	z	B (Å^2)		x	y	z	B (Å^2)
H1(C1)	621	1055	436	372	H1(C18)	276	857	29	444
H2(C1)	600	1212	336	372	H2(C18)	353	948	94	444
H1(C2)	718	1147	254	447	H3(C18)	282	1068	83	444
H2(C2)	764	1188	410	447	H1(C19)	592	910	63	364
H(C3)	757	897	460	369	H2(C19)	507	924	90	364
H1(C4)	719	788	213	394	H3(C19)	558	1102	90	364
H2(C4)	747	650	309	394	H(C22)	163	895	170	299
H(C5)	628	712	393	296	H1(C23)	213	1160	301	555
H1(C7)	487	481	187	411	H2(C23)	204	1032	425	555
H2(C7)	511	507	333	411	H3(C23)	142	1107	322	555
H(C8)	431	748	131	218	H1(C26)	40	715	122	350
H(C9)	514	839	392	288	H2(C26)	22	870	170	350
H1(C11)	444	1099	228	433	H(C27)	−34	687	333	272
H2(C11)	481	1159	345	433	H(C28)	49	439	276	358
H1(C12)	338	1126	329	395	H(C31)	−163	598	190	328
H2(C12)	383	966	429	395	H1(C32)	−21	435	450	532
H(C14)	398	653	354	310	H2(C32)	−105	360	360	532
H1(C15)	304	538	71	480	H3(C32)	−21	240	429	532
H2(C15)	351	406	197	480	H1(C34)	−103	804	13	477
H1(C16)	219	482	258	442	H2(C34)	−140	911	194	477
H2(C16)	207	586	136	442	H3(C34)	−177	770	11	477
H(C17)	264	767	368	269					

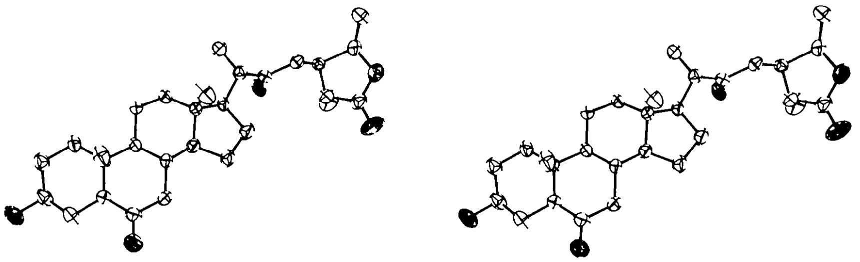
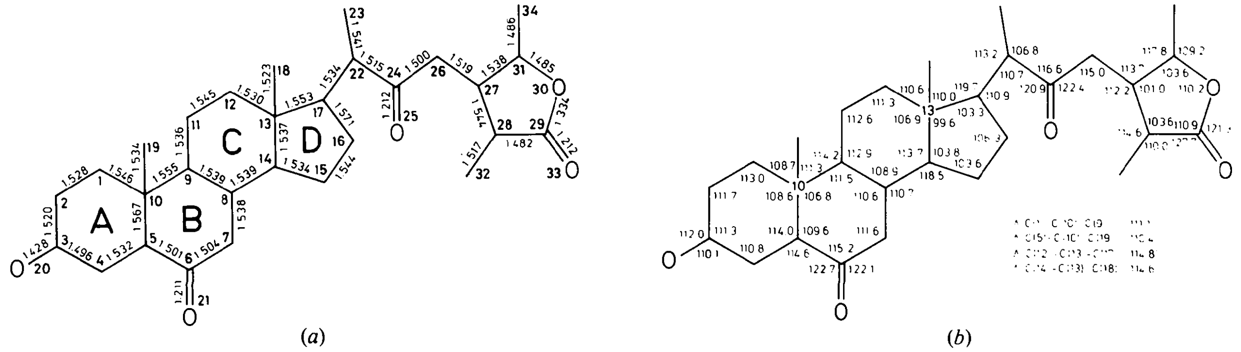


Fig. 1. A stereoscopic view of the molecule. H atoms have been omitted. The O atoms are represented by shaded ellipsoids.



(*a*) (*b*)

Fig. 2. (*a*) Bond distances (Å) and (*b*) bond angles (°) in the molecule.

were held constant throughout the refinement. The isotropic thermal parameters for the H atoms were chosen to be equal to those of the final isotropic value of their parent atoms. The atomic scattering factors for non-hydrogen atoms are those listed in *International Tables for X-ray Crystallography* (1962) and for H that of Stewart, Davidson & Simpson (1965). The final R value is 0·053. The positional parameters of the non-

Table 3. *Torsion angles* (°) *in the molecule*

C(1)–C(2)–C(3)–C(4)	57·4	C(11)–C(9)–C(8)–C(14)	−50·5	C(13)–C(17)–C(22)–C(24)	175·2
C(2)–C(3)–C(4)–C(5)	−56·6	C(9)–C(8)–C(14)–C(13)	57·6	C(16)–C(17)–C(22)–C(24)	55·1
C(3)–C(4)–C(5)–C(10)	55·1	C(8)–C(14)–C(13)–C(12)	−61·5	C(17)–C(22)–C(24)–C(26)	−132·2
C(4)–C(5)–C(10)–C(1)	−51·2	C(14)–C(13)–C(12)–C(11)	58·2	C(22)–C(24)–C(26)–C(27)	−176·2
C(5)–C(10)–C(1)–C(2)	50·9	C(13)–C(12)–C(11)–C(9)	−55·0	C(24)–C(26)–C(27)–C(28)	67·9
C(10)–C(1)–C(2)–C(3)	−55·4	C(12)–C(11)–C(9)–C(8)	50·8	C(24)–C(26)–C(27)–C(31)	178·2
C(5)–C(6)–C(7)–C(8)	51·8	C(17)–C(13)–C(14)–C(15)	48·5	C(27)–C(28)–C(29)–O(30)	−17·7
C(6)–C(7)–C(8)–C(9)	−50·3	C(13)–C(14)–C(15)–C(16)	−36·4	C(28)–C(29)–O(30)–C(31)	−3·0
C(7)–C(8)–C(9)–C(10)	57·4	C(14)–C(15)–C(16)–C(17)	9·8	C(29)–O(30)–C(31)–C(27)	22·5
C(8)–C(9)–C(10)–C(5)	−61·1	C(15)–C(16)–C(17)–C(13)	19·7	O(30)–C(31)–C(27)–C(28)	−31·2
C(9)–C(10)–C(5)–C(6)	58·9	C(16)–C(17)–C(13)–C(14)	−41·0	C(31)–C(27)–C(28)–C(29)	29·6
C(10)–C(5)–C(6)–C(7)	−56·6				

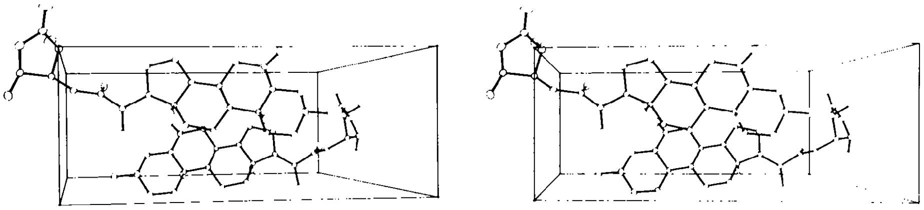
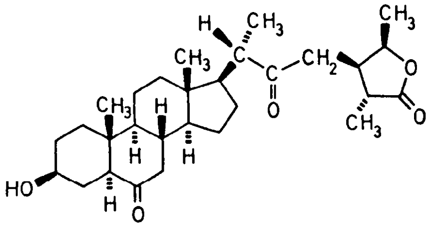


Fig. 3. A stereoscopic view of the molecular packing. The origin is at the bottom left corner of the cell, with +*a* pointing to the right and +*b* upwards. The *c* axis is directed towards the reader.

hydrogen atoms are listed in Table 1 and those for the H atoms in Table 2.*

Discussion. The result of the structure determination of (I) is illustrated in Fig. 1. Fig. 2 shows the bond lengths and angles involving the non-hydrogen atoms. Standard deviations, based solely on least-squares parameters, are of the order of 0·008 Å for the bonds and 0·5° for the angles. A wide range of $C(sp^3)$–$C(sp^3)$ bond lengths is found (1·496 to 1·571 Å). This is probably due both to interaction between non-bonded neighbouring substituents and to the ring closure.



(I)

The four rings *A*, *B*, *C* and *D* are *trans*-fused. As shown by the intra-ring torsion angles (Table 3) the *A*, *B* and *C* rings have chair conformations, and the four-atom group C(5), C(6), C(7), O(21) shows small deviations from planarity, maximally 0·005 Å. The conformation of the *D* ring is intermediate between a half-chair and a C(13)-envelope, as shown by the parameters Δ = 11·0° and φ_m = 48·7° (Altona, Geise & Romers, 1968) and by the torsion angles (*cf.* Table 3). The conformations of the C(17) side chain and the γ-lactone ring are also given in Table 3. The group C(22), C(24), O(25), C(26) is planar within 0·009 Å. The γ-lactone ring has a C(27)-envelope conformation, as shown by the torsion angle C(28)–C(29)–O(30)–C(21) = −3·0°. The atom C(27) deviates by 0·52 Å from the plane defined by the other four atoms, which are coplanar within 0·016 Å. The atom group C(28), C(29), O(30), O(33) is planar within 0·009 Å.

The arrangement of molecules is shown in Fig. 3. Molecules related by the twofold screw axis are weakly hydrogen bonded to one another, the hydroxyl group of one donating a proton to form a hydrogen bond with the carbonyl O atom of the next. The distance O(28)···O(25) is 2·99 Å.

The authors wish to express their gratitude to Professor Peder Kierkegaard for his stimulating interest in this work. Our thanks are also due to Dr Dan Behr for supplying the crystals. Dr Sven Westman who corrected the English of this paper is cordially thanked. The present investigation has received financial support from the Tricentennial Fund of the Bank of Sweden and from the Swedish Natural Science Research Council.

---

* Lists of structure factors and anisotropic thermal parameters of the non-hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32706 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

References

ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–32.

BEHR, D., BERG, J.-E., KARLSSON, B., LEANDER, K., PILOTTI, A.-M. & WIEHAGER, A.-C. (1975). *Acta Chem. Scand.* B**29**, 401.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

HUGHES, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737–1752.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1977). B**33**, 2930–2932

Eremofortin D, a Valencane-Class Sesquiterpene

BY BERNADETTE ARNOUX AND CLAUDINE PASCARD

Institut de Chimie des Substances Naturelles, CNRS, 91190 *Gif-sur-Yvette, France*

AND SERGE MOREAU

Unité INSERM U 42, *Domaine du Certia,* 369 *rue J. Guesde,* 59650 *Villeneuve d'Ascq, France*

(*Received* 21 *April* 1977; *accepted* 18 *May* 1977)

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 $5{\cdot}7\%$. Fractional coordinates for the heavy atoms are given in Table 1 and those for H in Table 2.*

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)

---

* 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.